

CLEAN AIR ENGINEERING

MANUAL OF STANDARD OPERATING PROCEDURES

SOURCE TESTING DIVISION

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USING THE SOP TEMPLATE TO WRITE AN SOP

ISO NUMBER: OO-1

AUTHOR: Jim Wright

EFFECTIVE DATE: 10/01/96

APPROVED BY: Technical Review Board

1. PURPOSE

This procedure provides guidance in the use of the CAE Standard Operating Procedure (SOP) template for writing SOPs.

2. SCOPE

This procedure applies to any CAE employee who authors a SOP for general or specific company use.

3. MATERIALS

The following materials are needed to properly execute this procedure:

- 3.1 -SOP Template File - this file needs to be located in the Templates folder defaulted for Microsoft Word in order for the document to operate correctly.
- 3.2 Computer with Microsoft Word Version 6.0 or higher
- 3.3 Information for SOP

4. DEFINITIONS

- 4.1 SOP - Standard Operating Procedure - generally, a document which contains information and instructions for performing a specific task or series of operations. The document has been peer-reviewed to a sufficient extent to be considered "standard." An SOP may also be a set of guidelines (as opposed to specific instructions) pertaining to a certain activity.
- 4.2 -SOP Template File - a Microsoft Word document which contains a specific structure which is used as a template for SOPs. The document contains all of the styles and formatting necessary for proper presentation of the SOP.
- 4.3 Style - in Microsoft Word, a group of formats identified by a unique name. Styles are used to ensure consistent formatting within a document. The primary styles used in the SOP Template File are Headings 1, 2, 3, 4 and 5.

- 5.3 About the Template. Upon opening the template, a new document is created which contains the following information and items:
- 5.3.1 Customized style sheets;
 - 5.3.2 Pre-formatted headers and footers;
 - 5.3.3 Fields for automatically inserting common items such as SOP Title, ISO Number, Author, Effective Date and Approval List. These fields are indicated by a gray shaded font, and should not be edited on-screen. They are changed using the Summary Info. dialog box and the update fields command;
 - 5.3.4 Pre-formatted sections for writing the SOP. These sections are arranged as follows with the specified information:
 - 5.3.4.1 Purpose: This section should give a brief description of the overall reason that the procedure exists.
 - 5.3.4.2 Scope: This section may contain two sets of information. First, it should designate who or what areas of operations are affected by the SOP. Second any appropriate limitations of the applicability of the SOP should be identified here.
 - 5.3.4.3 Materials: All major items required to properly execute the procedure should be listed.
 - 5.3.4.4 Definitions: Give a brief definition of all major terms which are used by the SOP.
 - 5.3.4.5 Procedure: This section contains the actual substance of the SOP. The procedure should be described in a series of simple, short instructions.
 - 5.3.4.6 Flow Chart: This is an optional section which should be used if a flow chart diagram helps to summarize the procedure.
 - 5.3.4.7 References: Use this section to list any sources of information which were referenced in the SOP.
 - 5.3.4.8 Attachments: List any documents which exist separately from the SOP itself.

5.4 Set Up Boiler-Plate Fields for SOP. The template makes use of the Summary Info. dialog box to organize common body and header information into a single place. The following steps should be performed immediately after opening the document for the first time, and anytime thereafter when the designated information needs to be changed.

5.4.1 Select File>Summary Info. from the top menu bar.

5.4.2 The Summary Info. dialog box will appear as shown in Figure 5-2. Enter the Title, Effective Date, Author, ISO Number (without the date) and Approval List in the locations indicated below. Tab between fields. Click OK when done.

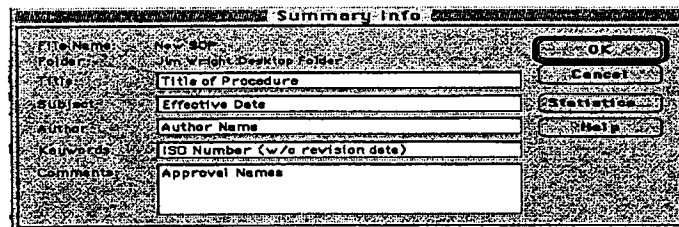


Figure 5-2- Summary Info Dialog Box

5.4.3 Select entire document by typing ⌘a (Command-a). Then press function key F9. This updates all of the fields in the document with the information from the Summary Info. dialog box.

5.5 Save File. Now is a good time to save the document. Use the File>Save As command and name the document using the entire ISO Number as the filename.

5.6 Write SOP. Construct the SOP by placing all of the relevant information into the pre-formatted sections as described above. Utilize the existing style sheets to help separate and clarify groups of information and procedures. Use the following guidelines for organizing each section and using the style sheets.

5.6.1 Purpose - The purpose should be typed in Normal style.

5.6.2 Scope - The scope should be typed in Normal style.

5.6.3 Materials

5.6.3.1 The initial introductory statement for this section should be in Normal style.

5.6.3.2 Immediately below the introductory statement, type each material item in Heading 2 style.

5.6.3.3 Generally, just the name of the material needs to be listed. In some cases, however, it may be advantageous to give a brief description or specification for an item. In these cases, separate

the item name from the description using a spaced hyphen (-).

5.6.3.4 Hard return after each entry except the last one.

5.6.4 Definitions

5.6.4.1 Definitions should be typed in Heading 2 style.

5.6.4.2 Manually underline the name of the term to be defined (use the underline button).

5.6.4.3 Place a spaced hyphen (-) between the name and the actual definition.

5.6.4.4 Hard return after each entry except the last one.

5.6.5 Procedure

5.6.5.1 Use Heading 2 style for major instructional steps.

5.6.5.2 Use Heading 3, Heading 4 and Heading 5 styles to further subdivide steps within each major step.

5.6.6 Flowchart

5.6.6.1 The initial introductory statement for this section should be in Normal style.

5.6.6.2 Immediately below the introductory statement, insert the flowchart (e.g., from MacFlow or similar drawing software).

5.6.6.3 Format the flowchart using the -Figure style.

5.6.7 References

- 5.6.7.1 The initial introductory statement for this section should be in Normal style.
- 5.6.7.2 Immediately below the introductory statement, list the references in the order which they are referred in the document.
- 5.6.7.3 Format the references using the Numbered List style.

5.6.8 Attachments

- 5.6.8.1 The initial introductory statement for this section should be in Normal style.
- 5.6.8.2 Immediately below the introductory statement, list the attachments in the order which they are referred in the document.
- 5.6.8.3 Format the attachments using the Bulleted List style.

5.6.9 General Guidelines

- 5.6.9.1 All major section titles (e.g., PURPOSE, SCOPE, etc.) are in Heading 1 style.
- 5.6.9.2 To remove a major section that is not applicable to the SOP being created:
 - 5.6.9.2.1 Use the cursor to highlight the entire contents of the section, beginning at the first word of the title and ending at the last paragraph marker within the section.
 - 5.6.9.2.2 Type ⌘x (Command-x) to delete the highlighted text.
- 5.6.9.3 Generally, it is not necessary to add space between sections or subsections using paragraph returns. If additional space is necessary, however, then format the hard return as Normal style to avoid additional numbering of the blank space.
- 5.6.9.4 Before printing or saving document, it is a good practice to first update fields (see section 5.4.3).

6. REFERENCES

The following references were used to help develop this procedure:

1. Microsoft Word template document: *SOP Template.10/96*
2. Microsoft Word User's Guide, Version 6.0

CAE STANDARD OPERATING PROCEDURE

CLIENT INTERACTION

ISO NUMBER: FLD-1

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

The purpose of this document is to outline a series of guidelines to follow when interacting with our Clients.

The interaction between the field test leader and the client is probably the most important relationship that takes place during any job. This is sometimes the only face to face contact that takes place between CAE and our clients. This relationship will leave a lasting impression on the client concerning CAE's capabilities and professionalism.

We should do everything possible, within reason, to satisfy the clients requests in the field. Only a few jobs go according to plan, therefore the test leader needs to be flexible to the clients needs. However, this does not mean that we should sacrifice the quality of our work or sacrifice safety issues.

Also, even though we are paid by our clients we should never allow them to take advantage of that situation. We need to accurately follow the methods we are performing. At no time should we allow the client to influence the way that we test.

The following is a rough guide of how a typical job should be run concerning client interaction:

1. When arriving on site, ALWAYS find the client and introduce yourself before any setup begins. There may be some safety issues that you need to be aware of before setting up. There also may be a change in plans about what units need to be tested. Find out what unit that you will be starting on. Find out what state people will be on site and when. Inform the client about the size of the field crew and where we will be working. Find out any safety concerns that we should know about (radio communications, Gaitronics, etc.). Go over the schedule to make sure that everyone is on the same page about the testing that will be done. Find out who will be making the final decision about testing or not testing. Inform the client about the hotel you're staying at in case there are any last minute changes. Find out where the client is staying.
2. Before leaving on the first day of setup. Inform the client about the status of our tests for the next day (will we be ready). Ask about the starting time for the next day. Inform the client of any problems or safety concerns that have developed during the day. Tell them that we're leaving the site.
3. At the beginning of the first and every following day of testing; check to see when they will be ready to start. Inform them of our scheduled start time. (Do not lie but give an honest estimate of our start time. Inform them on any delays that we foresee.) Discuss the days testing and coordinate any process changes that may need to be made (i.e. soot blows, CEM calibrations, etc.).

4. During testing, inform the client about our start and stop times for each test. Inform client about any problems (bad leak check, blown runs). Ask about any odd process variations.
5. At the end of the day, inform client of the last stop time. Ask client about the following days testing. Give client preliminary results if he or she asks about them. Only give data and information concerning the field job to the client and no one else. Data should be distributed through the client and then they can decide what to do with it afterward. Never give data directly to outside contractors or state people.
6. At the end of the field job, ask client about testing (how did everything go), inform client of any delays that might have been incurred. Make sure to have client sign time sheet. Ask about any special details that the client wants included in the report. Ask if there will be any additional testing coming up in the future.

Important things to remember: all clients are not alike, all jobs are not alike, and what we do for some clients may not apply to other clients.

Some common sense practices: do not let client see any CAE faults (incomplete data sheets, messy locations, messy trailers, anything that does not present a professional image)

Fill out change of scope form whenever client asks for additional testing, always have them sign it.

Always make client sign field time sheet. Give them a copy .

Never argue with client, if a resolution cannot be reached, call project manager and have them discuss problem with client. Always keep the field relationship as cordial as possible.

Never discuss price issues with the client, always defer that discussion to the Project Manager.

Never negotiate any CAE delays with the client. Never give client any credit for our delays, leave all negotiations to project manager.

CAE STANDARD OPERATING PROCEDURE

FIELD CREW/CLIENT DINNERS

ISO NUMBER: FLD-2

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

EFFECTIVE DATE: November 1, 1996.

REVISION: 1.0.

ISO NUMBER: NA.

1.0 PURPOSE

This procedure is designed to provide guidelines for field crew/client dinners on CAE field projects. A crew/client dinner can take place to reward a crew for an outstanding project performed under very difficult circumstances, to foster teamwork and solidarity among personnel who have widely varying responsibilities, to allow the crew to interact as peers, to encourage teamwork in a situation where it is vital and to boost crew morale. A crew/client dinner can also take place to strengthen, solidify or enhance a client relationship.

2.0 SCOPE

Any personnel on a field project are affected by this procedure.

3.0 MATERIALS

The following materials are needed to properly execute this procedure:

- expense report rules and expense reports.

4.0 DEFINITIONS

Clean Air's Alcohol Policy - The alcohol policy is presented under Rules of Conduct in the Clean Air Engineering 1996 Employee Handbook, "The possession or use of alcohol on company premises, on company work sites, including client locations, in company owned or leased vehicles, in any private vehicles parked on company owned property, or during work hours is strictly prohibited. Use of alcohol on meal break is not approved."

5.0 PROCEDURE

5.1 A field crew/client dinner may occur when any of the following circumstances exist:

- Outstanding project with unusually long days, extreme weather and/or difficult test locations.
- High dollar/high profile project.
- Potential to improve the client/CAE relationship.
- Projects at least one week in length.

- 5.2 A field crew/client dinner may not occur when any of the following circumstances exist:
- CAE places a moratorium on all outings due to financial situation.
 - The Project Manager and Test Leader decide the estimate for the project can not withstand a crew dinner. It is possible that the project is already way over budget due to extra shipments, flying people in, etc. It is also possible that the project was underbid just to obtain the project.
- 5.3 A field crew/client dinner may take place only when the Test Leader decides it is appropriate and will be present to pay for the expense as a representative of CAE. A field crew/client dinner may not take place more than once per project. The Test Leader must discuss with the Project Manager whether or not the estimate for the project can withstand a dinner. The entire field crew is welcome to participate. The maximum allowed for dinner for each crew member is \$30.00 including tax and gratuity. If the bill including tip and gratuity is over \$30.00 per crew member, the crew members spending more than their limit must contribute to the bill at that time. Clean Air Engineering will not pay for alcohol. The client, if present, does not have a dollar limit.
- 5.4 If the Test Leader buys dinner for the crew, the crew members are not allowed a dinner expense for that day. The dinner expense is \$10.00. Deduct the dinner expense for that day by leaving the dinner portion of the expense report blank. Test Leaders need to indicate on their receipt which crew members attended the meal and whether or not a client was present.

6.0 REFERENCES

Expense report rules updated from The Clean Air Engineering Business Leader's Manual of Procedures, January 15, 1991 and the Clean Air Engineering 1996 Employee Handbook.

CAE STANDARD OPERATING PROCEDURE

FIELD TIME SHEETS

ISO NUMBER: FLD-3

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

The following procedure is to give guidance and to allow for uniformity on the completion of the job field time sheet. The purpose is to allow a client to have more accurate invoices and a clearer understanding of costs.

The field time sheet is the job leader's responsibility until relinquishing it to the project manager. Field time sheets should have only one job number. If two jobs are in one mobilization, project hours need to be broken down by the test leader as best as possible.

Travel time is the time from the office to the job site not including meals. If flying, travel time starts one and a half hours before the flight is scheduled to leave unless an excessive drive is necessary. For example, driving from the job site in the middle of Nevada to the closest airport in Las Vegas will be longer than an hour and a half. When driving, not all people driving to site will necessarily have the same travel time. Truck travel may be slower. Travel time will be given daily if travel from hotel to job site is over 30 minutes, and the travel time given will be the additional time over 60 minutes per day. Mileage will be given when the distance from the hotel to the job site is over 20 miles.

Field time sheets should be done daily and posted if possible to allow all job personnel to dispute or correct it.

In general, a field worker is given 8 hours at a minimum for Monday to Friday work unless he or she is late or has been released from the job site and is able to travel home. For example, if it takes him or her 4 hours to return from Indianapolis on Friday morning from a job that finished Thursday night, he or she can stay in the office for 4 hours or take ATO for 4 hours.

"Changes in scope of work and /or delays" section of the time sheet

The importance of completing this accurately cannot be emphasized enough. It is this section that a project manager relies upon to complete the invoice. It is also important that clients fully understand any changes or delays so they will better understand the invoice. The jobs that are not per diem should give hours worked that were not expected in the jobs costing due to Client delays.

For example:

- Client process problems
- Power problems due to Client failure to provide agreed upon power
- Time spent removing port caps
- Time spent repeating tests at the client request

CAE delays and problems will not be noted on this time sheet.

It is imperative that the time sheet be signed by the client before the crew leaves the site. There can be no excuses for not having the time sheet signed. If the client leaves earlier than the crew, then have the client sign the time sheet up to the present time. If the client does not agree with the delays listed on the bottom, then have the client write a statement that he or she does not agree with the delays.

The time sheets main purpose is to document the amount of the time the crew spent at the site. This will hopefully prevent any misunderstandings when the invoice arrives.

Make sure the client receives their copy of the time sheet after they sign it.

All hours will be recorded to the nearest half an hour.

CAE STANDARD OPERATING PROCEDURE

RESERVED

ISO NUMBER: FLD-4

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

nn #	
Date	
Job Number	Job Title
Crew Summary Job Leader: Location Leaders: Tech Technicians: CFM Technicians: Trainees: CAF Observers: Others:	
Initial Client Contact Information <div style="background-color: #cccccc; height: 40px;"></div>	
Job Plan Exceptions <div style="background-color: #cccccc; height: 40px;"></div>	

nn #								
Date								
Job Number	Job Title							
Tour Summary Safety Concerns Housekeeping Concerns Unexpected Findings at c								
Set-Up Notes <table border="1"> <tr> <td>General Info</td> <td>Ship List</td> </tr> <tr> <td>Malfunctioning Equipment</td> <td rowspan="4"><div style="background-color: #cccccc; height: 40px;"></div></td> </tr> <tr> <td>Missing Equipment</td> </tr> <tr> <td>Wrong Equipment</td> </tr> <tr> <td>Unforeseen Equipment</td> </tr> </table>		General Info	Ship List	Malfunctioning Equipment	<div style="background-color: #cccccc; height: 40px;"></div>	Missing Equipment	Wrong Equipment	Unforeseen Equipment
General Info	Ship List							
Malfunctioning Equipment	<div style="background-color: #cccccc; height: 40px;"></div>							
Missing Equipment								
Wrong Equipment								
Unforeseen Equipment								
Location/Plant/ Facility Problems Encountered During Set-Up <div style="background-color: #cccccc; height: 40px;"></div>								

dd.#	
Date	
Job Number	Job Title
DAILY LOGGING - Record for every day General Logging Arrival/Departure Times Delays Specific Problems Process Changes Shift Changes Other Applicable Info.	
Time Stamps	Ship List for Day <div style="background-color: #cccccc; height: 20px;"></div>
	Day-In-General <div style="background-color: #cccccc; height: 20px;"></div>

pg.#	
Date	
Job Number	Job Title
Other Helpful Information <div style="background-color: #cccccc; height: 100px;"></div>	

CAE STANDARD OPERATING PROCEDURE

JOB LOG CONSTRUCTION

ISO NUMBER: FLD-5

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

Purpose: The purpose of this document is to provide guidance and establish the minimum requirements for a CAE Job Log. It is intended to give the Test Leader/Field Project Manager a framework for the recording of pertinent job related information.

Procedure: The following procedure is a general outline of how data and information should be organized in order to ensure the generation of a complete and comprehensive CAE Job Log

1. **Page Numbers:** Each page of a CAE Job Log Book should be numbered by the book's manufacturer
2. **Page Heading:** The page heading should always contain the date, the job title, and the job number. This heading shall appear on each and every page of the Job Log.
3. **Crew Summary:** The crew summary should appear on at least the first page of the Job Log. This summary should be comprised of the names of each CAE technician and CAE engineer assigned to the filed portion of the project. If applicable, special designations should be defined such as, location leaders, lab technicians, and monitor technicians. Also if applicable, locations should be assigned and manpower distribution recorded. Any trainees, specialist, observer, sub-contractor or other agent of CAE should be listed in the Crew Summary section along with their purpose for attending. Also, Identify who you are in this section, the author of the job log sometimes goes unnoticed.
4. **Initial Client Contact:** The first page of the job plan should contain a synopsis of the initial meeting or contact with the on site client. Their initial instructions or other information conveyed at this meeting should be carefully documented.
5. **General Job Plan Exceptions:** General Job Plan Exceptions should also appear on the first page of the Job Plan. These types of exceptions would include different or additional site contacts and a description of their role both at the plant or facility and on the project, any problems with travel directions or travel accommodations, or any other significant discrepancies between the CAE Job Plan and the expectations of the Test Leader/Field Project Manager.
6. **Tour Summary:** Any potential location specific problems or complications should be noted in the Tour Summary section of the Job Log. These would include any safety concerns and their resolutions, general condition or housekeeping concerns, drastic differences in expected and actual duct conditions or sizes, ambient conditions, or other such scenarios. Nomenclature for the name of each location along with any aliases should also be noted, as well as any process or operational information that may be helpful in further developing or improving upon the Description of Installation located in the Job Plan.
7. **Set-up Notes:** Any pertinent information involving the initial dispatch and set-up of equipment should be included in this sections(s). Equipment problems should be organized under the following headings:
 - **Malfunctioning Equipment.** This would include any equipment received broken or in a state of disrepair, or equipment that does not meet the expectations of the Test Leader or user.

- **Missing Equipment.** This would include any equipment helpful or necessary for the execution of the test program that was not received on the job site.
- **Wrong Equipment.** This would include any excess or inappropriate equipment received.
- **Unforeseen Equipment.** This would include any equipment that is missing due to a miscalculation, additional test request by the client or similar add-on, or other situations that may have arisen on site that are outside the scope of the original Job Plan.

Any equipment problem listed should be accompanied by a description of what was done to resolve it and if applicable, any ideas or information that may be helpful to avoid similar problems in the future. A **Ship List** should also be included in this section that identifies all equipment that required immediate shipment from CAE offices or equipment to be shipped from the job site back to CAE. Means of shipping should be noted as well as Airbill numbers if available.

Any location, plant or facility specific problems that could or will affect the test program in any way should be called out in this section. These problems would include equipment clearance, extraordinary duct pressure or temperature, power problems, or other such scenarios. Any problem listed should be accompanied by a description of what was done to resolve it as well as an accounting of the time and manpower required.

8. **General Logging:** All significant events should be logged, by entering the time in the left hand margin next to a brief description of the event as it occurs. A list of events typically would include arrival and departure times, all delays, specific problems, process changes or shifts, or other information for which a time stamp is appropriate.
9. **Ship Lists:** A Ship List should be recorded every day for any samples, supplies, equipment, or other necessary items that have required shipping. The Ship List should include such information as what was shipped, carrier or freight company, number of pieces, terms or type of service and consignee name and address.
10. **Day In General:** This is the final entry of the day. This section allows the author to assess the days happenings and accomplishments. It is the catcher of stray thought. The forum for one to sum up the day, to celebrate the victories and ponder the defeats. Ideas and creativity are valuable assets and should be put to pen here.
11. **Helpful Information:** This is the spot to jot down helpful information for the next crew that comes out to this site. Fed Ex locations, stores that sell vermiculite and nearby hardware stores would be examples of this kind of information.

Two copies of the job log should be turned into the project manager. One copy goes into the Field Data Binder and the other copy is included for reports. The Project Manager will give one copy to reports and give a copy of the equipment problem section to Dept. 67 if applicable.

The job log can be written electronically if the test leader chooses. However, when the project is complete the test leader must print out one copy of the log and then initial each page.

Finally, it should be noted that the Job Log is a legal document. Our clients can have access to these logs if they desire. Therefore, they should be written in a professional manner. Comments about the client should not be included here unless it was critical in the outcome

of this job. Also, non-work related comments such as where the crew is going to dinner or tourist spots should not be included here.

nn. #	
Date	
Job Number	Job Title
Crew Summary	
<div style="border: 1px solid black; padding: 2px;"> Job Leader: Location Leaders: Lab Technicians: CEM Technicians: Trainees: CAE Observers: Others: </div>	
Initial Client Contact Information	
<div style="border: 1px solid black; height: 40px; background-color: #f0f0f0;"></div>	
Job Plan Exceptions	
<div style="border: 1px solid black; height: 40px; background-color: #f0f0f0;"></div>	

nn. #	
Date	
Job Number	Job Title
Tour Summary	
<div style="border: 1px solid black; padding: 2px;"> Safety Concerns Houskeeping Concerns Unexpected Findings etc. </div>	
Set-Up Notes	
<div style="border: 1px solid black; padding: 2px;"> General Info. Malfunctioning Equipment Missing Equipment Wrong Equipment Unforeseen Equipment </div>	<div style="border: 1px solid black; padding: 2px; width: 60px;"> Ship List <div style="border: 1px solid black; height: 40px; background-color: #f0f0f0;"></div> </div>
Location/Plant/Facility Problems Encountered During Set-Up	
<div style="border: 1px solid black; height: 40px; background-color: #f0f0f0;"></div>	

pq. #	
Date	
Job Number	Job Title
DAILY LOGGING - Record for every day	
Time Stamps	<div style="border: 1px solid black; padding: 2px;"> General Logging Arrival/Departure Times Delays Specific Problems Process Changes Shift Changes Other Applicable Info. </div>
	<div style="border: 1px solid black; padding: 2px;"> Ship List for Day <div style="border: 1px solid black; height: 20px; background-color: #f0f0f0;"></div> </div>
	<div style="border: 1px solid black; padding: 2px;"> Day-In-General <div style="border: 1px solid black; height: 20px; background-color: #f0f0f0;"></div> </div>

pq. #	
Date	
Job Number	Job Title
Other Helpful Information	
<div style="border: 1px solid black; height: 150px; background-color: #f0f0f0;"></div>	

CAE STANDARD OPERATING PROCEDURE

FIELD SAFETY ISSUES

ISO NUMBER: FLD-6

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

The purpose of this document is to address field safety issues. In general, the test leader has sole authority over all safety issues. If at any time the test leader feels it is not safe to test, he or she has the authority to stop testing until it is safe to continue. Some of these issues are addressed below.

- Hours that will be worked by field personnel without a break
- Minimum hours off before the next shift of hours may start
- Hot and Cold Pay
- Stack Pay
- Hazardous Pay
- Weather constraints

Hours that will be worked by field personnel without a break

- No test crew may plan to work more than 12 consecutive hours under ideal conditions.
- After 16 consecutive hours, the test run in progress will be finished, but no further test runs may be started.
- If the plant is in excess of 30 minutes from the hotel, total drive and work time may not exceed 16 hours. This may mean cutting the day short to avoid driving while fatigued.

Minimum hours off before the crew may begin another shift

- The crew must be given 8 hours at the hotel. This does not include travel from the plant to the hotel. Given the fact that an hour is usually needed before and after testing for the day, and a half hour travel each way is normal, this would entail 11 hours from the end of a test to the start of another.

Hot and Cold Pay

- Hot and cold pay is equal to an additional 5 dollars an hour paid for periods in extreme conditions. Cold pay shall be received when wind chills are below 0° F. Hot pay shall be paid when the Heat index is over 100° F.

Stack Pay

- Stack pay is occurred when a ladder climb in excess of 100 feet is done. This bonus will be paid in a lump sum per day. Anyone who climbs (with or without a squirrel, with no other alternative) over a 100 feet will receive \$50 for that day. Climbs over 150 feet will receive \$75. Climbs 200 feet and above will receive \$100. This pay will only be given once per day not for every climb.

Hazardous Pay

- When an employee is required to have a forty hour hazardous materials certification to perform a task, the employee shall be paid an additional 5 dollars per hour for the duration of that task, only if dressed out in Level A, B or C.
- If the test leader decides a location or task is hazardous, actions must be taken by the client, at his expense to make it safe, or no testing will be conducted there.

Weather Conditions

No one may work outside during the following conditions:

- Lightening is visible.
- Tornado warning is issued.
- Wind chills are below -25° F without any shelter
- Heat index is above 120° F
- Strong Winds

Other miscellaneous policies

- Use safety tape and Men working above signs when work is being conducted above eye level.
- Tie off when working off the ground without a railing.
- Hard-hats, safety glasses, steel toed shoes at all times.

CAE STANDARD OPERATING PROCEDURE

TEST LEADER AUTHORITY

ISO NUMBER: FLD-7

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

The purpose of this document is to outline the authority given to a test leader.

The test leader will have the final authority over all issues that occur in the field.

All issues should be discussed with the project manager before any decisions are reached, but it is the ultimately the test leaders decision. The following items are guidelines to the limits of his authority.

A test leader can send home any employee for gross insubordination and acts of unprofessional behavior. Examples are listed below.

- Showing up impaired for work (drunk, hungover, etc.)
- Refusal to perform reasonable requests
- Altering data
- Cheating on methods
- Fighting

If an employee is sent home, he or she will wait at the hotel until the test leader can arrange for transportation back to the office. The test leader will inform the person why they are being sent home and will document the reasons for this action. The employee will have the opportunity to respond to the behavior to the test leader as well to their business leader.

Any employee sent home by the test leader will be automatically suspended for a week without pay.

At the end of the job, the test leader will meet with the employee's business leader to discuss the problem and any further action.

The test leader has the authority to review field crew members. (Both positively and negatively)

The test leader has final authority over additional testing requested in the field. Safety will be the primary consideration.

Minor infractions shall be dealt with in the following manner. (Minor occurrences are showing up late, unprofessional conduct, horseplay, sleeping on the job, etc.)

- A verbal warning will be issued for the first infraction
- Written documentation will be required for the second infraction
- After the third infraction the employee will be sent home following the guidelines listed above.

The test leader can have additional people sent out on the job if necessary.

The test leader will have sole authority as to the safety of the testing. He or she can call testing off at any time if it is unsafe.

CAE STANDARD OPERATING PROCEDURE

TEST LEADER REQUIREMENTS

ISO NUMBER: FLD-8

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

Purpose: It is the purpose of this document to outline a series of requirements which must be met for a tester to achieve the position of test leader.

The tester shall have demonstrated, through on the job experience and a technical review, that they have the required knowledge of the common test methods and processes with which they work the most. They must also have shown the ability to learn and adapt to a variety of uncommon methods and processes.

This process will be follow a structured path from a T4 to a P4. All employees will start off as a T4. After they have been through the basic field training they will become T3's. After they have demonstrated the ability to run a location (setup unistrut, know how to recover all trains) they will become T2's.

To jump from a T2 to a T1 they must have demonstrated that they can perform lab and CEM work. All increases in levels will be approved by the technical review board. All increases in levels will be accompanied by an increase in pay.

When the tester has demonstrated these abilities, he or she shall then begin to lead jobs, on a trial basis, with another test leader as backup. During these trial jobs, he or she shall be judged by the backup test leader as to whether, or not, they have the following qualities:

- The ability to handle the increased stress that is involved with this position
- The ability to lead others (i.e. delegation of tasks, authority toward his peers)
- The necessary ethics for the position.
- Communication skills needed to discuss problems with the client.
- A knowledge of the legal aspects of ownership of data.
- Practice of necessary safety guidelines.

If the back-up test leader feels that the trial test leader has demonstrated these requirements he or she shall notify the business leader of which traits have been fulfilled. If the back-up test leader feels that the trial test leader has failed in any of these traits, it is imperative that this is discussed between the two.

If the tester has a problem meeting any of these requirements, he or she should discuss possible seminars with his business leader.

When the tester has shown all of these qualities and has led a minimum of three jobs with a backup test leader, they may then lead jobs on their own.

The test leader will start leading small jobs (easy testing and small crews) at first. Then they will advance into larger jobs as a P3. When they are capable of handling the largest jobs with the most complicated methods (i.e. trial burns) they will become P2's. To become a P1 you must be published in this field.

CAE STANDARD OPERATING PROCEDURE

TEST LEADER RESPONSIBILITIES

ISO NUMBER: FLD-9

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

Purpose: The purpose of this document is to list various responsibilities which fall to the test leader. It is the test leaders responsibility to make sure these tasks are carried out, not necessarily to do it themselves.

- Ice: The test leader must decide how much ice will be needed for the day, and ensure that it is purchased before testing.
- Communication: It is imperative that the test leader is in communication with all client contacts and agencies involved in the test. Test times must be coordinated with plant operating conditions.
- Results: The test leader must know what results the client wants to see, and when. They must also know how accurate the results are when given early (i.e. preliminary weights done on site).
- It is the test leaders decision to assign crew members to their work locations. A toolbox meeting will be held everyday to talk about the day's testing.
- It is the test leaders decision when the crew members must be on site and when they can leave.
- It is the test leaders decision when packing up or moving equipment may begin.
- It is **not** the test leaders responsibility to make restaurant reservations, wake up calls, or act as taxi driver for the crew.
- It is the test leaders responsibility to reprimand a crew member if they are not performing their job satisfactorily. (i.e. tardiness, sleeping on the job, etc.)
- It is the test leader's responsibility to organize the field data binder
- It is the test leader's responsibility to make sure the data is accurate and complete
- It is the test leader's responsibility to make sure everyone in the crew has travel arrangements back home
- It is the responsibility of the test leader to call the appropriate equipment coordinator before the end of the job to see if any equipment needs to be sent back immediately
- It is the responsibility of the test leader to send back any equipment, that is not being used or will not be needed, back to the main office

CAE STANDARD OPERATING PROCEDURE

CALIBRATION OF BALANCE

ISO NUMBER: CAL-1

EFFECTIVE DATE: Pending

AUTHOR: Dept.68

APPROVED BY: Pending

I. CALIBRATION OF BALANCE GA110:

- A. Turn scale on by pressing the on/off button.
- B. Level the scale
 - 1 Adjust the knob underneath the balance so that the liquid bubble is in the middle of the circle.
- C. Zero the balance by pressing the rezero button.
- D. Take out the calibration kit which is located in the right hand drawer of the desk.
- E. Open the plastic sliding doors of the balance
- F. Take out the .5000 gram weight from the calibration kit and place it on the weighing plate of the balance.
- G. Close the doors of the balance and wait for the balance digital reading.
- H. If the number is +/- .0001 mg off from the actual weight then record that weight in the calibration log for the GA110 balance and go to step L.
- I. If the number is over or under the estimated allowance then go to step J.
- J. To calibrate balance GA110, flip the switch on the right side of the instrument to CAL.
- K. After about fifteen seconds the digital display will blink CAL several time and stop. At that point the calibration is complete and the switch should be put back on 'weighing' and repeat steps A-H.
- L. Take out the 1.000 gram weight from the calibration kit and place it on the weighing plate of the balance and repeat steps G-K.
- M. Calibration is complete once steps A-L are performed.

II. CALIBRATION OF GA200D:

- A. Turn scale on by pressing the on/off button.
- B. Level the scale
 - 1 Adjust the knobs underneath the balance so that the liquid bubble is in the middle of the circle.
- C. Zero the balance by pressing the rezero button.
- D. Take out the calibration kit which is located in the right hand drawer of the desk.
- E. Open the plastic sliding doors of the balance
- F. Take out the 1.0000 gram weight from the calibration kit and place it on the weighing plate of the balance.
- G. Close the doors of the balance and wait for the balance digital reading.
- H. If the number is +/- .0001 mg off from the actual weight then record that weight in the calibration log for the GA200D balance and go to step L.
- I. If the number is over or under the estimated allowance then go to step J.
- J. To calibrate balance GA200D, flip the switch on the left side of the instrument to CAL 200.
- K. After about fifteen seconds the digital display will blink CAL several times and stop. At that point the calibration is complete and the switch should be put back on 'weighing' and steps A-H should be repeated.
- L. Take out the 100.000 gram weight from the calibration kit and place it on the weighing plate of the balance and repeat steps G-I.
- M. Calibration is completed once steps A-K are performed.

III. CALIBRATION OF BALANCE AJ100

- A. Turn on balance by pressing the on/off button.
- B. Zero the balance by pressing the rezero button.
- C. Take out the calibration kit which is located in the right hand drawer of the desk.
- D. Open the balance door by lifting upwards.
- E. Take out the 1.0000 gram weight from the calibration kit and place it in the middle of the

CALIBRATING A BAROMETER WATCH

ISO NUMBER: CAL-2

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

SCOPE: Anyone using a barometer watch in the field

All barometer watches must be calibrated against the mercury barometer in the lab. This must be done at least every 90 days and whenever the watch is readjusted. The calibration must be recorded in the logbook provided in the same lab.

Use the following procedure:

Record the date, your name, reference barometric pressure & ambient temperature.

Look up the temperature correction factor in the table in the front of the log book.

Apply the temperature correction factor to the reference Bp as follows:

$Bp \text{ corrected} = Bp + (Bp \times \text{correction factor})$ note: the correction factor is (-)

Subtract 0.01"Hg from the result to correct for the Coriolis acceleration. This is the final corrected reference Bp, record this in the book.

Convert the corrected Bp to millibars by using 0.02953"Hg/mB.
(Divide Bp by 0.02953).

Record the result in the book.

Read & record your indicated watch barometric pressure in the book.

Record the difference.

If the difference is more than $\pm 3\text{mB}$, adjust your watch until it is in agreement.

If your watch has not been previously calibrated, run through the procedure once to make the required adjustments. The watch must agree to within $\pm 3\text{mB}$ (0.1"Hg) of the reference barometer to be considered in calibration.

This information will be included in reports as proof of calibration of field barometers. Calibrations must be completed as early as possible.

ADDITIONAL NOTE: When several barometers are on a project, pick one as the source of all barometric pressure readings at all test locations for that project. The only acceptable alternative is to compare the barometers at the site to a primary standard and adjust them to insure agreement.



Clean Air Engineering

Standard Operating Procedures

Equipment Calibration

Meter Cal./Critical Orifice
Field Project Management
SOP I.D.: CAL-3
CTPM Section J Page 1

Effective Date: 11/1/95

Drafted By: JE Wright

Approved By: Process Owners

POST-TEST CALIBRATION OF A METER BOX USING A CRITICAL ORIFICE

1.0 PURPOSE

The purpose of this procedure is to provide instructions for calibrating a meter box console in the field using a critical orifice.

2.0 SCOPE

These procedures are written explicitly for post-test verification of the dry gas meter calibration factor. However, they may also be used to verify the overall integrity of the metering system at any time during a test job (e.g., during equipment set-up).

3.0 MATERIALS

The following materials are needed to properly execute this procedure:

- Critical Orifice Set
- Standard Data Sheet CS 005
- Meter Box Console with Quick-Connect sample inlet fitting
- Meter Calibration Card
- (Optional) Standard Worksheet or Calculator program for performing calculations

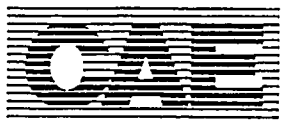
4.0 DEFINITIONS

Dry Gas Meter Calibration Factor (Y) - an expression of the relative accuracy of a dry gas meter. This factor is dimensionless, and typically has a value of 1.00 ± 0.02 . When written as Y_d , this parameter represents the calibration factor as determined from a full-test calibration of the meter against a wet test meter. When written as Y_i , it represents the calibration factor of the i^{th} run determined using a critical orifice.

Critical Orifice - A device used to verify the calibration of a dry gas meter. The device consists of a small bored plate orifice assembled into a quick-connect fitting which can be plugged directly into the meter box sample port. The pressure drop across the orifice is used to determine the flow rate through the assembly.

Critical Orifice Set - A set of five critical orifices of varying diameters to allow a range of flow rates for meter box calibration purposes.

Critical Pressure - As the flow rate of gas through a critical orifice increases, so does the pressure drop across the orifice. At a certain pressure drop known as the critical pressure, the flow rate becomes constant - pressure drops higher than the critical pressure will not result in more flow. This critical pressure drop is normally at least 53% of the barometric pressure. Also called critical vacuum.



Clean Air Engineering

Effective Date: 11/1/95

Standard Operating Procedures

Equipment Calibration

Drafted By: JE Wright

Meter Cal./Critical Orifice
Field Project Management
SOP I.D.: CAL-3
CTPM Section J Page 2

Approved By: Process Owners

5.0 PROCEDURE

- 5.1 Perform a positive pressure leak check of the meter box (see appropriate procedure). The leak check is valid if the manometer shows no visible movement in 1 minute.
- 5.2 Pick a critical orifice from the set that will produce a ΔH close to that of the test conditions.
- 5.3 Plug the orifice into the meter inlet.
- 5.4 With the meter box running, leak check by plugging the inlet to the orifice assembly. The leak check is valid if no visible movement of the meter is seen for 1 minute.
- 5.5 Adjust the pump by opening the course valve and/or closing the fine valve until the ΔH stabilizes and the orifice reaches its critical pressure (vacuum). If the critical vacuum cannot be reached, then a smaller orifice must be used.
- 5.6 Allow the pump to run for about 10 minutes at the critical vacuum so that the pump and meter temperatures stabilize.
- 5.7 While the meter box is warming up, fill in the initial data for the calibration on the standard data sheet CS 005.
- 5.8 After at least 10 minutes, begin the calibration. Reset the elapsed time meter to zero, and record the meter volume, elapsed time (equals 0), meter inlet and meter outlet temperatures onto the first line of the data sheet. The meter volume must be read on the fly at precisely the time designated as the start time (elapsed time = 0).
- 5.9 At five-minute (minimum) intervals, record the meter volume, elapsed time, inlet and outlet meter temperatures, ambient temperature, meter box ΔH , and meter vacuum. Once again, the meter must be read on the fly at precisely the time at which the elapsed time was recorded. Allow a minimum of 5 minutes between readings. More is OK, as long as the meter is read exactly at the time of the recorded time.
- 5.10 Repeat step 5.9 two more times for a total of three readings and at least 15 minutes of operating time.
- 5.11 If a spreadsheet or calculator program is available, input the data from the data sheet directly into the appropriate spreadsheet (or equivalent calculator program) to calculate the individual Y_i factors for each of the three 5-minute runs. Use these results to complete the data sheet, then proceed to Step 5.18.

If no automated program is available, the calculations may be done manually by following steps 5.12 through 5.17 below.



Standard Operating Procedures

Clean Air Engineering

Equipment Calibration

Meter Cal./Critical Orifice
Field Project Management
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Approved By: Process Owners

- 5.12 Calculate the net run time for each run by subtracting the elapsed time at the end of the run by the elapsed time at the end of the previous run. Record the net run time in decimal minutes to the nearest 0.1 minute. Note: If the elapsed time was recorded in minutes:seconds, convert to decimal minutes by dividing the seconds value by 60 and adding this value to the minutes value.
- 5.13 Calculate and record the average meter temperature for each run by averaging the inlet and outlet temperature readings recorded before and after each five minute period constituting the run (total of four temperature readings per average).
- 5.14 Calculate and record the net meter volume for each run by subtracting the initial volume from the final volume recorded for the run.
- 5.15 Calculate the Y_i factors using the following equation:

$$Y_i = \frac{K' \times P_b \times (T_m + 460) \times \theta}{17.64 \times V_m \times \left(P_b + \frac{\Delta H}{13.6} \right) \times \sqrt{T_{amb} + 460}}$$

where:

K'	=	critical orifice calibration factor
ΔH	=	average differential pressure across DGM orifice during run (inches W.C.)
P_b	=	barometric pressure (inches Hg)
θ	=	net duration of individual calibration run, minutes
T_{amb}	=	average ambient temperature during run (°F)
T_m	=	average meter temperature during run (°F)
Y_i	=	post-test meter calibration factor for run i.

- 5.16 Average the Y_i values obtained for the three runs using the following equation:

$$\bar{Y}_i = \frac{Y_1 + Y_2 + Y_3}{3}$$

- 5.17 Calculate the calibration error using the following equation:

$$Cal.Error = \frac{\bar{Y}_i - Y_d}{Y_d} \times 100$$

where Y_d is the full-test meter calibration factor.



Clean Air Engineering

Standard Operating Procedures

Equipment Calibration

Meter Cal./Critical Orifice
Field Project Management
SOP I.D.: CAL-3
CTPM Section J Page 4

Effective Date: 11/1/95

Drafted By: JE Wright

Approved By: Process Owners

5.18 An acceptable post test calibration error is $\pm 5\%$. If the post calibration exceeds this specification, then the following action must be taken.

- i) The calibration repeated on-site using an orifice that is second closest to the average ΔH sampled.
- ii) If the second calibration is out of specification, then the test leader must indicate on the calibration sheet that the meter is out of calibration. Upon return to the office, the meter will be post-test calibrated with the wet test meter. If this calibration is out of specification, then the lowest Y_d will be used for the emission calculations, and the meter will be full-test calibrated and adjusted if necessary.

5.19 Calculate the percent variation for each Y_i value using the following equation (performed automatically by computer program):

$$\Delta Y_i = \frac{Y_i - \bar{Y}_i}{\bar{Y}_i} \times 100$$

5.20 If the calibration error is acceptable, but the percent variation is greater than 2%, then repeat the calibration using the same orifice.

5.21 Upon completion of the calibration, document the results on the enclosed yellow meter calibration card. Also indicate any other notes or problems with the meter performance on this card. Return all meter post calibration data sheets to the Job Leader or the designated data assembly area.

6.0 ATTACHMENTS

The following attachments are included as reference:

- Blank Data Sheet CS 005 (Revision 10/15/95)
- Completed hand-written example of Data Sheet CS 005
- Computer Printout of completed data sheet CS 005

CAE STANDARD OPERATING PROCEDURE

METER CALIBRATION – POST-TEST

ISO NUMBER: CAL-4

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

PURPOSE: This procedure provides a detailed account of post test meter calibrations. Allowable calibration tolerances are included. This procedure is intended to ensure our clients of quality field operations.

SCOPE: This procedure applies to all Clean Air Employees associated with the quality control and assurance of field instrumentation.

MATERIALS:

- Standardized wet test meter with a capacity of 1 ft³/rev
- Thermometer for wet test meter
- Field Meter Box
- Hg Barometer
- Flexible Tygon tubing
- Ring Stand with clamp
- H₂O filled impinger
- Bottle filled with silica gel
- Timer or stopwatch if meter box is not equipped
- Control valve

GENERAL:

References

40 Code of Federal Regulations, Part 60, Appendix A, Method 5, Revised as of July 1, 1992

Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods, U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory, Research Triangle Park, NC, August 1977

Procedure for Gas Monitor Operations, Clean Air Engineering, Pallantine, III

POST TEST METER CALIBRATION PROCEDURE

Meter Box Inspection and Leak Check

1. Visual inspection of exterior housing of meter box.
2. Clean exterior of meter box (use Windex or soapy water mixture).

3. Remove front and rear covers and examine for visible damage. Check for oil leaks, Tygon tube damage, and frayed power cords.
4. Check manometer oil and pump oil. Fill or change if needed.
5. Clean interior of meter box.
6. Level manometer assembly.
7. Plug in pump cord to specified meter box receptacle.
8. Connect pump vacuum and pressure connections to their corresponding meter box connections.
9. Plug in meter box to a grounded electrical receptacle.
10. Before calibration of the metering system, a positive and negative leak check should be performed.

Positive leak check of the system

- a. Disconnect the orifice meter line from the downstream orifice pressure tap (the one closest to the exhaust of the orifice). Plug this tap.
- b. Vent the negative side of the orifice to the atmosphere.
- c. Place a one hole rubber stopper with a tube through its hole in the exit of the orifice, and connect a piece of rubber tubing to the tube.
- d. Open the positive side of the orifice manometer.
- e. Plug the inlet of the vacuum pump. If a leak-free quick connect is on the meter box then this would not be necessary.
- f. Open the main valve and by-pass valve.
- g. Blow into the tubing placed at the orifice exit until a pressure of 5-7 inches of H₂O have built up in the system.
- h. Plug or crimp the tubing to maintain the pressure.
- i. Observe the manometer for 1 minute. No movement should be observed.

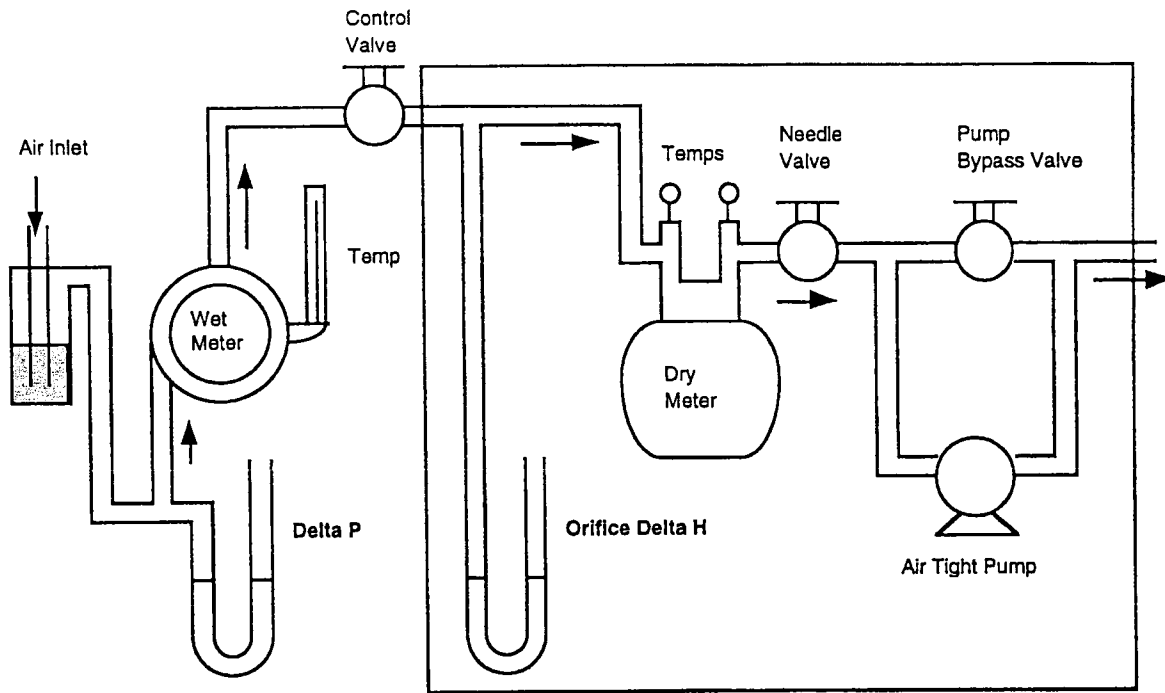
Negative leak check of the system

- a. Plug up the inlet to the vacuum pump.
- b. Turn on the pump and pull a vacuum of 3 inches Hg.
- c. Observe the dry gas meter. The metering system should not exceed beyond 0.005ft³/min leakage.

Post test meter calibration

Post-test meter calibrations are to be performed at the highest vacuum and average orifice settings from prior field operations. These are found on the meter calibration card located on the front panel of the meter box. Consult the attached figure for help.

EPA Method 5 Dry Meter Calibration



Meter Box System

1. Open the main pump valve. Run the meter box for 15 minutes at the appropriate vacuum and orifice setting before beginning post calibration. Vacuum and orifice adjustments are made with the control valve at the inlet to the meter box and the fine tune bypass valve. Close the main pump valve. This is to warm the meter box to a stable operating temperature and to condition the inside of the wet test meter.
2. Record the volume of the wet test meter and the volume on the meter box to be calibrated. The wet test meter needle should be located at the 12:00 o'clock position as the calibration characteristics of the wet meter are optimum on integer volumes. Also reset the meter timer.
3. Start timer at the moment the valve is opened.
4. Record inlet and outlet dry gas meter temperatures. Record wet test meter temperature. Refer to Post Test Meter Calibration Data Sheet included in this procedure.
5. A standard meter volume (the wet test meter in this case) of 10 ft³ is desired for a calibration run. Close the main valve and stop the timer when the wet test meter reaches the 12:00 o'clock position on the 10th rotation.
6. Record final gas volumes and temperatures for both meters. Run 1 is complete.
7. Take the barometric pressure for the day.

8. Re-set timer and start Run 2. Run at the same rate as in Run 1. Starting volume for Run 2 will be the same as final of Run 1. Record starting temperatures for both meters.
9. Complete calculations for DelH@, Y, and Q. Record results on post-cal sheet. Tolerances are $0.95 < Y < 1.05$ and $\text{DelH@} = 1.84 \pm 0.25$.
10. Complete Runs 2 and 3 in the same manner as Run 1.
11. Complete the calculations and make a comparison of results between Runs 1 through 3. An additional Run may be necessary if: 1) Y of any run differs ± 0.02 from Yavg 2) DelH@ of any run differs ± 0.2 from DelH@avg.
12. If no additional Runs are necessary complete the meter calibration card and place it on the meter panel. Note that if post-calibration Y resulting is $< 5\%$ from the full calibration Y (Y on the calibration card) another full meter calibration is needed. If the post calibration results are within 5% from the meter card then only the DelH@ from post calibration should be placed on the card.
13. Remember to note any equipment modifications or test observations in the meter calibration log.

MEASUREMENT:

Post test meter calibrations are to be performed each time an individual meter returns from the field. Full calibration of meter is necessary if post test meter calibration fails.

The wet test meter should be calibrated once a year with a spirometer or liquid displacement meter. Documentation of the wet test meter certification should be filed appropriately.

The Hg barometric measurements should include gravimetric and ambient temperature corrections.

APPENDIX:

CAE STANDARD OPERATING PROCEDURE
METER CALIBRATION AND MAINTENANCE
RECORDKEEPING

ISO NUMBER: CAL-5
EFFECTIVE DATE: Pending

AUTHOR: Dept. 68
APPROVED BY: Pending

1.0 PURPOSE

This procedure provides instructions and guidance for maintaining records of calibration and maintenance activities for meter consoles.

2.0 SCOPE

All personnel engaged in calibration, inspection, or inventory check-in of meter consoles should use this procedure. The procedure covers the following general topics:

- Filling out Meter Box Calibration Record forms
- General recordkeeping using Meter Console Logbook
- Calibration record database management.

This document does not address specific calibration or maintenance procedures.

3.0 MATERIALS NEEDED

The following materials are required to properly execute this procedure:

- Meter Console Calibration Record form (Form No. NA 6801)
- Meter Console Logbook
- Meter Console Archival Folder
- Most recent meter calibration data
- Microsoft Excel worksheet - *Meter-x.dbc.date* (where x=specific meter console identification number)
- Microsoft Excel worksheet - *Meter Full Cal.wks*, *Meter Post Cal.wks* or *Meter Post w/Orifice.wks* (whichever is appropriate)
- Standard data sheet - *CS 001 Full-Test-English*, *CS 002 Post Test-English*, or *CS 005 Critical Orifice* (whichever is appropriate)

4.0 DEFINITIONS

- **Meter Console** - a complete isokinetic sampling console consisting of a dry gas meter, vane pump, dual inclined manometers, pyrometer, and associated valves and electrical controllers. Also referred to as a Meter Box.
- **Full-Test Calibration** - a calibration of the meter console against a standard wet-test meter over the full range of operating flow rates as required by section 5.3.1 of EPA Method 5 (40 CFR 60, Appendix A). This calibration is performed at three operating flow rates, corresponding to orifice pressure drops (ΔH) values of (nominally) 0.5, 1.7, and 3.0 inches WC. The full-test calibration is required to be performed before the console is first used, and at least once annually thereafter. It is also required anytime major changes, adjustments or repairs are made to the meter.

- **Post-Test Calibration** - a calibration of the meter console at a single specific flow rate performed after a project and required by section 5.3.2 of EPA Method 5 (40 CFR 60, Appendix A). This calibration may be performed by comparing the meter against either the standard wet test meter or a calibrated critical orifice. It is required after each project in which the meter console was used.
- **NA 6801 Meter Console Calibration Record Form** - this is a proposed standard data sheet used to keep a record of meter console calibrations and routine maintenance activities.
- **Meter Console Logbook** - a 1-inch three-ring binder used to store meter calibration and maintenance information. A separate binder is kept for each meter console. The binder contains the following information specific for the associated meter console: Meter Console Calibration Record form, Calibration History Plot, Full-Test Calibration Data sheets and Post-Test Calibration sheets. The logbook contains the calibration and maintenance information generated since the second most recent full-test calibration. The Table of Contents for a given logbook is as follows:
 - Calibration History Plot
 - Meter Box Console Calibration Record
 - Calibrations since the current full-test calibration
 - Calibrations performed during the previous full-test calibration period
- **Meter Console Archival Folder** - a standard file folder which is used to archive old meter calibration and maintenance information generated prior to the second most recent full-test calibration.
- **Meter Calibration Computer Folder** - a computer folder which contains all of the calibration data worksheets and databases for a specific meter console. Each meter console has its own folder, identified as *Meter-x.Calibration*, where *x* stands for the specific I.D. of the meter console. This folder is located on the file server.
- **Meter Database** - a historical record of calibration information since the most recent full-test calibration of the console. This information is recorded in a Microsoft Excel file called *Meter-x.dbs.date*, where *x* stands for the specific I.D. of the meter console, and *date* is the date of the most recent full-test calibration of the console.
- **Calibration History Plot** - a graphical representation of the meter coefficient (Y_d) from each post-test calibration performed since the most recent full-test calibration of the meter. This plot is generated by the *Meter-x.dbs* worksheet.
- **CS 001 Full Test English Data Sheet** - this is a standard data sheet used to record the data from a full-test calibration.
- **CS 002 Post Test English Data Sheet** - this is a standard data sheet used to record the data from a post-test calibration using a standard wet test meter.
- **CS 005 Critical Orifice Data Sheet** - this is a standard data sheet used to record the data from a post-test calibration using a critical orifice. This data is normally recorded in the field.
- **Meter Full Cal.wks Worksheet** - Microsoft Excel worksheet used to perform meter calibration calculations for a full-test calibration.

- **Meter Post Cal.wks Worksheet** - Microsoft Excel worksheet used to perform meter calibration calculations for a post-test calibration using standard wet test meter.
- **Meter Post w/Orifice.wks Worksheet** - Microsoft Excel worksheet used to perform meter calibration calculations for a post-test calibration using a critical orifice.

5.0 GENERAL

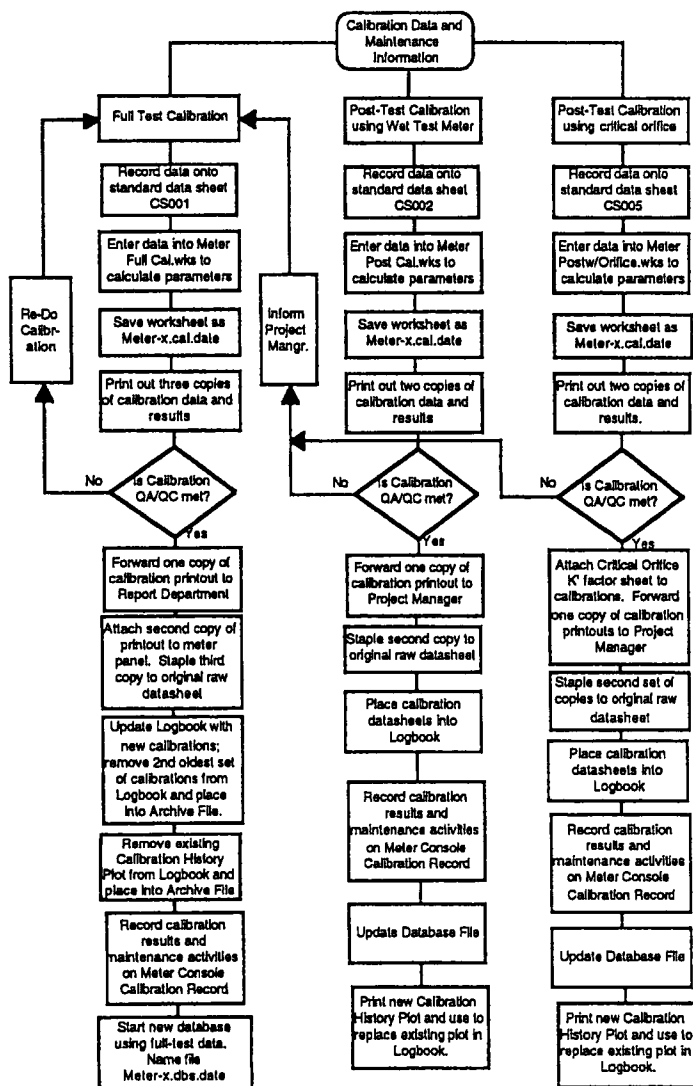
Documentation of meter console calibration and maintenance activities is important for data processing, recordkeeping, and legal purposes. A meter console is normally calibrated after every project. This data is manually recorded on a standard data sheet. The data is then transferred to a computer worksheet which calculates the appropriate calibration factors. The output of the computer worksheet is printed and affixed to the datasheet, and both items are placed into the Meter Console Logbook. A copy of the computer printout is forwarded to the appropriate Project Manager. The results of the calibration, as well as any specific maintenance procedures performed on the console, are recorded on the Meter Console Calibration Record, which is also contained in the logbook. The calibration results are also entered into a computer database for the console. A graphical plot of the calibration history of the meter is printed from the database, and this plot is placed into the logbook.

6.0 PROCEDURE

- 6.1 Record all calibration information on the appropriate standard datasheet.
- 6.2 Enter the calibration data into the appropriate computer worksheet. Save the completed worksheet using the "Save As" option. Name the file using the following convention: Meter-x.cal.date, where x is the meter console ID and date is the date of the calibration. The saved file should be placed into the appropriate Meter Calibration Computer Folder located on the File Server.
- 6.3 Print two hardcopies of the completed computer worksheet. If the calibration is full-test, print a third copy and attach to inside of the front meter panel.
- 6.4 For post-test calibrations using critical orifices, attach a copy of the appropriate *Critical Orifice K'.wks* to the meter calibration printout. Printouts of these sheets are contained in a separate binder labeled "Critical Orifice Calibrations".
- 6.5 For full-test calibrations, forward one copy of the calibration computer printout to the Report Department in Palatine. For post-test calibrations, forward one copy of the calibration computer printout (and Critical Orifice K'.wks if done with a critical orifice) to the applicable project manager.
- 6.6 Staple a copy of the calibration computer printout to the original data sheet and place the pair into the Meter Console Logbook. If the calibration is a post-test, then place the sheets at the front of the most recent full-test calibration section of the book. If the calibration is a full-test, then the contents of the logbook should be arranged appropriately to update the book so that only the two most recent full-test calibrations remain in the book. The oldest set of calibrations should be removed from the book, clipped with a small binder clip, and placed into the Meter Console Archival Folder.

- 6.7 Record the results of the calibration on the Meter Box Console Calibration Record. Also record any maintenance activities which were performed. If the calibration is a full-test, then record "N/A" under the columns for Job Number, Job Name, and Vacuum; record the ΔH range under the ΔH column. If the calibration is a post-test performed using a critical orifice, then record the critical vacuum under the Vacuum column, and record "N/A" for the ΔH @.
- 6.8 Enter the calibration results into the appropriate Meter Database. If the calibration is a full-test, then start a new database, using the date of the calibration as the filename suffix. For example, if Meter 68-A is full-test calibrated on January 4, 1995, then name the database "Meter-68A.dbs.1/4/95".
- 6.9 While in the Meter Database, double-click on the plot window and then print the calibration history plot. Replace the existing calibration history plot in the logbook with the new plot. If the new plot results from a full-test calibration, then place the old plot into the Meter Console Archival Folder. Otherwise, discard the old plot.

7.0 PROCEDURAL FLOW SHEET



CAE STANDARD OPERATING PROCEDURE

NOZZLE CALIBRATION RECORDKEEPING

ISO NUMBER: CAL-6

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1.0 PURPOSE

This procedure provides instructions and guidance for maintaining records of calibration data of probe nozzles used for isokinetic sampling.

2.0 SCOPE

All field personnel engaged in conducting an isokinetic sampling method should use this procedure. The procedure covers the following general topics:

- Field assignment of nozzle identifications
- Field calibration of nozzles
- Documentation of nozzle information

This document does not address permanent identification procedures for nozzles. However, the procedures contained herein for field assignment of nozzle ID's could be expanded to include assignment of permanent ID's.

3.0 MATERIALS NEEDED

The following materials are required to properly execute this procedure:

- Micrometer capable of measuring within 0.001 inches
- Nozzle calibration data sheet (Standard data sheet code DS 006)
- Nozzles
- Title 40 of the Code of Federal Regulations, Part 60, Appendix A (40 CFR 60, Appendix A)

4.0 DEFINITIONS

- **Nozzle** - any metal or glass button-hook or elbow shaped conduit affixed to the end of an isokinetic sampling probe. Normally, the nozzle has a sharp, tapered leading edge, with the taper angle less than 30°. The taper is on the outside of the tubing to preserve a constant internal diameter.
- **Nozzle ID** - an alpha-numeric code which uniquely identifies the nozzle for purposes of data sheet documentation. Whenever practical, the nozzle ID should be permanently inscribed into the nozzle itself. In cases where the permanent ID becomes illegible, or if a nozzle cannot be inscribed, this document outlines the process of developing an ID in the field which serves to identify the nozzle for the current project on which it is being used.
- **Nozzle Diameter** - The interior diameter of a nozzle at the sampling end opening.
- **Nozzle Calibration** - the process of measuring and recording the nozzle diameter. This procedure is derived from EPA Reference Method 5 of 40 CFR 60, Appendix A.

- **Nozzle Calibration Data Sheet** - A standard data sheet used to document the results of nozzle calibrations. Multiple nozzles may be recorded on the same sheet. However, the sheet is job-specific, and in some cases may be specific to a given test location. This sheet is kept on the Palatine file server as DS 006. A copy of the sheet is included with this document.

5.0 GENERAL

Documentation of nozzle diameters is important for data processing, recordkeeping, and legal purposes. Whenever a nozzle is used in the field, it should be calibrated. This calibration should be properly documented and referenced to the specific ID of the nozzle. If no ID exists for the nozzle, then one should be field-assigned for recordkeeping purposes. The nozzle ID serves to identify the nozzle material and type, as well as to reference the nozzle with its calibration. It should be used on all field sampling data sheets relating to the nozzle.

6.0 PROCEDURE

6.1 Assignment of Field Nozzle ID's. The following guidelines apply to any nozzle, which is used in the field and is found to be missing a legible ID.

6.1.1 The nozzle is given a three-part identification (ID) code. The format for this identification is as follows:

M - NNN - D

where:

M = material identification code
NNN = nominal nozzle opening diameter
D = specific alphabetic character unique to each nozzle in a given set

6.1.2 Material identification codes are as follows:

S = stainless steel
P = standard pyrex glass (for union connectors)
T = tapered pyrex glass
I = inconel steel
Q = quartz (usually part of a single-unit probe liner)
P = EPA Method 201 or 201A steel nozzle designs

6.1.3 For example, the following nozzle identifications would be used:

S-250-A => 1/4-inch stainless steel nozzle; "A" indicates that it is the first in a series.
P-375-C => 3/8-inch pyrex nozzle, third in a series.

6.1.4 The nominal size stated in the ID may be the actual measured size of the nozzle, but is not required to be. The purpose of the nominal size is strictly to serve in uniquely identifying the nozzle. The actual nozzle diameter which is used in isokinetic calculations should always be obtained from the nozzle calibration sheet.

6.2 **Nozzle Calibration.** After assigning a nozzle ID, or verifying that an ID already exists, calibrate the nozzle using a micrometer.

6.2.1 Follow the procedures given in Section 5.1 of EPA Method 5 of 40 CFR 60. This procedure requires that the nozzle be measured along three different diameters to within 0.001 inches.

6.2.2 Record the results of the calibration on the Nozzle Calibration data sheet. Reference the nozzle ID with the calibration.

6.2.3 The average of the three measurements should be used as the true diameter of the nozzle.

6.2.4 If the difference between any of the three measurements exceeds 0.004 inches, then the nozzle cannot be used without being reshaped and resharpened.

6.3 **Referencing ID's.** On the applicable field sampling data sheet, reference the permanent or field-assigned nozzle ID in the "Nozzle I.D." blank. Record the nozzle diameter from the field calibration in the "Nozzle Diameter" blank.

CAE STANDARD OPERATING PROCEDURE

**WET TEST METER CALIBRATION PROCEDURE
(DISPLACEMENT METHOD)**

ISO NUMBER: CAL-7

EFFECTIVE DATE: Pending

AUTHOR: Dept. 68

APPROVED BY: Pending

1.0 PURPOSE

This procedure provides instruction for performing a Wet Test Meter (WTM) calibration check utilizing the water displacement method as outlined in Section 3.5.2.1 of the EPA "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods", August 1988 (EPA-600/4-77-027b).

2.0 SCOPE

All personnel performing meterbox calibrations either with the WTM and/or critical orifices should be familiar with this procedure to perform periodic calibration checks of the primary laboratory standard. This procedure should be followed at least once annually, or whenever the accuracy of the WTM is in question.

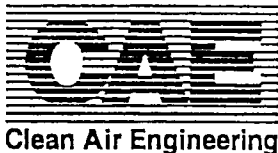
3.0 MATERIALS NEEDED

The following materials (or equivalents) are needed to properly perform this procedure:

- WTM apparatus
- Container 1
- Container 2
- Tubing clamp
- 2-Liter plastic beaker
- Balance (capacity at least 3,000g)
- Pyrometer
- Calibration data sheet - NA 6803 WTM-CAL
- Microsoft Excel worksheet - WTM-CAL.wks
- Calibration Area

4.0 DEFINITIONS

- **Wet Test Meter apparatus** - a complete WTM assembly consisting of an American WTM (or equivalent), thermometer, draft gauge connected to the WTM inlet, saturator and associated hose and hardware connections. Figure 1 shows the general assembly of this apparatus.
- **Container 1** - a leak-free, rigid-walled vessel with sufficient volume to displace at least one complete revolution of the WTM (10 gallons for a one cubic foot displacement WTM, 3 gallons for a 0.250 cubic foot displacement WTM). Container 1 must have a leak free fitting to facilitate a hose connection to the WTM from a spot above the water line at full capacity (preferably in the cap) as well as a hose connection (with 3-4' section of hose) at the bottom of the vessel to drain water into container 2.



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Approved By: TRB

- **Container 2** - a second leak-free, rigid-walled free vessel with sufficient volume to displace at least one complete revolution of the WTM (10 gallons for a one cubic foot displacement WTM, 3 gallons for a 0.250 cubic foot displacement WTM). Container 2 does not require a top hose fitting or a cap. The bottom hose connection should be valved to facilitate control while emptying displacement water into the measuring beaker.
- **Tubing clamp** - A clamp of sufficient size to control the displacement water flow from one container to the next.
- **2-Liter plastic beaker** - A leak-free 2-liter Nalgene beaker (or equivalent) that, when filled to capacity, does not exceed the maximum measurable mass of the balance.
- **Balance** - A balance (preferably electronic with an auto-tare feature) with a capacity of at least 3,000g to measure the displacement water mass
- **Pyrometer** - A Fluke (or equivalent) pyrometer with solid thermocouple to record displacement water temperature. An equivalent immersible thermometer may also be used.
- **Calibration data sheet** - NA 6803 WTM-CAL - this is a standard data sheet to record the parameters of a WTM calibration by the displacement method.
- **Microsoft Excel worksheet** - WTM-CAL.wks - this is the standard worksheet to perform percent error calculations of WTM calibration by the displacement method.
- **Calibration Area** - A temperature-controlled area to allow the WTM and displacement water temperatures to stabilize for a period of time to reach equilibrium. In addition, the room should have a counter top of sufficient height to allow the water level in Container 1 to remain above Container 2 for the duration of the displacement run.

5.0 BACKGROUND

WTMs are shipped by the supplier calibrated to an accuracy tolerance of $\pm 0.5\%$, usually against a primary standard. As per the EPA QA/QC manual, it is required that the WTM have a calibration check performed immediately upon receipt of the instrument from the manufacturer and at least once annually thereafter. Ideally, this calibration check should be conducted by using a primary standard such as a bell prover or spirometer. In lieu of these standards, a calibration check accurate to within $\pm 1.0\%$ can be conducted using the displacement method as outlined in this procedure. All data generated by these calibrations will be kept in a one-inch three-ring binder. A standard WTM calibration data file will also be maintained on the file server in the meters database section.



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Approved By: TRB

6.0 PROCEDURE

6.1 Assemble the apparatus as illustrated in Figure 1.

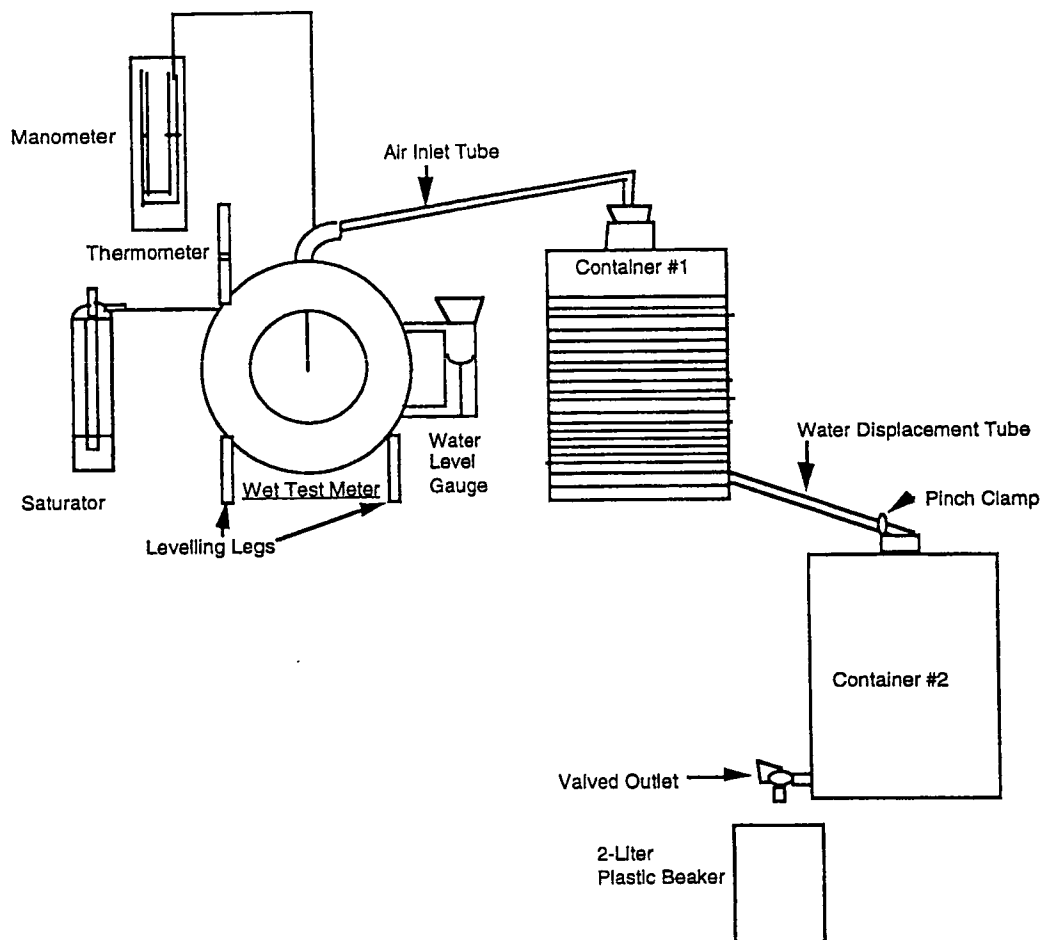


Figure 1: Wet Test Meter Displacement Set Up



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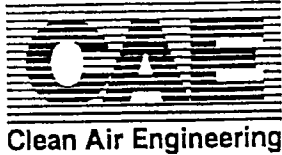
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Approved By: TRB

- 6.2 Check the level (bulls eye level) of the WTM. Adjust the bubble to the center of the bulls eye using the leveling knobs located on each of the four legs on the WTM.
- 6.3 Adjust the water level in the WTM so that the pointer (right hand side) is level with the meniscus of the water in the reservoir. If you must add water to the reservoir to accomplish this be sure to pull air through the WTM, at least one cubic foot or one revolution, and recheck the level after water has been exposed to all four chambers of the WTM. Inversely, perform the same one revolution "pull" if the reservoir is overfilled and water must be drained. (The pointer should appear in the reflection of the reservoir water as a single line tapering to a point intersecting with its mirror image at the bottom of the meniscus.)
- 6.4 Fill Container 1 with sufficient water to displace at least one complete revolution (one cubic foot) of the WTM (10 gallons). Any extra water in the vessel can be used to conduct a system leak check prior to performing the calibration check.
- 6.5 Allow Container 1 and the WTM to sit at least overnight in the calibration area to allow the displacement and WTM water to reach thermal equilibrium.
- 6.6 Recheck the WTM water level after the thermal stabilization period. Adjust water level if necessary with water at the same temperature.
- 6.7 Fill out WTM displacement data sheet (NA 6803 WTM-CAL) date and ambient temperature and prepare to begin the calibration check.
- 6.8 Conduct system operation check. To accomplish this, open the tubing clamp at the Container 1 outlet and begin siphoning water through the system. Allow approximately one gallon of water to flow through the system. At this point record the WTM ΔP reading from the inlet manometer to the data sheet. If the ΔP is less than 0.4" w.c. the system is functioning normally and you may proceed to section 6.8. If the ΔP is greater than 0.4" w.c. refer to section 6.8a.
- 6.8a Most likely causes for excessive ΔP are: too much water in the saturator, inlet hardware (piping) is too small and creating excessive pressure drop, or the WTM is defective. If removing water from the saturator and/or replumbing the WTM inlet do not drop the ΔP to under 0.4" w.c., then the WTM is most likely defective and should be repaired in-house or sent out for repair and recalibration. (See step 6.16 for name and address of repair company.)
- 6.9 After approximately one gallon of water has siphoned through the system, plug the saturator inlet. If the flow of water ceases, the system is leak free. If flow continues, then there is a system leak which must be found and corrected before proceeding. At the conclusion of the system leak check, pinch the tubing connecting Container 1 to Container 2 as close as is practical to the end of the tube to insure that the water volume in the tube is consistent from run to run.
- 6.10 Drain the displaced water from Container 2 back into Container 1 taking care to remove all the water from Container 2. Any water left in Container 2 at this time will bias the initial trial displacement volume.



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Approved By: TRB

6.11 Begin the calibration check. Record the WTM initial volume and temperature on the data sheet. Open the tubing clamp and allow the water to begin siphoning through the system. Allow the WTM to displace at least one complete revolution and close the tubing clamp taking care to place the clamp in the same position as it was at the commencement of the trial. The volume displaced by the WTM must be a whole number (i.e. 1.000 ft³). The nature of the instrument is that it will measure accurately in whole number increments of the capacity, but fractions thereof, i.e. 2.500 ft³, are not necessarily accurate, as each chamber in the meter does not displace the same volume.

6.12 Record the WTM final volume. At this time convert the WTM volume from ft³ to liters by multiplying by 28.317. At this time also measure the displacement water temperature and record it on the data sheet in the appropriate spot.

6.13 Place the 2-liter plastic beaker onto the balance and compensate for the tare weight of the beaker by auto-taring the beaker. Drain the water in Container 2 into the 2-liter beaker using the valved fitting at the bottom of the vessel. Stop short of maximum capacity so to minimize the chance of spillage and measure the mass in grams on the electronic balance. Dump the beaker's contents back into Container 1. Repeat this process until Container 2 is completely empty.

6.14 Determine the total mass of the displacement water catch in grams and enter it on the appropriate line of the data sheet. Convert the resulting mass into volume (liters) by dividing the sum by 1,000. Enter this number on the appropriate line of the data sheet.

6.15 Calculate the percent error using the following equation:

$$\% \text{ Error} = \frac{(\text{WTM volume (L)} - \text{Displaced Volume (L)})}{\text{Displaced Volume (L)}} \quad (\text{Note that \%Error is equivalent to } Y_d.)$$

If the percent error is acceptable ($\pm 1\%$), repeat steps 6.10 through 6.15 twice more for a total of three trials. If the error exceeds 1%, then refer to step 6.16.

6.16 Average the values for the three acceptable runs and record on the data sheet.

If the average percent error is greater than $\pm 1\%$, and the pretest leak check and pressure drop criteria are met, adjust the water level of the wet test meter (see the manufacturers manual) upward if the meter is slow (negative error) or downward if the meter is fast (positive error) and repeat steps 6.10 through 6.15 until three acceptable trial are conducted. If the WTM still fails the calibration check it will most likely need to be sent out for repair and recalibration to:

Carl Poe Company
99 Reinerman Street
Houston, TX 77007
(713) 861-3816



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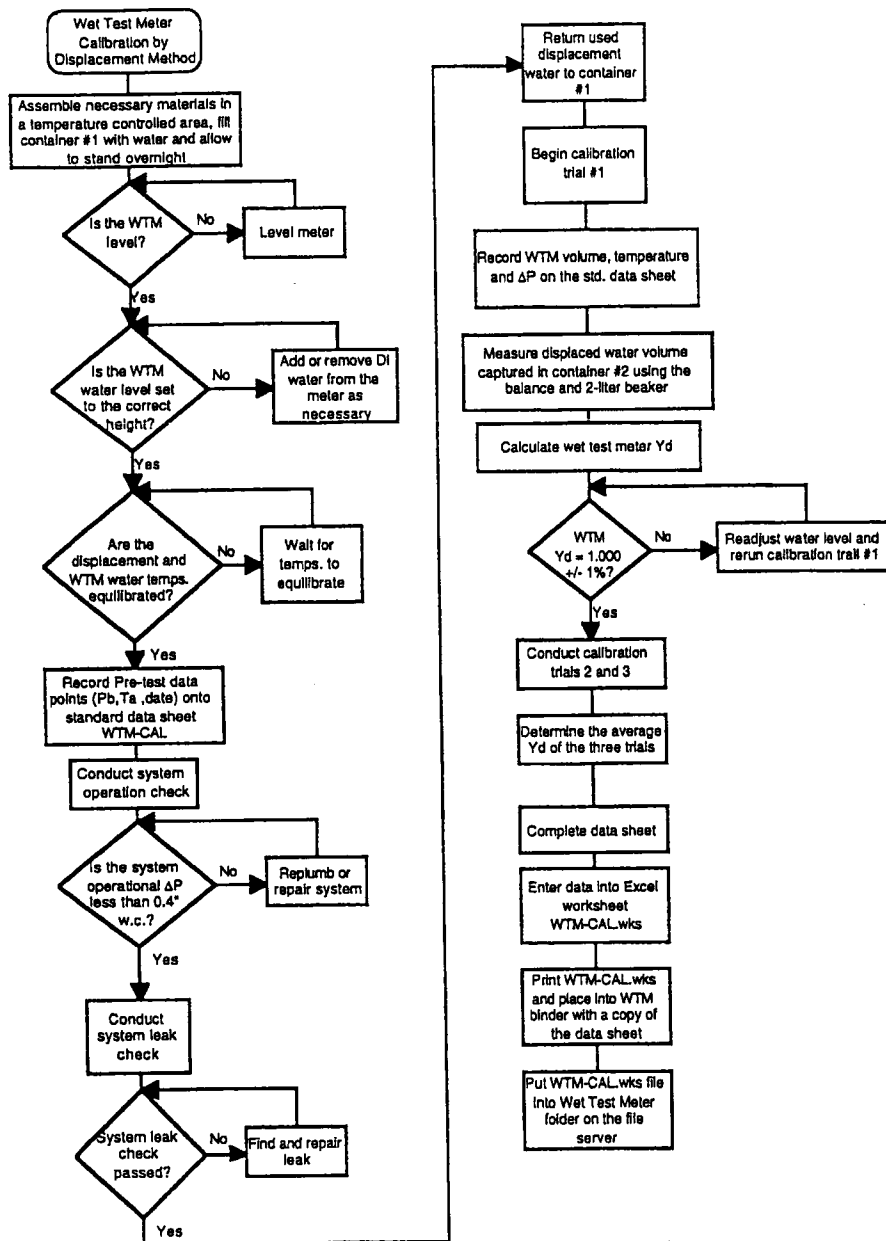
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Be sure that they forward their calibration results along with the meter. Keep a copy of the Carl Poe calibration results in the WTM record file along with the displacement calibration check conducted immediately after receipt of the meter.

- 6.17 Enter the results on the Excel worksheet WTM-CAL.wks and save in the WTM calibration file on the server. Name the calibration file by date - WTM serial number-department i.e. 2/21/95-17404-68.
- 6.18 Make a copy of both the data sheet and worksheet and place in the three ring WTM calibration record file.

7.0 PROCEDURAL FLOW SHEET





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8.0 DATA SHEET NA 6803 WTM-CAL



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Approved By: TRB

9.0 SECTION 3.5.2.1 OF EPA /600/4-77/027b

CAE STANDARD OPERATING PROCEDURE

GENERAL TEST LOCATION EQUIPMENT BREAK DOWN

ISO NUMBER: GEN-1

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

- Purpose: The purpose of this procedure is to outline the steps necessary in breaking down a location after testing is complete.
- Scope: This procedure will be used for all locations when applicable.
- Materials: The materials needed vary depending on the scope of the work. These will be the same materials used during the set up period.
- Definitions: Port Covers: This refers to the covering over the test location access ports.
- Procedure: The following steps are taken when breaking down the equipment from a test location.
1. It is the Test Leader's responsibility to make the decision that the project is complete and that breakdown may begin. No equipment is to be removed from a sampling location without the test leader's approval.
 2. CEM systems must be purged with ambient or zero air or with nitrogen before being broken down. All particulate sample probes must be cleaned and sealed.
 3. Roll up all extension cords and umbilicals in a neat and orderly fashion securing all coiled hoses, etc. with duct tape.
 4. Return all items to their original containers - tools to their toolboxes, meters, glassware, pyrometers, etc. to their proper boxes. Pack loose items with their packing materials and into their proper containers.
 5. Remove all trash from all CAE work areas, place in proper receptacle.
 6. Return borrowed items to the plant contact who obtained them.
 7. Safely transport all equipment to the van packing area. Follow all applicable safety precautions when roping equipment, using elevators, winches, etc...
 8. Refer to the procedure titled "" for proper truck packing instructions.

9. Replace all port covers using anti-sieze compound on all threads. Notify the client contact of any deteriorated or un-sealable ports.
10. Make a final inspection of all CAE work areas to insure that areas are clean and secure.
11. DO NOT:
 - THROW, DROP OR BE CARELESS WITH ANY EQUIPMENT
 - PACK GLASSWARE WITH HEAVY ITEMS
 - PACK COOLERS OR SAMPLE BOXES WITH WATER, ICE OR TRASH IN THEM
 - PACK ANYTHING IN A WET COOLER
 - FAIL TO CLEARLY TAG BROKEN ITEMS

CAE STANDARD OPERATING PROCEDURE

EQUIPMENT EVALUATION TAGS

ISO NUMBER: GEN-2

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

Purpose: The purpose of this procedure is to instruct you when completing equipment evaluation tags.

Scope: This procedure will be used for all equipment evaluation tags.

Materials: The equipment needed for this procedure is an equipment evaluation tag. These are supplied to all field personel.

Procedure: The following steps are taken when completing an equipment evaluation tag.

I. Properly operating equipment with permanent tags (meter boxes).

a. Record all information required on the tag after each use.

II. Failed Equipment

a. Obtain a "HOLD FOR INSPECTION" tag.

b. Record on the tag:

1. A description of the problem encountered,
2. Project number,
3. Your name,
4. The date.

c. Attach the tag to the item in a secure manner in a prominent area.

d. Notify the test leader of the problem, the test leader should note it in his log and on a "Field Equipment Problem Report."

CAE STANDARD OPERATING PROCEDURE

KNOTS AND HAULING

ISO NUMBER: GEN-3

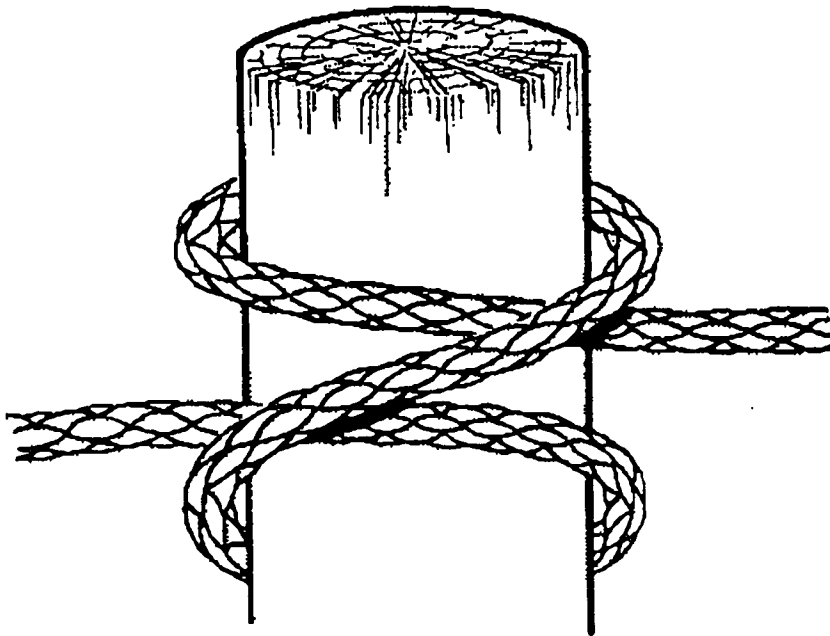
EFFECTIVE DATE: Pending

AUTHOR: Dept. 68

APPROVED BY: Pending

Clove Hitch

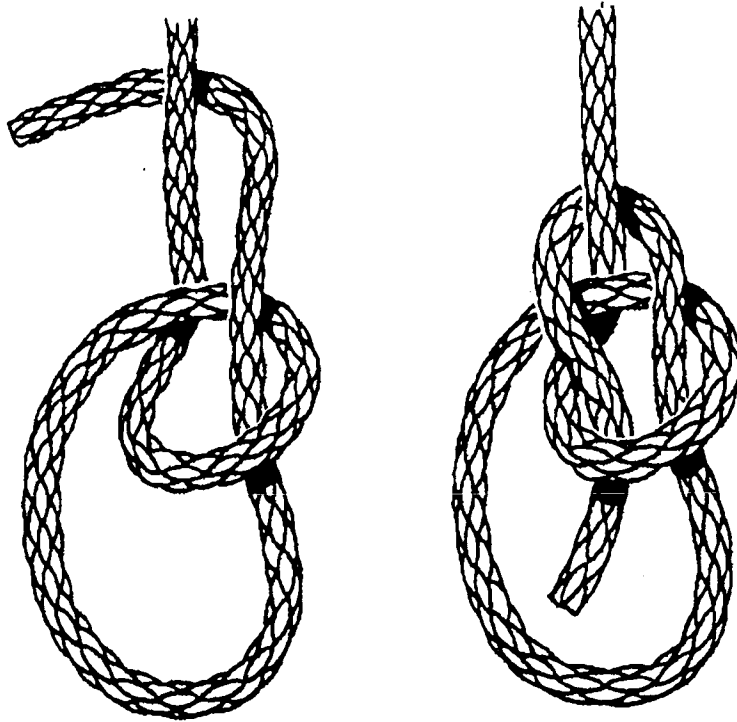
Commonly referred to as the "Probe Knot."



1. Create a 'half hitch' by wrapping the rope around the probe and over the haul line. Leave about four feet on the free end to finish the rest of the knot.
2. Continue wrapping in the same direction and create a second half hitch. This comprises the top part of the probe knot.
3. Finish the knot by creating a second clove hitch about two feet lower on the probe.

The knot can also be used to tie off a rope to a rail. For greater security, add one or two extra half hitches to the clove hitch.

Bowline



1. Create a loop in the haul line.
2. Feed the free-end through the loop and behind the haul line.
3. Bring the free-end back through the loop.

Use this knot for hauling equipment or for attaching a carabiner. The knot will not slip and can always be untied by folding the haul-line in the direction of the loop.

General Hoisting Guidelines

1. Never stand under the haul line. Always stand off center when hoisting from below.
2. If lifting an object made of several pieces (eg. a sample box attached to a probe) all connected objects are attached securely.
3. When possible, attached a carabiner to the haul line and use lifting straps to attach the equipment to the carabiner.

CAE STANDARD OPERATING PROCEDURE

UNISTRUT ASSEMBLY - CYLINDRICAL STACKS

ISO NUMBER: GEN-4

EFFECTIVE DATE: Pending

AUTHOR: Dept. 68

APPROVED BY: Pending

1.0 PURPOSE

This procedure provides detailed information on how to set-up unistrut in the field for a cylindrical stack with two or four ports.

2.0 SCOPE

This procedure applies to all CAE employees involved in setting up unistrut in the field or packing it for a specific job.

3.0 MATERIALS (FOR FOUR PORTS)

- 4, 5, or 6 ft. uprights (4)
- 8 or 10 ft. booms (4)
- L - brackets (4)
- Clothesline (100 ft.)
- $\frac{3}{4}$ Bolts and unistrut nuts (at least 16)
- Socket wrench with $\frac{3}{4}$ inch socket
- Trolleys (at least 5)
- $\frac{3}{16}$ inch wire rope
- Wire rope pullers (Come-a-longs) (at least 6)
- Carabiners (at least 10)
- Chains (4 to 8 links long)

4.0 DEFINITIONS

5.0 GENERAL PREPARATION

The setting up of unistrut requires at least two people. Before going into the field, perform the following:

- 5.1 Attach the L-brackets about six inches from one end of the upright.
- 5.2 Place a trolley in each boom before it goes into the field and place duct tape around the boom on each side of the trolley so that it will not fall out.
- 5.3 Put in an eyelet in each upright (opposite the L-bracket) and in each boom.
- 5.4 Tie two 10 to 15 ft. pieces of clothesline to the eyelets on the booms.



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Effective Date: 5/1/95

Standard Operating Procedures

Field Sampling

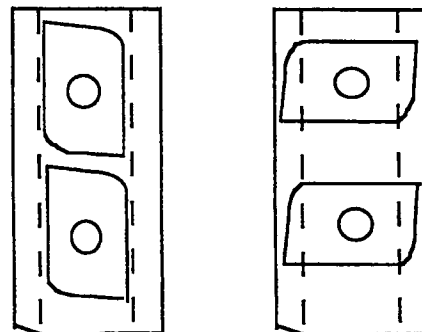
Drafted By: Dept. 68

Unistrut Assembly - Circular
General Field Technicians
SOP I.D.: GEN-4
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Approved By: TRB

6.0 PROCEDURE

- 6.1 Identify the location description. *Is it a square duct or a round stack?* As mentioned above, the steps for setting up on a cylindrical stack will be discussed here. Remember the job has to be packed according to the type of location that will be tested.
- 6.2 Once at the location, walk-out the wire rope on the wire rope puller (or come-a-long as it is more widely known) by releasing the safety latch and pull around the stack. If the wire cable does not reach around the circumference of the duct, then another wire rope will have to be added to connect one end of the come-a-long to the other. Make sure all wire ropes used are looped together at each end with wire rope U-clips. The distance between each clip should be no less than six times the cable diameter. **Note:** Be sure to place the wire rope and come-a-long about six to eight inches above the ports being tested.
- 6.3 Connect one end of another come-a-long to the eyelet on the upright. This will serve as a support line when connected to the eyelet on the boom.
- 6.4 Start to tighten (but not completely) the wire rope around the stack with the handle on the first come-a-long. Start slipping the uprights between the rope and the stack wall. Position the upright vertically and rest it on top of the port. Repeat for all ports.
- 6.5 Once all uprights are in place start tightening the come-a-long. The wire rope should cross on top of the L-bracket.
- 6.6 Stretch another come-a-long out and attach additional wire rope (if needed). Connect the come-a-long and wire rope around the stack and as far up the upright as possible.
- 6.7 Tighten both come-a-longs which encircle the stack as much as possible.
- 6.8 Once all uprights are tight against the stack wall, the booms can be connected to the uprights. Before connecting the uprights to the booms, connect the other end of the come-a-long to the eyelet on the boom, otherwise you will have to reach out over the railing to connect it when it is in place. Make certain when putting in the bolts, that the unistrut nuts are flush with the inside edges of the unistrut (see picture at right). It is suggested that the nuts with springs attached to them be used since they will not fall out of place when screwing in the bolts.



- 6.9 After the boom is connected firmly to the L-bracket pull the come-a-long tight until the

position of nuts before
and after tightening



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Drafted By: Dept. 68

Approved By: TRB

boom is level with the grating. The other person should be holding the boom until the come-a-long is tightened so that the weight of the boom does not bend the L-bracket. Once testing begins the wire rope around the stack may stretch a little causing the boom (or booms) to sag. Therefore it might be necessary to tighten the come-a-long every now and then.

- 6.10 Tie down the boom with the clothesline that is already attached to the eyelet. The best place to tie down to is the stack railing. One should tie to the railing as far away from the boom as possible. The further out, the more support.
- 6.11 At this point, the set-up of the unistrut should be finished. All that is left to do is test the unistrut and see if it will hold up under weight. Connect the probe to the split filter box. Attach a caribiner to the top of the filter box (if it is not already) and hook it to the trolley. Remember to remove the duct tape if it had not been removed yet. Carefully slide the probe and filter box forward to see if the probe will easily enter the port. The port cover should be removed when checking for clearance. If the probe does not clear the port because the boom is too high then a chain will have added between the trolley and caribiner. If the boom is too low, then the boom has to be taken off and the L-bracket raised a few inches and the boom reconnected. If care is taken to bolt on the L-brackets far enough from one end of the upright, one should never have to disconnect the boom in the field. Prevent the removal of the boom from happening, it will save a lot of time!

7.0 DIAGRAMS

On the following pages are drawings of the unistrut showing the front and side view and top view showing it connected to a stack with four ports.

METER BOX ASSEMBLY AND SET-UP

ISO NUMBER: MET-1

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

Author: Bill Ansell

List of Materials: Pump Box, Control Console, Meter Fitting Quick Connect, Pitot Line Quick Connect (If necessary), Pump oil

Definitions:

Pump Box - The bottom part of the meter box which houses the vacuum meter box, the pump provides the vacuum to pull sample gas through the sampling train.

Control Console - The upper part of the meter box assembly which contains the dry gas meter, heater controllers, thermocouple readouts, and draft gauges. The dry gas meter is used to measure gas volume, heater controllers are used to heat the sample probe and filter in the sampling train to a set temperature. The thermocouple readouts allow monitoring of stack temperature, dry gas meter inlet and outlet temperatures, probe and filter temperatures, and sample gas temperature exiting the last impinger. The draft gauges are inclined manometers which read in inches of water. The manometers are used for measuring the velocity pressure of the gas within the stack and the pressure drop across the orifice in line after the dry gas meter. The pressure drop across the orifice is used to determine the sampling rate.

Meter Fitting Quick-Connect - Leak-proof quick-release connector which allows the sample line to be connected to the control console.

Pitot Line Quick-Connect - Leak-proof quick-release connector similar to the meter fitting quick connect except smaller. This fitting allows simple bulkhead fittings that the pitot line can be slid onto.

Pump Oil - 10 weight single viscosity oil for lubricating pump assembly

Procedure Steps:

Refer to Figure 1 for a general schematic of the meter box assembly

1. Remove the pump box and control console from their shipping boxes.
2. Place the pump box onto a flat, level surface with the front facing toward you. The pump shipping box or control console shipping box works well for this purpose.
3. Remove the front cover of the pump box.
4. Remove the short power cord from the pump box and untangle and straighten the two hose lines connected to the pump.
5. Connect the female end of the short power cord to the plug on the side of the pump box.
6. Check that there is oil in the front oil jar of the pump. If not, unscrew the jar and fill it with oil to approximately half an inch from the bottom of the jar. A small plastic sample bottle containing 10 weight oil should be packed inside the pump box.
7. Place the control console on top of the pump box with the front facing toward you.
8. Secure the clasps on the sides of the pump box to the control console.
9. Remove front cover of the control console. A copy of the meter calibration and thermocouple calibrations should be taped to the inside of the front cover. These should be removed and given to the test leader to be included with the test report.
10. Check that the main console power switch is in the off position.
11. Unroll the main power cord from the control console and plug into nearest 110v, 15A outlet. (Note: This outlet must first be tested with a circuit tester or voltmeter to ensure that it is a properly wired 110V service.)
12. Plug the short power cord from the pump box into the outlet on the front of the control console labeled "pump".
13. Connect the two hose lines from the pump box to the control console. One line will have a "V" or "VAC" engraved on the fitting. Connect this line to the fitting labeled "VACUUM" on the front of the control console. The other line should be engraved with "P" or "Pressure" and should be connected to the fitting labeled "Pressure" on the front of the control console. Normally, the vacuum fitting is to the left of the pressure fitting.
14. The Meter Box Assembly is now complete and ready for the sample train umbilical to be connected.

CAE STANDARD OPERATING PROCEDURE

METER CALIBRATION WITH CRITICAL ORIFICE

ISO NUMBER: MET-2

EFFECTIVE DATE: Pending

AUTHOR: Dept. 68

APPROVED BY: Pending

List of Materials: Critical Orifice Set, Standard Data Sheet CS 005, Standard Worksheet Post-Test Cal-CO.xls

Definitions:

Critical Orifice - A device used to verify the calibration of a dry gas meter. The device consists of a small bored plate orifice assembled into a quick-connect fitting which can be plugged directly into the meter box sample port. The pressure drop across the orifice is used to determine the flow rate through the assembly.

Critical Orifice Set - A set of five critical orifices of varying diameters to allow a range of flow rates for meter box calibration purposes.

Critical Pressure - As the flow rate of gas through the assembly increases, so does the pressure drop across the orifice. At a certain pressure drop known as the critical pressure, the flow rate becomes constant - pressure drops higher than the critical pressure will not result in more flow. This critical pressure drop is normally at least 53% of the barometric pressure. Also called critical vacuum.

Procedure Steps:

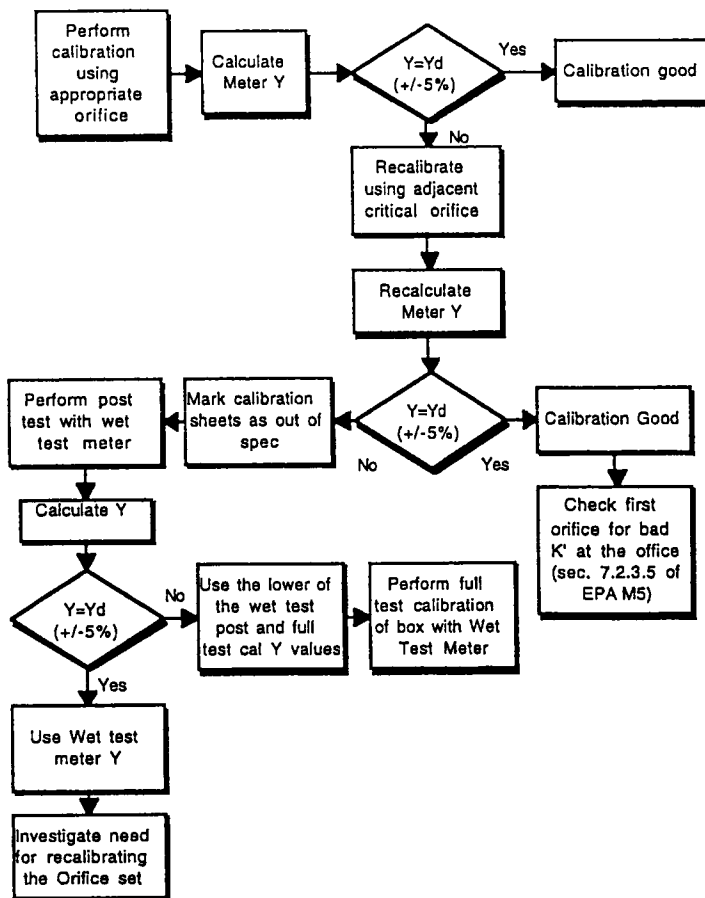
1. Pick a critical orifice from the set that will produce a ΔH close to that of the test conditions.
2. Plug the orifice into the meter inlet.
3. With the meter box running, leak check by plugging the inlet to the orifice assembly. The leak check is valid if no visible movement of the meter is seen for 1 minute.
4. Adjust the pump by opening the course valve and/or closing the fine valve until the ΔH stabilizes and the orifice reaches its critical pressure (vacuum).
5. Allow the pump to run for about 10 minutes at the critical vacuum so that the pump and meter temperatures stabilize.
6. While the meter box is warming up, fill in the initial data for the calibration on the standard data sheet CS 005.
7. After at least 10 minutes, begin the calibration. Reset the elapsed time meter to zero, and record the meter volume, elapsed time (equals 0), meter inlet and meter outlet temperatures onto the first line of the data sheet. The meter volume must be read on the fly at precisely the time designated as the start time (elapsed time = 0).

8. At five-minute (minimum) intervals, record the meter volume, elapsed time, inlet and outlet meter temperatures, ambient temperature, meter box ΔH , and meter vacuum. Once again, the meter must be read on the fly at precisely the time at which the elapsed time was recorded. Allow a minimum of 5 minutes between readings. More is OK, as long as the meter is read exactly at the time of the recorded time.
9. Repeat step eight two more times for a total of three readings and at least 15 minutes of operating time.
10. Input the data from the data sheet directly into the Post-Test Cal-CO.xls spreadsheet (or equivalent calculator program) to calculate the individual Y coefficients for each of the three 5-minute runs. The Y coefficients are calculated using the following equation:

$$Y = \frac{K' \times P_{bar} \times T_m \times \theta}{17.64 \times V_m \times \left(P_{bar} + \frac{\Delta H}{13.6} \right) \times \sqrt{T_{amb}}}$$

11. Refer to the attached figure for evaluation of the results of the calibration. An acceptable post test calibration average Y value is $\pm 5\%$ of the full-test Y_d . Also, each individual Y value must be within 2% of the average of the three values. If the post calibration is out of specification then the following action must be taken.
 - i) The calibration repeated on-site using an orifice that is second closest to the average ΔH sampled.
 - ii) If the second calibration is out of specification, then the test leader must indicate on the calibration sheet that the meter is out of calibration. Upon return to the office, the meter will be post-test calibrated with the wet test meter. If this calibration is out of specification, then the lowest Y_d will be used for the emission calculations, and the meter will be full-test calibrated and adjusted if necessary.
12. Upon completion of the calibration, document the results on the enclosed yellow meter calibration card. Also indicate any other notes or problems with the meter performance on this card. Return all meter post calibration data sheets to the Job Leader or the designated data assembly area.

Post-Test Calibration Corrective Action



CAE STANDARD OPERATING PROCEDURE

POST-TEST CALIBRATION OF A METER BOX USING A CRITICAL ORIFICE

ISO NUMBER: MET-3

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1.0 PURPOSE

The purpose of this procedure is to provide instructions for calibrating a meter box console in the field using a critical orifice.

2.0 SCOPE

These procedures are written explicitly for post-test verification of the dry gas meter calibration factor. However, they may also be used to verify the overall integrity of the metering system at any time during a test job (e.g., during equipment set-up).

3.0 MATERIALS

The following materials are needed to properly execute this procedure:

- Critical Orifice Set
- Standard Data Sheet CS 005
- Meter Box Console with Quick-Connect sample inlet fitting
- Meter Calibration Card
- (Optional) Standard Worksheet or Calculator program for performing calculations

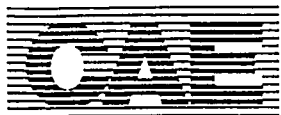
4.0 DEFINITIONS

Dry Gas Meter Calibration Factor (Y) - an expression of the relative accuracy of a dry gas meter. This factor is dimensionless, and typically has a value of 1.00 ± 0.02 . When written as Y_d , this parameter represents the calibration factor as determined from a full-test calibration of the meter against a wet test meter. When written as Y_i , it represents the calibration factor of the i^{th} run determined using a critical orifice.

Critical Orifice - A device used to verify the calibration of a dry gas meter. The device consists of a small bored plate orifice assembled into a quick-connect fitting which can be plugged directly into the meter box sample port. The pressure drop across the orifice is used to determine the flow rate through the assembly.

Critical Orifice Set - A set of five critical orifices of varying diameters to allow a range of flow rates for meter box calibration purposes.

Critical Pressure - As the flow rate of gas through a critical orifice increases, so does the pressure drop across the orifice. At a certain pressure drop known as the critical pressure, the flow rate becomes constant - pressure drops higher than the critical pressure will not result in more flow. This critical pressure drop is normally at least 53% of the barometric pressure. Also called critical vacuum.



Clean Air Engineering

Standard Operating Procedures

Equipment Calibration

Meter Cal./Critical Orifice
Field Project Management
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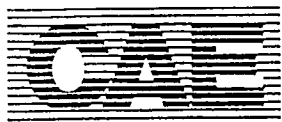
Drafted By: JE Wright

Approved By: Process Owners

5.0 PROCEDURE

- 5.1 Perform a positive pressure leak check of the meter box (see appropriate procedure). The leak check is valid if the manometer shows no visible movement in 1 minute.
- 5.2 Pick a critical orifice from the set that will produce a ΔH close to that of the test conditions.
- 5.3 Plug the orifice into the meter inlet.
- 5.4 With the meter box running, leak check by plugging the inlet to the orifice assembly. The leak check is valid if no visible movement of the meter is seen for 1 minute.
- 5.5 Adjust the pump by opening the course valve and/or closing the fine valve until the ΔH stabilizes and the orifice reaches its critical pressure (vacuum). If the critical vacuum cannot be reached, then a smaller orifice must be used.
- 5.6 Allow the pump to run for about 10 minutes at the critical vacuum so that the pump and meter temperatures stabilize.
- 5.7 While the meter box is warming up, fill in the initial data for the calibration on the standard data sheet CS 005.
- 5.8 After at least 10 minutes, begin the calibration. Reset the elapsed time meter to zero, and record the meter volume, elapsed time (equals 0), meter inlet and meter outlet temperatures onto the first line of the data sheet. The meter volume must be read on the fly at precisely the time designated as the start time (elapsed time = 0).
- 5.9 At five-minute (minimum) intervals, record the meter volume, elapsed time, inlet and outlet meter temperatures, ambient temperature, meter box ΔH , and meter vacuum. Once again, the meter must be read on the fly at precisely the time at which the elapsed time was recorded. Allow a minimum of 5 minutes between readings. More is OK, as long as the meter is read exactly at the time of the recorded time.
- 5.10 Repeat step 5.9 two more times for a total of three readings and at least 15 minutes of operating time.
- 5.11 If a spreadsheet or calculator program is available, input the data from the data sheet directly into the appropriate spreadsheet (or equivalent calculator program) to calculate the individual Y_i factors for each of the three 5-minute runs. Use these results to complete the data sheet, then proceed to Step 5.18.

If no automated program is available, the calculations may be done manually by following steps 5.12 through 5.17 below.



- 5.12 Calculate the net run time for each run by subtracting the elapsed time at the end of the run by the elapsed time at the end of the previous run. Record the net run time in decimal minutes to the nearest 0.1 minute. Note: If the elapsed time was recorded in minutes:seconds, convert to decimal minutes by dividing the seconds value by 60 and adding this value to the minutes value.
- 5.13 Calculate and record the average meter temperature for each run by averaging the inlet and outlet temperature readings recorded before and after each five minute period constituting the run (total of four temperature readings per average).
- 5.14 Calculate and record the net meter volume for each run by subtracting the initial volume from the final volume recorded for the run.
- 5.15 Calculate the Y_i factors using the following equation:

$$Y_i = \frac{K' \times P_b \times (T_m + 460) \times \theta}{17.64 \times V_m \times \left(P_b + \frac{\Delta H}{13.6}\right) \times \sqrt{T_{amb} + 460}}$$

where:

K'	=	critical orifice calibration factor
ΔH	=	average differential pressure across DGM orifice during run (inches W.C.)
P_b	=	barometric pressure (inches Hg)
θ	=	net duration of individual calibration run, minutes
T_{amb}	=	average ambient temperature during run (°F)
T_m	=	average meter temperature during run (°F)
Y_i	=	post-test meter calibration factor for run i.

- 5.16 Average the Y_i values obtained for the three runs using the following equation:

$$\bar{Y}_i = \frac{Y_1 + Y_2 + Y_3}{3}$$

- 5.17 Calculate the calibration error using the following equation:

$$Cal.Error = \frac{\bar{Y}_i - Y_d}{Y_d} \times 100$$

where Y_d is the full-test meter calibration factor.



5.18 An acceptable post test calibration error is $\pm 5\%$. If the post calibration exceeds this specification, then the following action must be taken.

- i) The calibration repeated on-site using an orifice that is second closest to the average ΔH sampled.
- ii) If the second calibration is out of specification, then the test leader must indicate on the calibration sheet that the meter is out of calibration. Upon return to the office, the meter will be post-test calibrated with the wet test meter. If this calibration is out of specification, then the lowest Y_d will be used for the emission calculations, and the meter will be full-test calibrated and adjusted if necessary.

5.19 Calculate the percent variation for each Y_i value using the following equation (performed automatically by computer program):

$$\Delta Y_i = \frac{Y_i - \bar{Y}_i}{\bar{Y}_i} \times 100$$

5.20 If the calibration error is acceptable, but the percent variation is greater than 2%, then repeat the calibration using the same orifice.

5.21 Upon completion of the calibration, document the results on the enclosed yellow meter calibration card. Also indicate any other notes or problems with the meter performance on this card. Return all meter post calibration data sheets to the Job Leader or the designated data assembly area.

6.0 ATTACHMENTS

The following attachments are included as reference:

- Blank Data Sheet CS 005 (Revision 10/15/95)
- Completed hand-written example of Data Sheet CS 005
- Computer Printout of completed data sheet CS 005



POSITIVE PRESSURE METER BOX LEAK CHECK

1.0 PURPOSE

This procedure describes the steps required to perform a positive pressure leak-check of a meter box system.

2.0 SCOPE

The positive pressure leak-check detects leaks which may occur between the pump and the outlet of the orifice meter of a standard meter box system. Leakage in this portion of the meter box may result in less volume being recorded than is actually sampled, or it may affect the accuracy of the orifice metering system. A leakage of this type cannot be detected using a standard negative leak-check procedure.

A positive pressure meter box leak-check should be performed once prior to use of any meter box which has been transported to a job site. This leak-check is also required prior to any meter box calibration, including both bench calibrations using a wet test meter and field calibrations using critical orifices.

3.0 MATERIALS

The following materials are needed to properly execute this procedure:

- standard isokinetic meter box console
- solid rubber stopper sized to fit the orifice outlet tube of the meter. (For most CAE Express-type boxes, a size 00 stopper works best, while a size 1 stopper fits most NuTech style boxes.)
- tubing pinch clamp
- (optional) spare manometer check-valve elbow fitting

4.0 DEFINITIONS

Refer to Figures 1 and 2 for definitions which describe specific meter box components.

Meter Box Console - a complete self-contained gas sampling system consisting of gas pump, dry gas meter (DGM), manometers, thermocouples, electrical system, and other associated hardware designed to sample a gas isokinetically. Two systems may be encountered in everyday use: the CAE Express-designed unit and a NuTech (Graseby-Andersen) unit (Model 2010 or equivalent).

Orifice Meter - an orifice mounted inside the outlet tube of the dry gas meter. The pressure drop across this orifice is known as the ΔH of the meter, and is used to control the gas flow rate through the sampling system.

Manometer - a dual inclined 8-inch manometer found on a meter box console. The manometer is used to measure both the ΔH (yellow oil) and the ΔP velocity head (red oil).

Leak-Check Pressurizing Tube (CAE Express Box only) - a tube used to pressurize the metering system for purposes of performing a positive pressure



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Field Sampling Procedures

Positive Press. Leak Check
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Effective Date: 10/15/95

Drafted By: JE Wright

Approved By: Process Owners

leak-check. This tube is located in the extreme upper right corner of the CAE Express console panel.

Course Control Valve - the main valve which controls the air flow into the meter box console.

+ Leak Check Power Switch (CAE Express Box Only) - a switch located on the console panel which, when activated, operates a solenoid valve which closes the low-pressure side of the orifice meter.

Valve Switch (CAE Express Box) or Null Switch (NuTech Box) - a switch located on the console panel which, when activated, operates a solenoid valve which isolates the ΔH column on the inclined manometer. This allows the ΔH manometer to be zeroed during meter box operation.

IGS Collection Tee - a copper fitting which slips over the outlet tube of the orifice meter. This tee allows an integrated gas sample (IGS) to be collected from the meter exhaust.

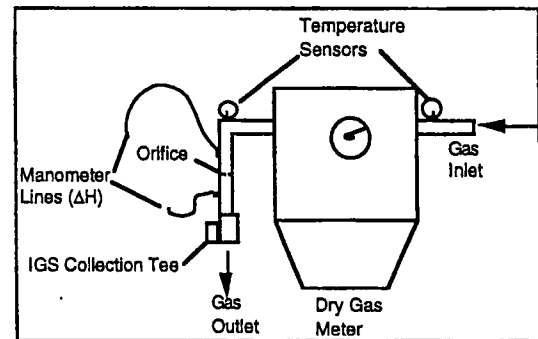
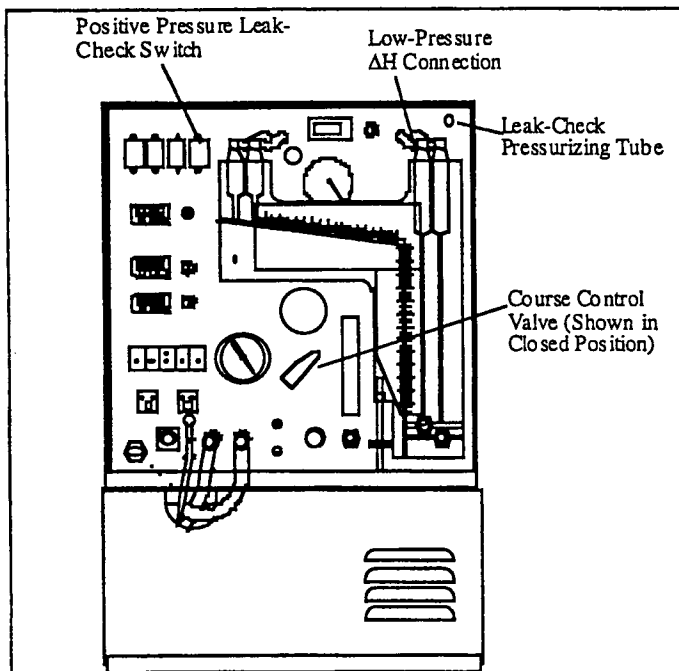


Figure 2 - DGM and Orifice Meter Schematic

Figure 1 - Isokinetic Meter Box Console (CAE Express Design)



Standard Operating Procedures

Clean Air Engineering

Field Sampling Procedures

Effective Date: 10/15/95

Drafted By: JE Wright

Positive Press. Leak Check
Field Project Management
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Approved By: Process Owners

5.0 PROCEDURE

- 5.1 Completely assemble the meter box according to standard procedures and verify the overall operability of the meter components (e.g., manometer, pump, etc.)
- 5.2 Unplug the main power cord of the meter console.
- 5.3 Remove the rear door of the console.
- 5.4 Slide the copper IGS collection tee off of the orifice meter tube.
- 5.5 Close the orifice tube opening using a solid rubber stopper inserted snugly inside the opening.
- 5.6 Plug the power cord of the unit in and turn the main power switch to the "ON" position and the pump switch to the "OFF" position.
- 5.7 Turn the Valve switch (CAE Express) or the Null switch (NuTech) to the "OFF" position.
- 5.8 If using a CAE Express box, turn the Leak-Check switch to the "ON" position.
- 5.9 Close the course control valve completely.
- 5.10 Disconnect the low side (right-hand side) of the manometer yellow gauge oil column. This can be done different ways, depending on the manometer design. If the manometer is equipped with quick release elbow fittings, and if a spare fitting is available, then temporarily replace the existing fitting with the spare fitting - do not connect the orifice tubing to the spare fitting.

If a spare elbow fitting is not available, or if the manometer is fitted with barb or compression fittings, then disconnect the actual tubing from the fitting, leaving the fitting in place.

- 5.11 Pressurize the meter box to 5 to 7 inches W.C. as follows:

- CAE Express Box: Place a tubing pinch clamp on the Leak-Check Pressurizing Tube and gently blow into the tube until the ΔH manometer registers to approximately 7 inches. Quickly close the pinch clamp to hold the pressure. If the pressure after closing the clamp is below 5 inches, then repeat the procedure until at least 5 inches is maintained.
- NuTech Box: Place a tubing pinch clamp on the loose low pressure ΔH tube and gently blow into the tube until the ΔH manometer registers to approximately 7 inches. Quickly close the pinch clamp to hold the pressure. If the pressure after closing the clamp is below 5 inches, then repeat the procedure until at least 5 inches is maintained.



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Field Sampling Procedures

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- 5.12 After pressurizing the box, allow 15 to 20 seconds for the oil in the manometer to drain to the meniscus, and note the exact pressure recorded at the bottom of the oil meniscus. Start the timer.
- 5.13 Watch the oil for 1 minute. A small piece of tape placed at the original oil level may make detection of movement easier. If no leaks are present, then the ΔH will remain steady. If any leaks are present, then the ΔH reading will decrease over time.
- 5.14 If no leakage of the system is observed in one minute, then the leak-check is acceptable.
- 5.15 If any amount of leakage is detected in one minute, then the system must be diagnosed to find the leak and repaired. It is recommended that the system be checked with a bubble-type leak detector fluid while it is under pressure. No testing with the meter box may proceed until a successful leak-check is performed. Potential areas in which leaks may commonly occur include:
 - rubber stopper used to close the orifice may not be snug
 - any fittings and connections between the pump and orifice meter exit
 - ΔH manometer valves, fittings and tubing
 - pump head
 - dry gas meter gasket
- 5.16 Upon successful completion of the leak-check, return the meter to its original working form by removing the rubber stopper (while unit is unplugged), replacing the IGS collection tee, reattaching the low pressure ΔH line, and turning the Leak-Check switch (if applicable) to the "OFF" position.

6.0 PROCESS FLOW DIAGRAM

Figure 3 summarizes the overall process flow for the procedural steps listed in Section 5.



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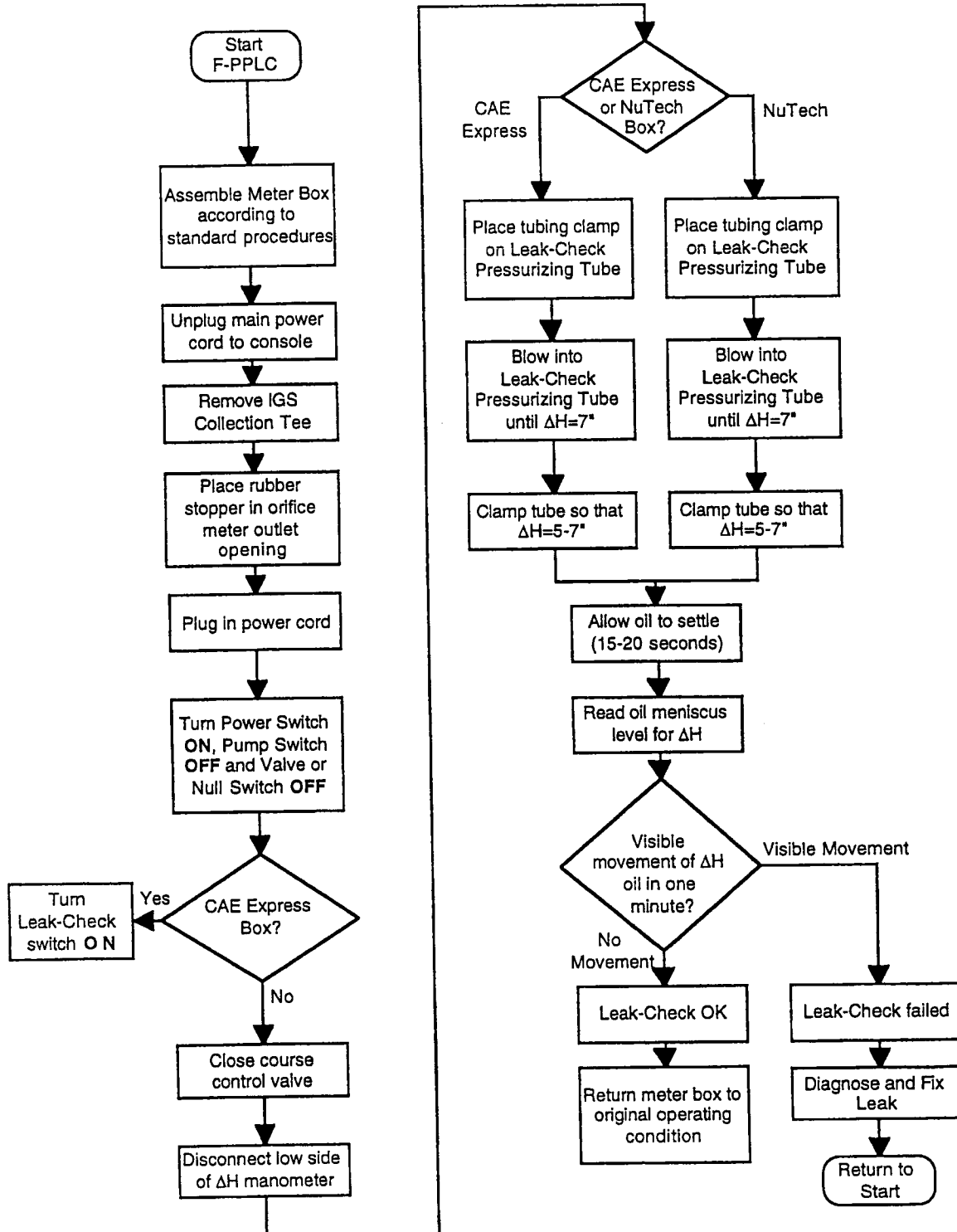


Figure 3 - Process Flow Diagram: Positive Pressure Leak-Check

CAE STANDARD OPERATING PROCEDURE

DATA SHEET COMPLETION

ISO NUMBER: FDC-1

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

- Purpose: The purpose of this procedure is to outline the required steps for completion of field data sheets, and to assign responsibility for completing data sheets.
- Scope: This procedure will be used for all field test data sheets.
- Materials: The materials needed for this procedure are as follows:
- The correct data sheet for the testing being conducted
 - A black pen
- Procedure: The following steps are taken by the field technician when completing a field test data sheet. The technician who records the test data is responsible for completing this procedure.

1. Complete the information boxes at the top of the data sheet before beginning the test run. Obtain the following from the test leader and transfer to the data sheet:
 - a. proper client, site, location, and method descriptions
 - b. CAE project number
 - c. run number
 - d. required number and duration for sampling points.
 - e. duct dimensions
 - f. nozzle and K factor information
 - g. barometric pressure
2. Sketch the **cross section** of the duct in the box provided. Show the duct outline and the locations of the ports. Circle the proper 'North' or 'Up' designator by the arrow. Also circle the gas flow direction and first point location designators.
3. Complete the test data section while sampling.
4. Add comments for any non-routine occurrences. Include leak checks, process upsets, mid-test start & stop times, train component changes, casual opacity observations, etc.
5. Each data sheet must be filled out completely, except for the exception noted below as item 7. Information that does not apply to the particular data set should be marked 'N / A'.
6. The technician must consult the test leader for instruction when unsure about any of the required information.

7. When multiple pages are used for one test, the first page top information section must be completely filled out. Consecutive pages require only that the plant information, run number, page number and data sections be completed.
8. After the test is completed the meter reader must average the data obtained.
9. The test leader is required to review each data sheet after the test. Any missing information should be brought to the meter readers attention for immediate correction.
10. All subsequent runs after the first must be completed using the same client, location, port & point designations.

CAE STANDARD OPERATING PROCEDURE

DATA SHEET COMPLETION – TOP SECTION

ISO NUMBER: FDC-2

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

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List of Materials : Traverse Data Sheet (DS 002 Velocity), pen, Job Plan

Definitions :

It is very important that all information is filled out on the top section of the Traverse data sheet. The following is a list of definitions and examples for information needed at the top of a traverse (DS002 Velocity) data sheet. A copy of a DS 002 Velocity data sheet is included.

1. Test Location - Test Location should be filled out for the location that the traverse is being done. A separate sheet should be used for each location. The test location designation should be consistent with the location name labeled in the job plan.
2. Unit - Document the unit being tested (e.g. Unit 1). Many plants have more than one unit therefore it is important to label the unit number.
3. Client - The Client Name from the job plan.
4. Plant - Name of plant or city where the plant is located. This should also be documented in the job plan.
5. Project No. - The job number listed on the job plan. The job number is usually in the format (Dept. - XXXX).
6. Date - The date that the traverse took is taking place
7. Meter Operator - Person reading the inclined manometer
8. Probe Operator - Person moving the traverse probe from point to point.
9. Source of Moisture and molecular Weight Data - Moisture and molecular weight data is needed to calculate the stack gas velocity. This space should note whether the molecular weight is being determined from an Orsat analysis or CEM (EPA Methods 3 and 3A respectively). Moisture information is obtained from EPA method 4, a wet bulb/dry bulb measurement or calculated from a saturated vapor pressure table.
10. Cross Section - This space is used for drawing a cross section of the location being tested. The cross section of the location is used in the final report to show the traverse points that were tested. Any notes or diagrams that can be drawn to define the orientation of the location is needed (e.g. Ladder locations or permanent CEM probe placements) Port designations should be consistent with the job plan and should remain the same from run to run. North or Up notations should also be made.

11. Amb. Temp (°F) - Ambient temperature reading taken during the traverse.
12. Barometric Pressure - Barometric pressure reading at the location during the traverse. This reading should be taken from a aneroid barometer or equivalent barometric watch. Pressure should be adjusted for location height in respect to where the reading was taken (e.g. Every 100 foot increase results in a Barometric pressure decrease of 0.1 inches of Hg. Conversely a 100 decrease shows a 0.1 inch of Hg increase in pressure.).
13. Pitot Cp - Both standard and Type S pitot tubes are used for velocity measurements. Type S pitot tubes are more frequently used and are assigned a coefficient of 0.84 if they meet calibration design specifications. Standard pitots are assigned a coefficient of 0.99. For further information on pitot tube types refer to the procedure, **Calibration of Pitot Tubes**.
14. Probe Id No. - The Probe Id. No. is the number assigned to each probe and is located on the back of the probe pitots or sheath. This number is important so that calibration sheets for the probes can be included in the final report.
15. Duct Diameters from Disturbance (Downstream) - This distance is the distance that the location being tested is from an expansion joint, elbow or other disturbance in the flow. (See Method 1 procedures for a more detailed description of duct diameters and disturbances).This information should be included in the job plan.
16. Duct Diameters from Disturbance (Upstream) - This information should be included in the job plan.
17. First Point all the Way - In this space circle whether the first traverse point sampled was located closest to the far wall of the duct (IN) or the near wall of the duct (OUT).
18. Gas Flow - According to orientation of the cross section that was drawn, document whether the flow of stack gas is in or out of the page.
19. Port Length - The measured length of the test port.
20. Duct Dimensions - The measured dimension of the duct in inches.
21. Run - The run number for the traverse.
22. Load - The load condition under which the traverse was conducted (e.g. high, low , 10% excess air etc.).
23. Start Time - The start time of the traverse.
24. Stop Time - The stop time of the traverse.
25. Static Pressure - The measured static pressure. See **Determining Static Pressure** procedure.
26. Post Test Leak Check - Mark in the appropriate box if the post test leak check passed or failed.

CAE STANDARD OPERATING PROCEDURE

MANAGEMENT OF CEM CONCENTRATION TEST DATA

ISO NUMBER: REP-1

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1.0 BACKGROUND

1.1 Scope

This SOP concerns the management of data and results obtained using any reference methods which utilize a continuous emissions monitor (CEM) and a digital data acquisition system. The SOP contains guidance concerning the control of the implicit and explicit accuracy of concentration data and results. The SOP affects the application of each of three major processes: data recording, data processing, and data reporting.

1.2 Applicability

This SOP applies to concentration data and results from EPA Reference Methods 3A, 6C, 7E, 10, 20 and 25A. A special clarification to this SOP which extends its applicability to relative accuracy results is included as an appendix to this document.

Data management for all other forms of data, including mass emission results derived from CEM concentration data, is covered under SOP H-GDM.

1.3 Basis

The SOP is based primarily on a compilation of implicit procedures which were in general use by various CAE departments as of April 1, 1995.

1.4 Limitations

Certain situations may arise in which application of this SOP may be inappropriate or undesirable. An example of this would be a client-specific request for data presentation which is inconsistent with the present SOP. In these cases, the Project Manager has ultimate authority to use his discretion concerning the specific application of the SOP.

1.5 Completeness and Revision

It is recognized that the SOP in this document may require modification in the future to address implications which are presently unforeseen. These situations will be incorporated into the overall SOP through revisions as they are addressed in the future. Any cases which are not adequately or appropriately covered by the present SOP should be brought to the attention of the Technical Director as they arise.

1.6 Definitions

The following definitions are implied in this SOP:

Raw CEM Data: The digital representation of data generated by a CEM system and stored in computer data files for a specific project.

CEM Run: a discreet period of time over which CEM data is collected for purposes of demonstrating compliance, performance, or accuracy.

Average Raw CEM Data: the arithmetic average of raw CEM data collected over a CEM run, excluding any non-sampling or calibration periods.

Calibration Drift Correction: The process of adjusting raw CEM data for calibration drift incurred during a CEM run. This adjustment is normally performed using Equation 6C-1 of EPA Reference Method 6C, and is performed on the average raw data over the run.

CEM Parameters: Information related to the collection and processing of raw CEM data. This information includes, but is not limited to, gas conditions (e.g., moisture, temperature, flow rate), calibration drift data, and calibration drift-corrected data.

CEM Emission Results: the concentration value of CEM-derived data which is shown in a table in the main body of a report. The result is normally derived after averaging the raw data over a specified time period, adjusting the average for calibration drift, and converting the number to a consistent moisture or diluent basis if necessary.

Significant Figure: A prescribed decimal place which determines the amount of rounding to be done. Also known as a significant digit.

Rounding: Neglecting decimals after some significant place, with the last significant decimal subject to alteration based on the first neglected decimal.

Truncating: Neglecting decimals after some significant place, with no change to the remaining portion of the number.

Formatting: To control a numerical value to a specified number of digits to the right of the decimal.

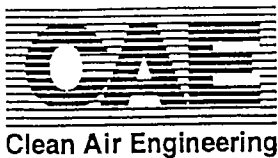
Standard: A federal- or state-enforceable limit on the emissions from or performance of a stationary source.

2.0 PROCEDURE

2.1 Data Recording

The following data formats should be used to collect and present raw CEM data:

Range of Result	Decimal Format to Record Raw Data	Decimal Format to Present Raw Data and Averages
ppm	x.xx (minimum)	x.x
%	x.xxx (minimum)	x.xx



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An exception to these formats may be made in situations where the calibrated range of the CEM is low enough such that additional decimal points may be justified to represent the actual accuracy of the measurements. In these cases, the reference materials used to establish the calibration curve for the instrument should be analyzed to an accuracy equivalent to the accuracy implied by the CEM data. For example, if the calibration range of the instrument is 0-1.00 ppm, then the calibration reference concentrations should be stated to the nearest 0.01 ppm.

2.2 Data Processing

Raw data will be truncated or rounded (depending on hardware/software characteristics) upon collection by the data acquisition system to the number of decimals specified in Section 2.1. All further processing of CEM data will retain all available decimals throughout all calculations involving these parameters. No internal truncations should be performed during these intermediate calculations.

2.3 Data Presentation

2.3.1 Tables of Results - all CEM concentration results will be presented using the following fixed decimal formats:

Range of Result	Decimal Format to Present Individual Runs	Decimal Format to Present Multiple-Run Averages
ppm	x (nearest whole number)	x (nearest whole number)
%	x.x	x.x

Two exceptions to the above formats may arise. The first exception involves the cases where the CEM is calibrated at a low enough range to imply additional accuracy, as discussed in Section 2.1. In these cases, the results should be presented to one decimal place less than that recorded in Section 2.1.

The second exception may arise in compliance situations in which the emissions standard is in terms of concentration or concentration corrected to a specific diluent level (e.g., ppm corrected to 7% O₂), or in CEMS certifications involving 40 CFR 75 in which all references to concentrations are to one decimal place. In these cases, the emission concentration for individual runs and multiple run averages should be presented to the same number of decimal places as that contained by the standard. (Note that this procedure differs from other SOP's as well as general EPA guidance concerning presentation of compliance results, in that the decimal is being fixed instead of the number of significant figures. This variance was deemed necessary in order to avoid overstating the accuracy of the CEM measurements.)

2.3.2 Executive Summary Tables - data presented in Executive Summary Tables will follow the same format as specified in Section 2.3.1.



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- 2.3.3 CEM Parameter Sheets - CEM Parameter Sheets will present all CEM concentration data and results using the presentation format specified in Section 2.1.
- 2.3.4 Presentation of Facility-Supplied CEMS Data - Facility data supplied for inclusion into a test report (e.g., CEMS certification data) will be presented in all report tables using the same decimal format as specified in Section 2.3.1. The data will be presented in its as-received format for inclusion in the report appendices.
- 2.3.5 Rounding - rounding of all numbers in report tables will follow the general rules as used by Excel. If the first digit to be discarded is less than five, then the last digit retained will not be changed. If the first digit to be discarded is five or greater, then the last digit retained will be increased by one unit. For example, 2.34 will become 2.3 when rounded to one decimal place. However 2.35 will become 2.4 when rounded to one decimal place.
- 2.3.6 Treatment of Negative Results - occasionally, the numerical value of CEM data or results may be less than zero. This may occur with the raw data, average raw data, or drift-corrected data. In these cases, the following guidelines should be used:
- 2.3.6.1 Raw CEM Data - all negative raw data (including facility-supplied data) will be presented as-collected with a preceding negative sign.
- 2.3.6.2 Average Raw CEM Data - averages of raw data which are negative will be presented as-calculated with a preceding negative sign.
- 2.3.6.3 CEM Emissions Results for Compliance - a negative final concentration result which is measured for compliance purposes should be reported in all tables as a whole number zero (0). All subsequent emission calculations and compliance determinations should also be zero. The results should be footnoted with the following comment:
"Negative CEM results are treated as zero in all calculations."
- 2.3.6.4 CEM Emissions Results for Relative Accuracy - a negative final concentration result which is measured for purposes of determining the relative accuracy of another CEM system should be reported in all tables as the actual negative number. All subsequent emission calculations and relative accuracy determinations should use the actual value of this number. However, if the average reference method value for all runs is negative, then the absolute value of this number should be used to calculate the relative accuracy - i.e., the relative accuracy should always be expressed as a positive value.
- 2.3.6.5 CEM Emissions Results Used for Compliance and Relative Accuracy - if the same CEM results are used for determining both compliance and relative accuracy, then negative results should be addressed separately for each comparison using the guidelines stated in 2.3.6.3 and 2.3.6.4.



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APPENDIX: Clarification to Extend Applicability to RATA Results

1.0 BACKGROUND

1.1 Scope

This document clarifies and expands upon the procedures for the management of data presented in relative accuracy test audit (RATA) reports.

1.2 Applicability

The clarifications in this document apply to any RATA report. The Reference Method data used in the report may be derived from either CEM or manual sampling methods.

1.3 Basis

This document is based on interpretation of SOP's H-GDM and H-CDM.

1.4 Limitations

The clarifications listed in this document are for guidance purposes only. The Project Manager has ultimate authority to use their discretion concerning the specific application of these guidelines.

1.5 Definitions

The following clarifications and additions to the original SOP definitions are made specifically in reference to RATA reports:

Applicable Emissions Standard: The applicable standard is normally a federal- or state-enforceable limit with which the facility or source must show compliance.

RATA Standard: The RATA standard is the federal- or state-enforceable limit on the relative accuracy of the CEMS.

Facility CEMS Results: Results obtained from the facility's continuous emissions monitoring system (CEMS) in units of the applicable emissions standard.

Reference Method Results: The results obtained from the Reference Method testing in units of the applicable emissions standard.

Final Result: The final result for a RATA is the relative accuracy itself.

Intermediate Result: In terms of RATA reports, all CEMS and Reference Method results are considered intermediate results. This also includes the differences, standard deviations, and confidence intervals calculated between the CEMS and Reference Method results.



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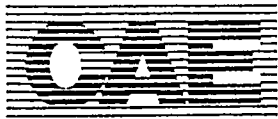
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2.0 CLARIFICATIONS

2.1 Presentation of Intermediate Results

The following guidelines apply to the procedure for presenting intermediate data in RATA reports:

Result	Presentation in Appendices	Presentation Format in RATA Tables
Facility CEMS Results	<ul style="list-style-type: none">• x.x for ppm results• x.xx for % results• x.xxx for lb/10⁶Btu results• x.x for lb/hr results• x.x for %removal results <p>* Note: If the data is supplied in hard-copy or a non-formattable electronic form, then include the data as given.</p>	<ul style="list-style-type: none">• x.x for ppm results• x.xx for % results• x.xxx for lb/10⁶Btu results• x.x for lb/hr results• x.x for %removal results <p>* Note: If the original data does not have enough accuracy to the right of the decimal to satisfy the above criteria, then present the data as given.</p>
Reference Method Results	<ul style="list-style-type: none">• x.x for ppm results• x.xx for % results• x.xxx for lb/10⁶Btu results• x.x for lb/hr results• x.x for %removal results	<ul style="list-style-type: none">• x.x for ppm results• x.xx for % results• x.xxx for lb/10⁶Btu results• x.x for lb/hr results• x.x for %removal results
Difference between CEMS results and RM results	N/A	<ul style="list-style-type: none">• x.x for ppm results• x.xx for % results• x.xxx for lb/10⁶Btu results• x.x for lb/hr results• x.x for %removal results
Average of Reference Method runs	N/A	<ul style="list-style-type: none">• x.x for ppm results• x.xx for % results• x.xxx for lb/10⁶Btu results• x.x for lb/hr results• x.x for %removal results
Standard deviation of differences	N/A	x.xxxx
Confidence intervals	N/A	x.xxxx



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2.1 Presentation of Final Results

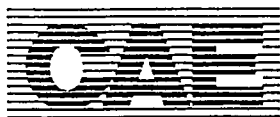
The final relative accuracy results in RATA reports should be presented to the same number of significant figures as contained by the Relative Accuracy Standard. For facilities which are regulated under 40 CFR 60, this implies that the relative accuracy results should be presented with two significant figures. For facilities regulated under 40 CFR 75, the results should be presented using three significant figures.

These guidelines result in the following decimal formats for presentation of relative accuracies:

Magnitude of Relative Accuracy	40 CFR 60 Facilities	40 CFR 75 Facilities
< 1%	0.xx	0.xxx
1%-9.99%	x.x	x.xx
≥ 10%	xx (no decimals)	xx.x

For facilities which do not fall under either Part 60 or Part 75, the specific Relative Accuracy Standard as stated in the source permit should be used to determine the presentation format of the relative accuracy results.

For sources at which the relative accuracy is based on an absolute emissions value instead of a percentage, the level of accuracy stated for the limit in the regulation should be used to determine the presentation format of the relative accuracy results.



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1.6 Definitions

The following definitions are implied in this SOP:

Significant Figure: A prescribed decimal place which determines the amount of rounding to be done. Also known as a significant digit.

Rounding: Neglecting decimals after some significant place, with the last significant decimal subject to alteration based on the first neglected decimal.

Truncating: Neglecting decimals after some significant place, with no change to the remaining portion of the number.

Formatting: To control a numerical value to a specified number of digits to the right of the decimal.

Emission Results: Results of an emission or performance nature for specific pollutants or control equipment. Examples - particulate concentration, organic compound mass emissions, incinerator destruction and removal efficiency (DRE).

Standard: A federal- or state-enforceable limit on the emissions from or performance of a stationary source.

Final Result: The result of an emissions test which is directly comparable to the Standard. Example - the average particulate concentration result from three EPA Method 5 test runs.

Parameter: Data and results which are presented as supplementary information to the final result. Examples - gas flow rate, moisture content, temperatures.



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2.0 PROCEDURE

2.1 Data Recording

The following guidelines apply to the procedure for collecting data during manual sampling tests such as EPA Methods 1 through 5 and other related methods. Field data should be recorded using the accuracy criteria shown below. Field calculations such as averages and net volumes should be recorded using the fixed decimal formats as shown.

Parameter	Accuracy to Record Raw Data	Decimal Format to Record Calculated Averages
Velocity Head - ΔP (in. WC)	<ul style="list-style-type: none"> 0.005" if $\Delta P \leq 0.1$" 0.01" if $0.1 < \Delta P \leq 1.0$" 0.1" if $\Delta P > 1.0$" 	N/A
Square Root of ΔP	N/A	x.xxxx
Orifice ΔH (in. WC)	<ul style="list-style-type: none"> 0.005" if $\Delta H \leq 0.1$" 0.01" if $0.1 < \Delta H \leq 1.0$" 0.1" if $\Delta H > 1.0$" 	x.xxxx
Meter Volume	<ul style="list-style-type: none"> 0.005 dcf for stopped meter readings (e.g., start, stop, and leak-check readings) 0.01 dcf for readings during meter operation 	x.xxx (for differences)
Temperature from Thermocouple (e.g., stack, meter)	Nearest whole degree ($^{\circ}F$ or $^{\circ}C$)	x.xxxx
Vacuum	Nearest whole inch Hg	N/A
Orsat Data (O_2 and CO_2)	0.1%	x.x
Impinger liquid gain	0.5 gram or nearest whole milliliter	x.x (for differences)
Static Pressure	0.1 in. WC	x.x
Barometric Pressure	0.01 in. Hg	x.xx
Meter Box Y_d	0.0001	x.xxxx
Meter Box $\Delta H @$	0.0001	x.xxxx
Duct Dimensions (to be measured exclusively in inches)	0.1 inch	x.x
Duct Area (ft^2)	N/A	x.xxxx
K-Factor	<ul style="list-style-type: none"> 0.1 for $K \geq 1.0$ 0.01 for $K < 1.0$ 	N/A
Leak Rate	0.001 cfm	N/A
Pitot C_p	0.01	N/A



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2.2 Data Processing

All parameters will retain all available decimals throughout all calculations involving these parameters. No internal truncation of any parameter will be performed.

2.3 Data Presentation

2.3.1 Tables of Results

2.3.1.1 Emission Results

2.3.1.1.1 Compliance Emission Results (except DRE) - the presentation of compliance emission results (except DRE) will be determined by the number of significant figures contained by the emission standard for the pollutant of interest. Compliance results for individual runs should be presented with one more significant figure than that of the emission standard. By definition, the EPA has defined emission standards to contain either two or three significant figures (refer to Attachment 1). Therefore, the individual run emission results will contain either three or four significant figures, depending on whether the standard has two or three significant figures, respectively.

The multiple-run average for compliance emission results will contain exactly the same number of significant figures as the standard (i.e., either two or three significant figures).

The above rules will apply to results in all units within a pollutant subset. For example, the emission standard may be in terms of grains/dscf only. However, if lb/hr and mg/dscm are also presented for this pollutant, then the number of significant figures for these results would still be governed by those of the standard.

2.3.1.1.2 Non-Compliance Emission Results (except DRE) - if the emission results concern a pollutant or parameter which is not being determined for comparison to a standard, then four significant figures will be used for individual runs, and three significant figures will be used for multiple run averages.

2.3.1.1.3 DRE Results - the presentation of destruction and removal efficiency (or similar results) will be determined by the number of decimal places contained in the applicable performance standard. Both individual run and multiple-run averages will be presented using one additional decimal place than that contained by the standard.

2.3.1.2 Parameters - all parameters contained in the Tables of Results will be presented in the same fixed-decimal format as specified below in Section 2.3.3.2. Additionally, values for volumetric flow rates (Q_a and Q_{std}) will be converted to a maximum of four significant figures for each run and multiple-run averages. The following equation will be used to perform this conversion:



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$$Q_i = \text{ROUND}[Q_i, (3 - \text{TRUNC}(\text{LOG}_{10} Q_i))] \quad (\text{rounds to 4 sig. figs.})$$

This conversion will be performed for display purposes only. Any and all calculations which involve volumetric flow rate will utilize the untruncated form of the flow rate parameters.

- 2.3.1.3 Conversion to Scientific Notation - the default for presentation of all parameters and emission results in Results Tables is standard decimal format. However, if the magnitude of the number is small enough so that more than two zeros are required to the right of the decimal to place the first non-zero digit, then standard scientific notation will be employed for all results for that parameter. For example, if the numerical results for Run 1 are 0.00034, then this value will be presented as 3.4×10^{-4} . Also, all other runs as well as the multiple-run average for this parameter must also be presented in scientific notation, regardless of the actual magnitudes of these other values.

The format for scientific notation will be of the floating exponent type. This means that there will always be exactly one non-zero digit to the left of the decimal, and the scientific exponent will be adjusted accordingly.

2.3.2 Executive Summary Tables

2.3.2.1 Emission Results

- 2.3.2.1.1 Compliance Emission Results (except DRE) - Compliance emission results (except DRE) in the executive summary will have the same number of significant figures as that of the standard. They will also be in the same decimal format (e.g., standard decimal or scientific notation) and units as those of the standard.

- 2.3.2.1.2 Non-Compliance Emission Results (except DRE) - if the emission results concern a pollutant or parameter which is not being determined for comparison to a standard, then three significant figures will be used to present these numbers in the executive summary table. These results will be in the same decimal format as that presented in the corresponding Table of Results.

- 2.3.2.1.3 DRE Results - the presentation of destruction and removal efficiency (or similar results) will be determined by the number of decimal places contained in the applicable performance standard. The results will be presented using one additional decimal place than that contained by the standard.

- 2.3.2.2 Parameters - any parameters contained in the executive summary table will be presented in the same fixed-decimal format as specified in Section 2.3.3.2.



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2.3.3 Parameter Sheets

2.3.3.1 Segregation of Emission Results - parameter sheets designated for velocity and moisture parameters will include only individual run values for the "Sampling Conditions" and "Flow Results". They will not include emission results or run averages. If emission results are to be included in the parameter sheets, then each different type of emission should be summarized on separate parameter sheet formats (e.g., CEM Parameters, PCDDs/PCDFs Parameters, etc.)

2.3.3.2 Fixed Decimal Format - all parameters will be reported using a fixed-decimal format. The decimal formats which should be used as defaults in all parameter workbooks and spreadsheets are shown below.

Parameter	Description	Fixed Decimal Format
Y _d	Dry gas meter correction factor	x.xxxx
C _p	Pitot tube coefficient	x.xx
P _g	Static pressure	x.x
A _s	Sample location area	x.xx
P _{bar}	Barometric pressure	x.xx
D _n	Nozzle diameter	x.xxx
O ₂	Oxygen	x.x
CO ₂	Carbon Dioxide	x.x
V _{lc}	Liquid collected	x.x
V _m	Volume metered, meter conditions	x.xxx
T _m	Dry gas meter temperature	x
T _s	Sample temperature	x
ΔH	Meter box orifice pressure drop	x.xx
Θ	Total sampling time	x
V _{wstd}	Volume of water collected	x.xx
V _{mstd}	Volume metered, standard	x.xxx
P _s	Sample gas pressure, absolute	x.xx
P _v	Vapor pressure, actual	x.xx
B _{wo}	Moisture in sample	x.xx
B _{ws}	Saturated moisture	x.xx
√ΔP	Velocity head	x.xxx
M _d	MW of sample gas, dry	x.xx
M _s	MW of sample gas, wet	x.xx
V _s	Velocity of sample	x.x
%I	Isokinetic sampling	x.x
Q _a	Volumetric flow rate, actual	x
Q _{std}	Volumetric flow rate, standard	x
Mn, C, E	General VOC/SVOC emissions	x.xx E ^{xx} (scientific)



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- 2.3.4 Rounding - rounding of all numbers in report tables will follow the general rules as used by Excel. If the first digit to be discarded is less than five, then the last digit retained will not be changed. If the first digit to be discarded is five or greater, then the last digit retained will be increased by one unit. For example, 2.34 will become 2.3 when rounded to two significant figures. However 2.35 will become 2.4 when rounded to two significant figures.



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ATTACHMENT 1 - EPA Memorandum

June 10, 1990

MEMORANDUM

SUBJECT: Performance Test Calculation Guidelines

FROM: William G. Laxton, Director
Technical Support Division, OAQPS (MD-14)

John S. Seitz, Director
Stationary Source Compliance Division, OAQPS (EN-341)

TO: New Source Performance Standards/National Emission
Standards for Hazardous Pollutants Compliance Contacts

The following guidelines should be used in calculating and reporting emission rates and concentrations when determining compliance with the new source performance standards (NSPS) and national emission standards for hazardous pollutants (NESHAP). These guidelines can also be used for State implementation plans (SIP's). The areas addressed in this memorandum concern metric and English measurement systems, significant figures (SF's) in the emission standards, SF's to carry in intermediate calculations, and the rounding of final emission value numbers to the proper SF's.

1. Use only the emission standard in the metric units to determine compliance.

The policy of using the metric system was established back in the early 1970's. When the Environmental Protection Agency (EPA) proposed standards for seven source categories (38 FR 15406) on June 11, 1973, EPA stated:

"The Environmental Protection Agency has adopted a policy of expressing standards in the metric rather than English system. Although technical terms in tests methods 10 and 11 are expressed in metric units, many of those in test methods 1 through 9 are expressed in English units. Test results derived through calculations in test methods 1 through 9 must be converted to metric units to agree with the form of the proposed standards."



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In keeping with this policy, EPA promulgated amendments to Subparts D, E, F, G, and H on June 14, 1974 (39 FR 20790). In the preamble of this rulemaking, EPA stated:

".... Also, to be consistent with the Administrator's policy of converting to the metric system, the standards of performance and other numerical entries, which were originally expressed in English units, are converted to metric units. Some of the numerical entries are rounded after conversion to metric units. It should be noted that the methods in the appendix will be changed to metric units at a later date."

The change to metric units for the test methods were proposed on June 8, 1976 (41 FR 23060) and promulgated on August 18, 1977 (42 FR 41754). Clearly, EPA's intent was to use the metric units to determine compliance.

However, on a practical scale, the use of the metric or the English system of units would not make any difference in determining whether a source is in compliance or out-of-compliance. Only in very rare cases will separate calculations in the metric and in the English systems with the same source test measurement values result in one system showing compliance while the other does not. Therefore, it is not necessary to require source testing firms to submit the results in the metric units on a routine basis. It is suggested that if the value in English units is within 1 percent of the emission standard and if such a difference is of concern in your enforcement strategy, then the value should be converted to metric to determine compliance. The numerical value in English units (in parentheses) are to be considered as close approximations of the metric and should not be used to determine compliance in borderline cases.

2. Consider all emission standards to have at least two SF's, but no more than three SF's.

As a review, a SF is any digit that is necessary to define the specific value or quantity. Zeros may be used either to indicate a specific value, like any other digit, or to indicate the magnitude of a number. Examples are given below:

- Ex. 1: 1300 or 1.3×10^3 has two SF's.
- Ex. 2: 1300. or 1.300×10^3 has four SF's.
- Ex. 3: 1300.0 or 1.3000×10^3 has five SF's.
- Ex. 4: 13040 or 1.304×10^4 has four SF's.
- Ex. 5: 0.034 or 3.4×10^{-2} has two SF's.
- Ex. 6: 0.03400 or 3.400×10^{-2} has four SF's.
- Ex. 7: 0.03 or 3×10^{-2} has one SF.



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Because the emission standards were not written with consideration to the rules of SFs, especially with the use of zeros, all existing emission standards are considered to have at least two SF's, but no more than three SF's, under this guideline. Thus:

- Case 1: 90 mg/dscm (Subpart I) is considered to be 90. (two SF's).
- Case 2: 520 ng/J (Subpart Da) is considered to be 520. (three SF's).
- Case 3: 0.05 kg/Mg (Subpart S) is considered to be 0.050 (two SF's).
- Case 4: 0.1 g/kg (Subpart BB) is considered to be 0.10 (two SF's).
- Case 5: 0.005 g/kg (Subpart BB) is considered to be 0.0050 (two SF's).
- Case 6: 3870 kg/28 days (Subpart BBB) is considered to have (three SF's).

The above rule differs from the previous guidance given by the predecessor of the Stationary Source Compliance Division. In his

August 20, 1980 memorandum to Ms. Louise Jacobs, Director of Enforcement Division of Region VII, Mr. Edward E. Reich, Director of the Division of Stationary Source Enforcement interpreted the emission standards as being absolute, i.e., as having an infinite number of SF's. Using the example of an emission standard of 0.04 gr/dscf, Mr. Ed Reich wrote:

"As a legal matter, anything greater than 0.04 is a violation (e.g., 0.0401). However, since the third digit was not established, most engineers in reporting results would tend to round off and therefore 0.044 would be reported as 0.04 and 0.045 would be reported as 0.05. A better guide would be that anything showing greater than a ten percent excess is worth considering for enforcement action."

Although the new guidance appears to be a major shift in Agency policy, it is not because only very limited cases, if any, would be involved. For example, if the emission standard is 90 mg/dscm, 90.0000001 mg/dscm would be in violation according to the August 20, 1980 guidance. However, such an occurrence would be highly unlikely. (Note also that strict adherence to an infinite number of SF's would require an infinite number of SF's in conversion factors, which is a practice that is impossible to follow.) Therefore, the effect of the change to two or three SF's in the emission standards would be practically no different from the initial guidance.

3. Carry at least five significant digits in intermediate calculations.

Since the measurement of variables and sample quantities during source performance tests are recorded in metric or English units or a combination of both, specifying specific rules to handle SF's in addition, subtraction, multiplication, and division would become cumbersome. To keep things on a practical basis for emission standards with two or three SF's, English or metric may be used provided that at least five SF's are retained (most



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calculators retain nine digits) in all intermediate calculations. The final calculation after averaging all the runs should produce the metric units, if necessary. Then the final number should be rounded off as described below to determine compliance.

4. Round off calculated emission numbers to the number of SF's determined by the rule stated in (2) above.

When rounding off a figure, the following procedure, based on practices given under the American Society for Testing and Materials (ASTM) (Standard for Metric Practice E 380), should be used: If the first digit to be discarded is less than five, the last digit retained should not be changed. When the first digit discarded is greater than five, or if it is a five followed by at least one digit other than 0, the last figure retained should be increased by one unit. When the first digit discarded is exactly five, followed only by zeros, the last digit retained should be rounded upward if it is an odd number, but no adjustment made if it is an even number.

For example, if the emission standard is 90, then 90.357 would be rounded to 90, 90.639 would be rounded to 91, 90.500 would be rounded to 90, and 91.500 would be rounded to 92.

Consideration was given to round upward when the last digit to be discarded is five; however, because the occurrence of the first digit to be discarded being exactly five followed by zeros is rare, it was decided to be consistent with the ASTM practice.

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CAE STANDARD OPERATING PROCEDURE

Detection Limit Reporting Policy

ISO NUMBER: REP-3

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

This document summarizes the procedures to be used for treating detection limits in CAE reporting schemes.

The matrices below are representative of a sampling train consisting of front and back-half fractions. Methods with additional fractions would be treated similarly.

Note: D => amount was detected at a value greater than the detection limit.
ND => amount was not detected; ND represents the value of the detection limit.

Detection Limit Treatment for Determining Emissions from a Single Run

Front Half	Back Half	Total
D_{fh}	D_{bh}	$D_{fh} + D_{bh}$
D_{fh}	ND_{bh}	D_{fh}
ND_{fh}	D_{bh}	D_{bh}
ND_{fh}	ND_{bh}	"<" $MAX(ND_{fh}, ND_{bh})$

Detection Limit Treatment for Determining Average of 3 Runs

Run 1	Run 2	Run 3	Average
D_1	D_2	D_3	$(D_1 + D_2 + D_3)/3$
D_1	D_2	ND_3	"<" $(D_1 + D_2 + ND_3)/3$
D_1	ND_2	ND_3	"<" $(D_1 + ND_2 + ND_3)/3$
ND_1	ND_2	ND_3	"<" $(ND_1 + ND_2 + ND_3)/3$

Detection Limit Treatment of Analytical Blank Corrections

Scenario	Action
Sample Fraction = ND	No blank correction
Blank = ND	No blank correction
Sample Fraction - Blank > 0	Corrected Sample = Sample Fraction - Blank
Sample Fraction - Blank = 0	Corrected Sample = ND
Sample Fraction - Blank < 0	Corrected Sample = ND

CAE STANDARD OPERATING PROCEDURE

TREATING DETECTION LIMITS AND BLANK CORRECTIONS IN THE EPA METHOD 29 DATA ANALYSIS

ISO NUMBER: REP-4

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

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The following procedures were used for treating detection limits and blank corrections in the EPA Method 29 data analysis.

1. Blank Correction Procedures

Note: $M_{f_{hb}}$ = front-half blank
 $M_{f_{bb}}$ = back-half blank
 $M_{f_{h}}$ = front-half sample
 $M_{f_{b}}$ = back-half sample

A. Determining allowable blank correction.

Front Half (all metals except mercury)

- a) If $M_{f_{hb}} \leq 11.46$, then allowable correction = $M_{f_{hb}}$.
- b) If $M_{f_{hb}} > 11.46$, then allowable correction is the greater of:
 - i) 11.46
 - ii) the lesser of $M_{f_{hb}}$ and 5% of $M_{f_{h}}$

Back Half (all metals except mercury)

- a) If $M_{B_{hb}} \leq 1.0$, then allowable correction = $M_{B_{hb}}$.
- b) If $M_{B_{hb}} > 1.0$, then allowable correction is the greater of:
 - i) 1.0
 - ii) the lesser of $M_{B_{hb}}$ and 5% of $M_{B_{h}}$

IF POOLED SAMPLE USE BACK HALF CRITERIA.

Mercury

- a) If $M_{f_{hb}} + M_{B_{hb}} \leq 6.0$, then allowable correction = $M_{f_{hb}} + M_{B_{hb}}$
- b) If $M_{f_{hb}} + M_{B_{hb}} > 6.0$, then allowable correction is the greater of:
 - i) 6.0
 - ii) 5% of $M_{f_{hb}} + M_{B_{hb}}$

B. Detection Limit Treatment of Analytical Blank Corrections.

Note: D => amount was detected at a value greater than the detection limit.
 ND => amount was not detected; ND represents the value of the detection limit.

Scenario	Action
Sample Fraction = ND	No blank correction
Blank = ND	No blank correction
Sample Fraction - Blank > 0	Corrected Sample = Sample Fraction - Allowable Blank
Sample Fraction - Blank = 0	Corrected Sample = ND
Sample Fraction - Blank < 0	Corrected Sample = ND

2. Detection Limit Procedures

Note: D => amount was detected at a value greater than the detection limit.
 ND => amount was not detected; ND represents the value of the detection limit.

A. Detection Limit Treatment for Determining Emissions from a Single Run

Front Half	Back Half	Total
D_{fh}	D_{bh}	$D_{fh} + D_{bh}$
D_{fh}	ND_{bh}	D_{fh}
ND_{fh}	D_{bh}	D_{bh}
ND_{fh}	ND_{bh}	"<" $MAX(ND_{fh}, ND_{bh})$

B. Detection Limit Treatment for Determining Average of 3 Runs

Run 1	Run 2	Run 3	Average
D_1	D_2	D_3	$(D_1 + D_2 + D_3)/3$
D_1	D_2	ND_3	"<" $(D_1 + D_2 + ND_3)/3$
D_1	ND_2	ND_3	"<" $(D_1 + ND_2 + ND_3)/3$
ND_1	ND_2	ND_3	"<" $(ND_1 + ND_2 + ND_3)/3$

CAE STANDARD OPERATING PROCEDURE

REPORT NAMING PROTOCOL

ISO NUMBER: REP-5

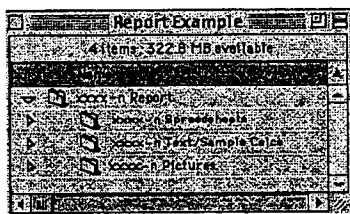
AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

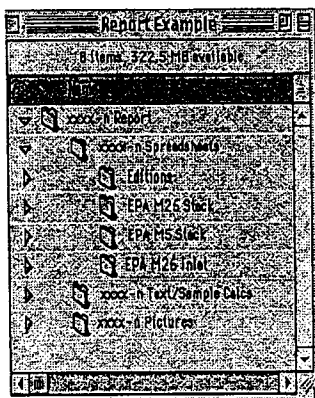
APPROVED BY: Pending

All report subfolders and their contents should use a consistent naming convention which references the job number. The conventions shown below should be used. (In all cases "xxxx" represents the 4-digit job number related to the project, and "-n" relates to separate report identification for multiple reports under one project.)

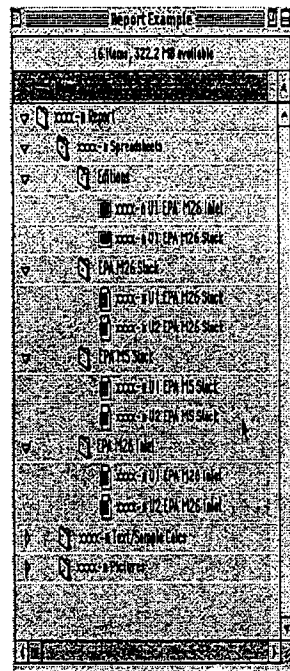
Report Name: All names for folders and files related to the report should be preceded with the 4-digit job number, followed by "Report". A "-n" suffix should be added to the job number if multiple reports are to be issued. For example, if two reports for job 8282 are to be created, then the files should be named 8282-1 Report and 8282-2 Report. If only one report is to be issued, do not use the suffix.



Spreadsheets: Individual folders should then be made to coincide with each EPA method performed for that particular job. These folders should identify the EPA method and location. Separate folders should be made for each method and location (e.g., Stack or Inlet).

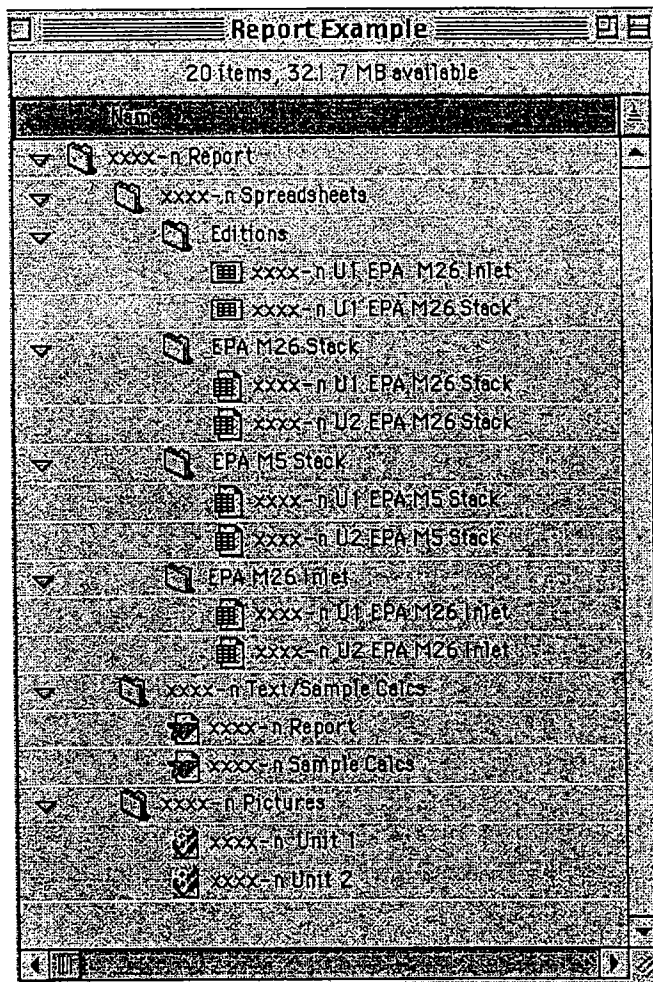


Spreadsheets and Editions within these folders should reference the job number (with the “-n” suffix if necessary) along with unit name (abbreviated as “U”), method and location, respectively.



Text: A separate folder should be made to hold the report text and sample calculations. These will be Word documents. The folder itself should reference the job number (again with the “-n” suffix if necessary) followed by Text/Sample Calcs. Each Word document will also reference the job number as shown below.

Pictures: A separate folder should also be made to hold pictures related to the project. The folder should reference the job number (again with the “-n” suffix if necessary) followed by Pictures. Each picture file should reference the job number along with a description of the picture (e.g., xxxx-n Unit 1).



CAE STANDARD OPERATING PROCEDURE

TEST REPORT REVIEW

ISO NUMBER: REP-6

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

Expectations:

- Entire document spell checked
- Entire document grammar checked
- All calculations checked
- All items complete (Lab data, Certification sheets, Client data etc.)
- All figures, drawings correct and referenced properly
- Table of contents correct

Things to check as a reader:

- Overall feel of report
- Consistency from report to report
- Methods referenced correctly and completely
- Limits referenced correctly
- Correct Regulations referenced properly (i.e. BIF, Part 60, Part 75)
- Completeness of data
- Completeness of calibrations and procedures
- Sample calculations

Step by Step Procedure:

- Title Page information correct
- Table of contents correct, references to page numbers correct
- Project Overview makes sense
- Data in summary of test results correct
- Rata table show correct units (i.e. @ 7%, @ 12%)
- Start times and date correct
- Limits correct
- Correct run was eliminated from calculations
- Calibration Error and Response Time Data correct
- Correct calculations in Calibration Error and Response Time
- Correct limits for Calibration Error and Response Time
- Seven day drift data correct
- Description of installation makes sense and is correct
- Diagram of process is correct
- Methodology is correct and referenced properly
- Listing of analyzers is correct
- Diagram of RM system is correct

Calibration gases used are correct
Appendix references are correct
Sample calculations are correct
Parameters are correct
Parameters have correct start and stop time
Calibration Data contains the correct calibration gases that were used
Calibration worksheet lists all calibrations, all calibration below limits
Field data complete and accurate
Field data printouts have correct calibrations
Field data printouts have correct times for every run
Monitor data is complete and is referencing the correct time for every run
Seven Day Drift data is complete and accurate

Section 2 General Report Process

2.1 General

2.1.1 Aims and Responsibilities

It is essential to understand the functioning of the reporting system as a process. The overall reporting process is actually several sub-processes working together. Each sub-process has an individual aim but it is essential that all sub-processes work together to achieve a common aim.

The overall aim of the reporting process is:

To provide the client with information of sufficient completeness, accuracy, relevance and timeliness and in such a format as to allow the client to achieve their objectives and to provide this information in an increasingly consistent and efficient manner.

This aim is also given to the Project Manager since he/she is responsible for oversight of the entire reporting process.

In this section, the sub-processes are defined by job titles -- Field Technician, Test Leader, Project Manager, Technical Writer, and Salesperson--because these are well known functions within CAE. It should be understood that the process referred to is not the individual but the process in which the individual is engaged.

It is also important to note that each process must be concerned both with the quality of input from the supplier and the quality of output to the customer. The saying "Garbage In - Garbage Out" has particular relevancy here.

2.1.1.1 THE FIELD TECHNICIAN

(a) Aim

To acquire, organize, and quality assure data which meet project objectives in an increasingly consistent and efficient manner.

(b) Input-Supplier

Information on client and project objectives and procedures-
Test Leader
Test equipment and supplies- Test Leader
Training on procedures, test methods, data handling -
Training Dept.

(c) Output-Customer

Data sheets - Test Leader

Lab Samples - Test Leader
Field log - Test Leader
Ideas and suggestions - Test Leader

(d) Responsibilities

- Understand client and project objectives.
- Understand the nature of the process and/or control device.
- Understand the operation of all equipment and instrumentation used to acquire data.
- Correctly execute test methods used to acquire data.
- Anticipate potential site-specific sampling problems.
- Distinguish between "good" and "bad"; "reasonable" and "unreasonable"; and "relevant" and "irrelevant" data
- Take appropriate corrective actions when bad, unreasonable, or irrelevant data is identified.
- Verify that proper data sheets/data formats are used to acquire data.
- Supply all information requested on data sheets.
- Write neatly and legibly.
- Line-out and initial and corrections to data sheet.
- Maintain a field log detailing activities, problems encountered, solutions found.
- Perform and record all QC activities on Level 1 data.
- Perform and record all other applicable QC procedures.
- Organize data according to requirements of next customer.
- Identify and suggest process improvements and innovations.

2.1.1.2 THE TEST LEADER

(a) Aim

To collect, organize, and quality assure all relevant on-site data to meet project objectives in an increasingly consistent and efficient manner.

(b) Input - Supplier

Information on client and project objectives and procedures-
Project Manager
Test Equipment and Supplies - Packing and Shipping Dept.
Travel Arrangements - Travel Dept.
Training on procedures, test methods, data handling -
Training Dept.
Data Sheets - Field Technician
Field Log - Field Technician
Operating data - Client
Ideas and Suggestions - All customers and suppliers

(c) Output - Customer

Organized, quality assured data packet - Project Manager
Field Logs - Project Manager
Preliminary Data - Client
Lab samples - Laboratory
Process improvements - All customers and suppliers

(d) Responsibilities

- Perform all functions necessary to ensure that Field Technicians and other suppliers can meet their responsibilities and fulfill their aim.
- Understand client and project objectives.
- Understand the nature of the process and/or control device.
- Understand the test methods used to acquire data.
- Understand the operation of all equipment and instrumentation used to acquire data.
- Anticipate potential site-specific sampling problems.
- Distinguish between "good" and "bad"; "reasonable" and "unreasonable"; and "relevant" and "irrelevant" data
- Take appropriate corrective actions when bad, unreasonable, or irrelevant data is identified.
- Perform quality assurance check on Level 2 data.
- Take appropriate corrective action if Level 2 fails quality assurance checks
- Maintain continuing client contact during the project to ensure that client objectives continue to be met.
- Collect any relevant on-site data not acquired by Field Technicians (i.e. process data).
- Reduce data as required in job plan.
- Perform and record all QC activities on Level 1 data.
- Perform and record all other applicable QC procedures.
- Maintain a field log detailing activities, problems encountered, solutions found.
- Organize data according to requirements of next customer.
- Identify and suggest process improvements and innovations.

2.1.1.3 THE PROJECT MANAGER

(a) Aim

To provide the client with information of sufficient completeness, accuracy, relevance and timeliness and in such a format as to allow the client to achieve their objectives and to provide this information in an increasingly consistent and efficient manner.

(b) Input - Supplier

Information on client needs and objectives - Salesperson,
Client

Organized, quality assured data packet - Test Leader
Field Logs - Test Leader
Laboratory data - Laboratory
Training on procedures, test methods, data handling -
Training Dept.
Ideas and suggestions - All customers and suppliers

(c) Output - Customer

Job plan - Test Leader
Report packet - Technical Writer
Report - Client
Process improvements - All customers and suppliers

(d) Responsibilities

- Perform all functions necessary to ensure that Test Leaders, Technical Writers and other suppliers can meet their responsibilities and fulfill their aim.
- Understand the client's needs and objectives for the project.
- Define and communicate project objectives.
- Define project-specific responsibilities for all project participants.
- Establish project timeline with appropriate milestones.
- Understand the nature of the process and/or control device.
- Specify appropriate test methods used to acquire data.
- Specify appropriate equipment and instrumentation used to acquire data.
- Specify data organization and presentation formats.
- Anticipate potential site-specific sampling problems.
- Distinguish between "good" and "bad" and "reasonable" and "unreasonable" data
- Take appropriate corrective actions when bad, unreasonable, or irrelevant data is identified.
- Perform quality assurance check on Level 2 data
- Take appropriate corrective action if Level 2 data fails quality assurance checks.
- Maintain continuing client contact during the project to ensure that client objectives continue to be met.
- Collect any relevant data not acquired by downstream suppliers.
- Write Project Overview
- Maintain a project log detailing activities, problems encountered, solutions found.
- Perform and record all QC activities on Level 1 data.
- Perform and record all other applicable QC procedures.
- Organize data according to requirements of next customer.
- Identify and suggest process improvements and innovations.

2.1.1.4 THE TECHNICAL WRITER

(a) Aim

To prepare and present project data that meet project objectives in an increasingly consistent and efficient manner.

(b) Input - Suppliers

Report Packet - Project Manager

Training on procedures, test methods, data handling -
Training Dept.

Ideas and suggestions - All customers and suppliers

(c) Output - Customers

Report - Project Manager

Process improvements - All customers and suppliers

(d) Responsibilities

- Understand client and project objectives.
- Understand the nature of the process and/or control device.
- Understand the test methods used to acquire data.
- Distinguish between "good" and "bad"; "reasonable" and "unreasonable"; and "relevant" and "irrelevant" data
- Take appropriate corrective actions when bad, unreasonable, or irrelevant data is identified.
- Perform quality assurance check on Level 2 data.
- Take appropriate corrective action if Level 2 data fails quality assurance checks
- Reduce data as required in job plan.
- Perform and record all QC activities on Level 1 data.
- Perform and record all other applicable QC procedures.
- Maintain a report log detailing activities, problems encountered, solutions found.
- Organize data according to requirements of next customer.
- Identify and suggest process improvements and innovations.

2.1.1.5 THE SALESPERSON

(a) Aim

To obtain and communicate accurate and relevant information regarding a client's needs and project objectives in an increasingly consistent and efficient manner.

(b) Input - Supplier

Information on client needs and objectives - Client

Training on procedures, test methods, data handling -
Training Dept.
Ideas and suggestions - All customers and suppliers

(c) Output - Customer

Information on client needs and objectives - Project Manager
Information on procedural agreements or assumptions -
Project Manager
Process improvements - All customers and suppliers

(d) Responsibilities

- Determine the client's objectives for the project.
- Determine the needs that produced these objectives.
- Understand the nature of the process and/or control device.
- Maintain continuing client contact during the project to ensure that client objectives continue to be met.
- Collect any relevant data not acquired by downstream suppliers.
- Maintain a client log detailing activities, problems encountered, solutions found.
- Perform and record all QC activities on Level 1 data.
- Perform and record all other applicable QC procedures.
- Organize data according to requirements of next customer.
- Identify and suggest process improvements and innovations.

CAE STANDARD OPERATING PROCEDURE

SAMPLING LOCATION ACCEPTABILITY AND SAMPLING POINT DETERMINATION - EPA METHOD 1

ISO NUMBER: EPA1-1

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1.0 PURPOSE

This procedure provides detailed step by step instructions for the field execution of the following EPA Method 1 processes: determining duct area, evaluating sampling location acceptability by flow disturbance criteria and cyclonic flow criteria, and sampling point selection.

2.0 SCOPE

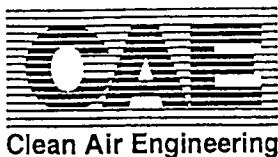
All field personnel should be familiar with this procedure to facilitate equipment setup as well as to assure location acceptability and proper sampling point determinations at each sampling location.

3.0 MATERIALS

- Tape Measure
- Calculator
- 40 CFR 60
- Sampling Probe
- Glass Tape
- Angle Finder (2)
- Angle Irons (2)
- Vise Grips
- Incline Manometer
- Cyclonic Flow Check Data Sheet (DS 002C)

4.0 DEFINITIONS

- **Tape measure** - a standard steel measuring tape of sufficient length to mark all sample points on the probe.
- **Calculator** - a typical battery or solar powered calculator to determine sampling points. No special function keys are necessary for Method 1 calculations
- **40 CFR 60-** The Code of Federal Regulations 40, Part 60, Appendix A. Specifically Method 1 and the associated Figures 1-1 and 1-2 as well as Table 1-2.
- **Sample probe-** a traverse pitot, either standard or S-type, or EPA style probe used to conduct velocity measurements singly or as part of another reference method.
- **Glass tape-** high temperature tape or equivalent to mark sample point on the sample probe



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- **Angle finder-** a contractors magnetic protractor to determine deflection angles. One of the required angle finders may be a bulls-eye level.
- **Angle iron-** a set of two (2) steel or aluminum angles used to set the probe orientation to the pitot tip for the determination of yaw angles during the cyclonic flow check.
- **Vise grips-** locking pliers, or equivalent, to secure angle irons to the sampling probe and the pitot tip.
- **Incline manometer-** a Dwyer, or equivalent, manometer to determine null (zero) ΔP readings during the cyclonic flow check.
- **Cyclonic flow check data sheet-** CAE standard data sheet (DS 002C) to record cyclonic flow check ΔP at the zero rotation angle and/or rotation angle (α) at each of the traverse points.
- **Duct-** a stack, pipe or other conduit housing the gas stream to be sampled. Referred to as stack, duct, etc.

5.0 BACKGROUND

Most sampling locations for source testing are required to meet certain minimum criteria as outlined in Method 1 of 40 CFR 60 Appendix A. These criteria must be met in order for the location to be considered an acceptable site to conduct any reference method testing that requires multi-point representative measurements. Examples of these measurements are velocity measurements made to determine either duct mass flow rates and/or isokinetic sampling rates. The Method 1 criteria include, but are not limited to, disturbances (expansion points, contraction points, or bends in the ductwork) to the gas flow and cyclonic (swirling) flow resulting in a velocity profile that is not perpendicular to the cross sectional plane of the duct or stack.

When and if the required sampling location criteria are met, Method 1 also provides the information necessary to establish a sampling grid, for both circular and rectangular ducts with the layout and number of points required, based on the location acceptability criteria. The method also provides guidelines for the sample grid layout based on the orientation and number of sampling ports available at the location. If the sample location acceptability criteria are not met, Method 1 provides alternative courses of action that are mentioned here for informational purposes only. Refer to the proper procedure for alternatives such as 3-dimensional traverses etc.



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6.0 PROCEDURE

Note: The step by step outline that follows is only for locations that CAE has not previously sampled. The majority of the time the sampling location has been tested previously and the duct area, port depth, sample points and the absence of cyclonic flow will have been provided in the job plan. It is good practice however to remeasure the duct dimensions and port depths (steps 6.1.1-6.1.7) to verify that there have been no modifications made to the location between test programs.

6.1 Traverse Point Determination and Probe Marking

- 6.1.1 Measure the port depth. Open the sample port, taking care to keep your face away from the port itself in case of positive duct pressure, and insert the measuring tape into the port hooking it on the top of the duct at the bottom of the port. Note the depth in inches and record on a piece of paper.

Hint: In ducts with high temperatures, or high negative or positive pressure, it will be next to impossible to use the measuring tape to accomplish this without ruining the tape. Instead, find a short piece of steel or other material with sufficient rigidity to hold a bend (keep the bend as flat as possible) and insert it into the port. Measure the depth to the bend after removing it from the stack.

- 6.1.2 Measure the duct or stack size in inches and record in your field notebook. For round stacks, this means measuring the inside diameter. For rectangular ducts, this means measuring both the inside width and length (depth) at the sampling location. The inside stack diameter or duct depth measurement is usually done by inserting a metal rod or sampling probe into the duct until it hits the other side. The insertion depth is noted, and measured upon removing the probe from the duct. The duct depth or stack diameter is then determined by subtracting the port depth from the total insertion depth. Due to insulation present on most ductwork, the width more often than not must be determined by plant drawings. Ask the client contact to obtain the needed dimension or better, a copy of the duct schematic.

- 6.1.3 Calculate the stack or duct area (A_s) in ft^2 using the following equations:

$$\text{For rectangular ducts: } A_s = \frac{L \times W}{144}$$

$$\text{For circular ducts or stacks: } A_s = \frac{\pi \times D_s^2}{4 \times 144}$$



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- 6.1.4 Calculate the equivalent diameter (rectangular ducts only) using:

$$D_e = \frac{2LW}{L + W}$$

Where L=length and

W=width.

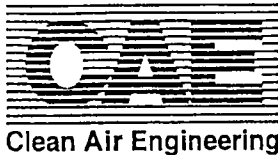
Since L and W are in inches, D_e is in inches.

- 6.1.5 Measure the distance (in inches) from the nearest disturbance upstream to the ports and record it in your field notebook. This is distance A. A disturbance is any type of deviation in the duct from a straight section of duct, such as a bend, expansion, contraction, etc.

Keep in mind that the ductwork passing through a wall or stack or any other feature external to the ductwork may not necessarily be a disturbance. A disturbance only applies to a bend, expansion, etc. that is affecting the gas flow within the ductwork, not its surroundings.

- 6.1.6 Measure the distance (in inches) from the nearest disturbance downstream to the ports and record in your field notebook. This is distance B.
- 6.1.7 Divide A and B by the duct diameter (for round ducts) or equivalent diameter (for rectangular ducts) to determine the number of duct diameters upstream and downstream, respectively, from the ports to disturbances.
- 6.1.8 Consult either Figure 1-1 (for particulate tests) or Figure 1-2 (non-particulate tests) of 40 CFR 60 Method 1 to determine the minimum number of traverse points based on the values obtained in Step 6.1.7. Always use the higher number if A and B give two different results. For example, if A = 1.0 and B = 6.5, Figure 1-1 indicates 24 points based on A and 16 points based on B. Therefore, 24 points would be required. A client may request more than the minimum number of points required by Method 1 for pollutant stratification checks, flow monitor placement or any other number of reasons. Adjust the sampling grid as necessary to accommodate the extra sampling points. Figures 1-1 and 1-2 of Method 1 are reproduced in Appendix A of this procedure.
- 6.1.9 For a circular stack or duct, consult Table 1-2 of Method 1 (contained in Appendix A) to obtain the percentage diameter multiplier for the number of points determined in Step 6.1.8. Note that Table 1-2 references the number of points on a diameter, which is half of the total number of points determined from Step 6.1.8. Multiply each %diameter value by the stack inside diameter and divide by 100 to obtain the distance from the inside wall to each traverse point*. A typical circular stack sample point grid is shown in Appendix A, Figure A-3.

For a rectangular stack, divide the total number of required points by the number of ports to obtain the number of points per port. Then divide the duct depth by the number of points per port to obtain the spacing between any two points. The distance to the first point equals 1/2 of this spacing, while each successive point



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can be found by adding the spacing to the previous point distance. A typical rectangular duct sampling grid is shown in Appendix A, Figure A-4.

*Note: Always check the first and last points for the following criteria:

i) If the diameter is less than or equal to 24", then the first and last points cannot be any closer than 1/2" to the stack walls. If the calculated point is less than 1/2" from the wall, then make it equal 1/2".

ii) If the diameter is greater than 24", then the first and last points cannot be any closer than 1" to the stack walls. The same rule applies as in the above example, adjust any points less than 1" from the duct wall to one inch.

6.1.10 Add the port depth to each point determined from Step 6.1.9 to determine the required marking distance for the probe.

6.1.11 Mark the sample probe by hooking the tape measure onto the pitot tip and marking each sample point (including port depth) with a magic marker. When all the points are placed in the probe, put a double or triple wrap of high temperature glass tape at each point. When you are finished, examine the probe for the following: For rectangular ducts, the points should be evenly spaced from one another; for circular ducts, the points should be mirror images of each from the point on the probe that corresponds to the center point of the duct. The distance between each successive point should also decrease from this "center point" outward in both directions.

Note: CAE convention is that the point farthest into or across the duct (all the way in) is always designated as point No. 1. Point No. 1 is also normally the first point sampled. However, in situations in which two trains are required to be simultaneously in a stack with only two ports, point No. 1 will be the last point sampled for one of the trains. If both samples started all the way into the duct, the probes would cross each other and it is likely that the nozzles would break at they moved across each other. In such cases it is necessary to "stagger" the starting points (one in, one out) to allow one train to work into the duct while the other comes out of the duct. Be sure to record the traverse points in descending order (e.g., 1-6, 1-5, 1-4, ... etc.) on the data sheet for the sampling train which starts at the last point and traverses into the stack.

6.1.12 If cyclonic flow checks have been conducted at the location previously, the probe is marked with the proper points for a representative traverse. If a cyclonic flow check is to be conducted, then the following Section 6.2 procedures should be followed.

6.2 Cyclonic Flow Check

Note: This procedure only applies to sampling locations in which the sample ports are oriented horizontally.

- 6.2.1 Attach an angle iron to the pitot tips with a vise grip. One inside leg of the angle iron should be placed parallel to the "high" and "low" side of the tip with the angle forming a perpendicular to the flow axis either on the "high" or "low" side of the pitot. Refer to Figure 6-1.

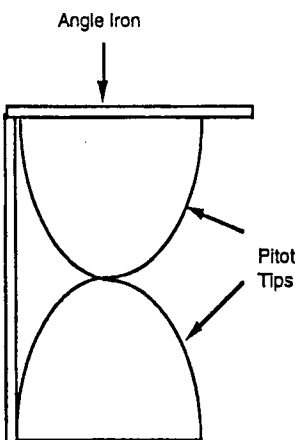


Figure 6-1: Pitot Tube With Angle Iron Assembly

- 6.2.2 With the pitot tips oriented horizontally, place an angle finder or bulls-eye level on the leg of the angle iron running parallel to the flow direction. See Figure 6-2.

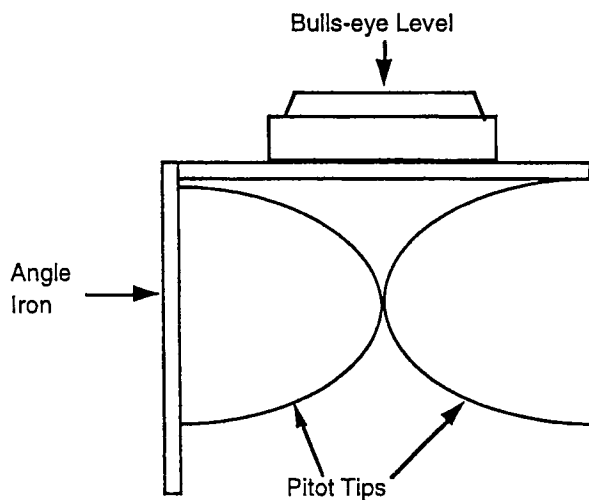
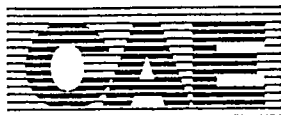


Figure 6-2: Pitot Tube/Angle Iron Assembly with Bulls-eye Level at Zero



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- 6.2.3 Rotate the probe to a zero angle (based on the angle finder) and center the bubble in the bulls-eye (bulls-eye level) and secure the probe in place. This orientation is referred to as "tip zero".
- 6.2.4 Using the second angle finder, align a second piece of angle iron as shown in Figure 6-3 to a zero degree reading that corresponds to the zero angle of the pitot tips. Be sure to double check that the probe has not rotated from tip zero. This angle finder will be used to read the actual probe rotational angle. Its reading at this point is referred as "probe zero". (Be sure to place the angle iron outside of the last marked sample point or else you'll have to start over from Step 6.2.1 when the finished assembly gets knocked off trying to reach the first point.)

Bulls-eye Level/Angle Iron assembly on Pitot Tips
(with bubble centered)

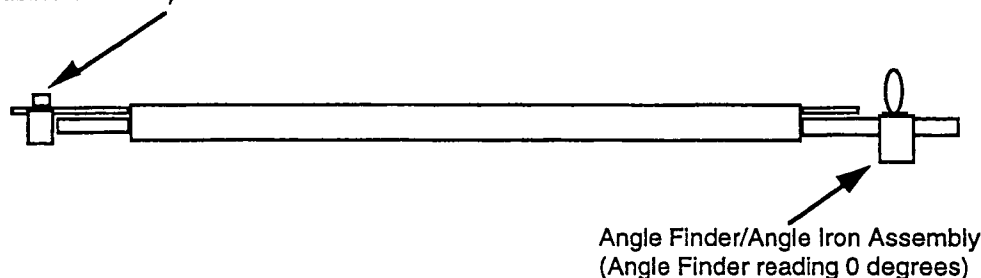


Figure 6-3: Probe Assembly Ready for Cyclonic Flow Check

- 6.2.5 When probe zero and tip zero are aligned attach the second angle iron to the probe using a vise grip. Attach the angle finder to the angle iron using tape or another means of securing it firmly so that it does not move during the cyclonic flow check.
- 6.2.6 Remove angle iron from the pitot tips.
- 6.2.7 Assemble probe, umbilical and manometer and leak check and zero the system as per Method 2 procedures.
- 6.2.8 Fill out date, client/plant, location (inlet, outlet, stack), identifier (A or B side) and operators section of standard data sheet DS 002C Verification of Absence of Cyclonic Flow.
- 6.2.9 Position the probe at point 1 (all the way in) at a zero angle on the angle finder. (Pitot tips should be positioned perpendicular to the stack flow and parallel to the cross sectional plane of the duct.
- 6.2.10 Record the velocity pressure (ΔP) at the zero angle. If the velocity pressure is negative rotate the probe 180 degrees to obtain a positive reading. The angle finder will function properly in an inverted position.



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- 6.2.11 Establish which rotation direction, clockwise (cw) or counter-clockwise (ccw), will be positive and which will be negative. It does not matter which is which so long as you are consistent from point to point.
- 6.2.12 If the velocity pressure at the zero angle is zero, record it as such and record a zero in the rotation angle column of the data sheet.
- 6.2.13 If there is velocity pressure at the zero angle rotate the probe either cw or ccw until a zero (null) reading is obtained. Record the direction (+/-) and the rotation angle (α) resulting in a null reading.
- 6.2.14 Move the probe to the next point and repeat Steps 6.2.10 through 6.2.13 for each sample point of the traverse grid. (Note that the necessity of obtaining a positive velocity pressure in Step 6.2.10 is no longer applicable).
- 6.2.15 At the conclusion of the cyclonic flow check calculate the average of the absolute values of the rotation angle (α) being sure to include any rotation angles of zero in the calculation.
- 6.2.16 Record the average absolute value (a.a.v.) of α on the appropriate line of the data sheet. If the a.a.v. is less than or equal to 20 degrees the flow conditions in the duct are acceptable and sampling may proceed. If a.a.v. is greater than 20 degrees, the flow conditions are not acceptable and alternative measurement action must be taken.



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7.0 EXAMPLES

The following examples illustrate the use of the Method 1 procedures:

Example 1

Given:

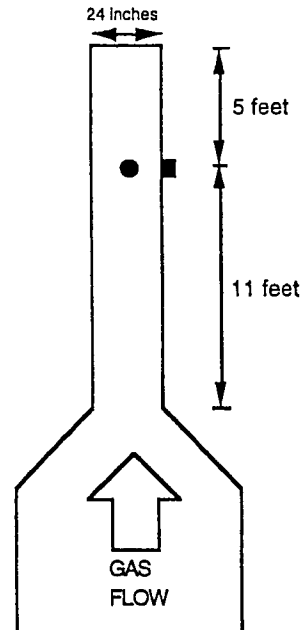
Round stack with two ports.
Inside Diameter = 24"
Upstream Distance = 5' = 60"
Downstream distance = 11' = 132"
Port Depth = 6"

Determine the number and location of sampling points for a particulate test, and calculate the distances for marking these points on the sampling probe.

Solution:

First, determine the distances A and B in terms of stack diameters:

$A = 60"/24" = 2.5$ diameters $B = 132"/24" = 5.5$ diameters



From Figure 1-1 of EPA Method 1, determine that the minimum number of points equals 12 based on A and 20 based on B. Choose the maximum number which would be 20 points.

Since there are two diameters which must be traversed, ten points per diameter (20/2) are required. This will also be the number of points used per port, since there are only two ports available.

Consult Table 1-2 using the column for ten points to determine the percentage diameter factors. Multiply each of these factors by the stack diameter divided by 100 (0.24) to obtain the point distance. Then add the six-inch port depth to obtain the probe marking distance. This information is summarized in the following table:

Point	P % Diameter	D Diameter	$X=(P)(D)/100$ Point Distance	X+6 Probe Mark
1	2.6	24"	0.62"	6.6"
2	8.2	24"	1.97"	8.0"
3	14.6	24"	3.50"	9.5"
4	22.6	24"	5.42"	11.4"
5	34.2	24"	8.21"	14.2"
6	65.8	24"	15.8"	21.8"
7	77.4	24"	18.6"	24.6"
8	85.4	24"	20.5"	26.5"
9	91.8	24"	22.0"	28.0"
10	97.4	24"	23.4"	29.4"

Example 2

Given:

Rectangular Stack with three ports.
24" Wide x 36" Deep
Upstream distance = 6 feet
Downstream distance = 18 feet
Port Depth = 4"

Determine the number and location of sampling points for a particulate test, and calculate the distances for marking these points on the sampling probe.

Solution:

First, since it is a rectangular duct, the equivalent diameter must be calculated:

$$D_e = \frac{2LW}{L + W} = \frac{(2)(24)(36)}{24 + 36} = 28.8 \text{ inches}$$

Next, determine the distances A and B in terms of the equivalent diameter (note the conversion factor of 12 inches per foot):

$$A = \frac{(6)(12)}{28.8} = 2.5 \text{ diameters}$$

$$B = \frac{(18)(12)}{28.8} = 7.5 \text{ diameters}$$

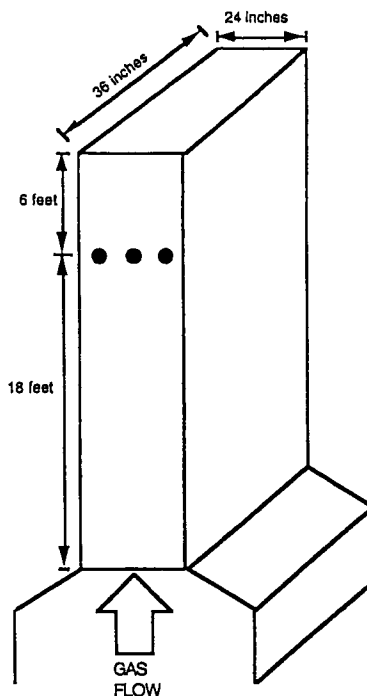
From Figure 1-1, the minimum number of points is 12 based on both A and B.

Therefore, the number of points per port equals the total number of points divided by the number of ports = $12/3 = 4$.

Divide the duct length by the number of points per port to determine the distance between any two points = $36"/4 = 9"$. The distance to the first point equals half of the point-to-point distance = $9"/2 = 4.5"$

Based on this information, the following table can be constructed:

	X	X+4
Point	Point Distance	Probe Mark
1	4.5"	8.5"
2	13.5"	17.5"
3	22.5"	26.5"
4	31.5"	35.5"





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Example 3

Given:

Round stack with 4 ports
Inside diameter = 10' (120 inches)
Upstream distance = 50'
Downstream distance = 80'
Port depth = 6"

Determine the number and location of sampling points for a particulate test, and calculate the distances for marking these points on the sampling probe.

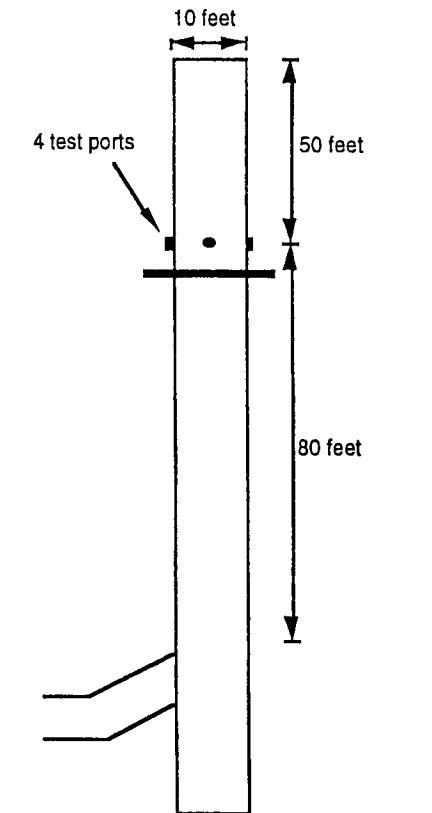
Solution:

First, determine the distances A and B in terms of stack diameters:

$$A = 50' / 10' = 5 \text{ diameters}$$

$$B = 80' / 10' = 8 \text{ diameters}$$

From Figure 1-1, the minimum number of points is 12 based on both A and B. Thus, six points are required per diameter. Since there are 4 ports, only the first three of the six points will be needed for probe marking. Therefore, when using Table 1-2, use only the first three points listed for the six-point column. Thus:



	P	D	$X = (P)(D) / 100$	X + 6
Point	% Diameter	Diameter	Point Distance	Probe Mark
1	4.4	120"	5.28"	11.3"
2	14.6	120"	17.52"	23.5"
3	29.6	120"	35.52"	41.5"



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Example 4

Cyclonic Flow Check Calculations

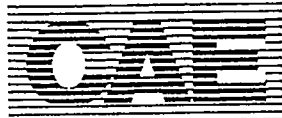
Given:

Cyclonic Flow Check Data For a Circular stack requiring 16 traverse points

Port/Point	Vel. press. at 0°	Rotation angle giving 0 v.p.
1 - 1	0.06	3
1 - 2	0.08	5
1 - 3	0.11	8
1 - 4	0.06	6
1 - 5	0	0
1 - 6	-0.02	- 3
1 - 7	-0.04	- 4
1 - 8	-0.05	- 10
2 - 1	0.03	3
2 - 2	0.03	3
2 - 3	0.05	5
2 - 4	0.02	1
2 - 5	-0.03	- 3
2 - 6	0	0
2 - 7	-0.05	- 4
2 - 8	-0.02	- 2
Absolute sum α		60
Absolute average α		3.75

Notes:

- 1.) The absolute sum is 60 and not 8 as it would be if you were not calculating absolute values. i.e. a positive four and a negative four result in an absolute value of eight, not zero.
- 2.) The average α calculation includes the zero points.
- 3.) The location is acceptable under Method 1 criteria (i.e. a.a.v \leq 20).



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APPENDIX A

REPRODUCTION OF EPA METHOD 1 FIGURES



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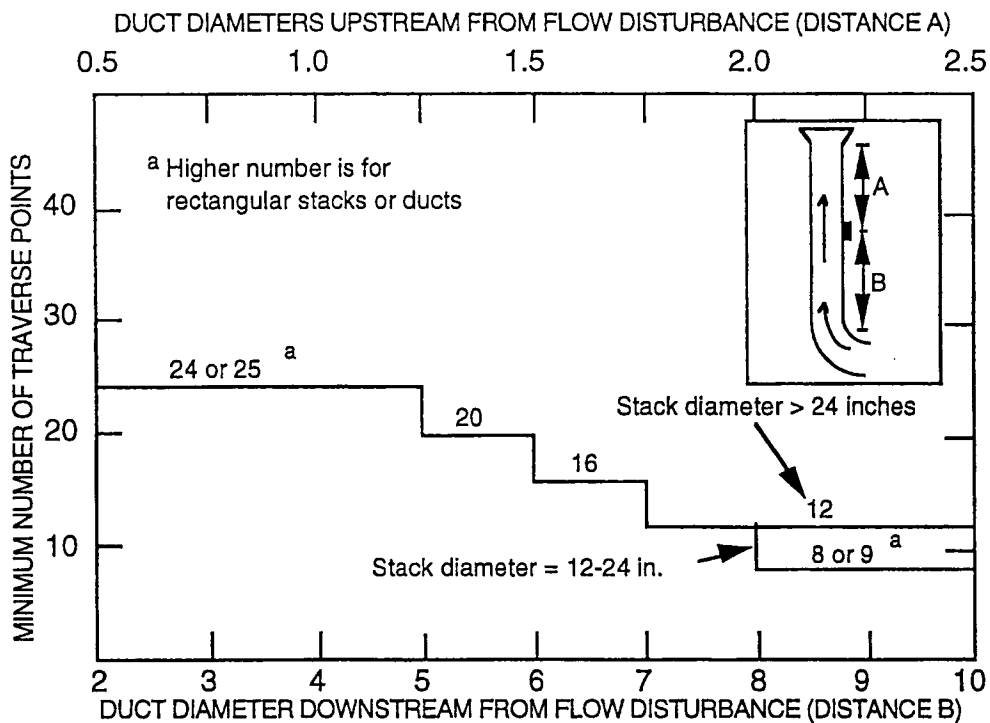


Figure A-1: Figure 1-1 of EPA Method 1 - Minimum Number of Traverse Points for Particulate Traverses



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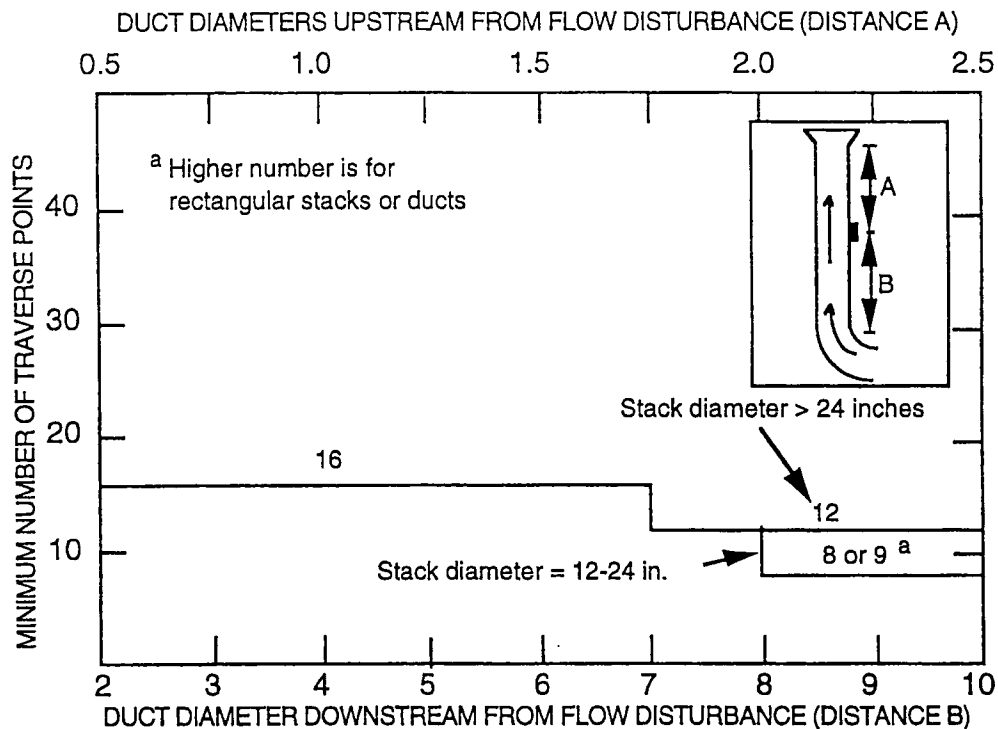


Figure A-2: Figure 1-2 of EPA Method 1 - Minimum Number of Traverse Points for Velocity (Non-Particulate) Traverses



Clean Air Engineering

Effective Date: x/x/95

Standard Operating Procedures

Field Sampling Procedures

Drafted By: S. Williams

EPA Method 1 Procedures

General Source Testing

SOP I.D.: EPA1-1

CTPM Section F Page 16

Approved By: TRB

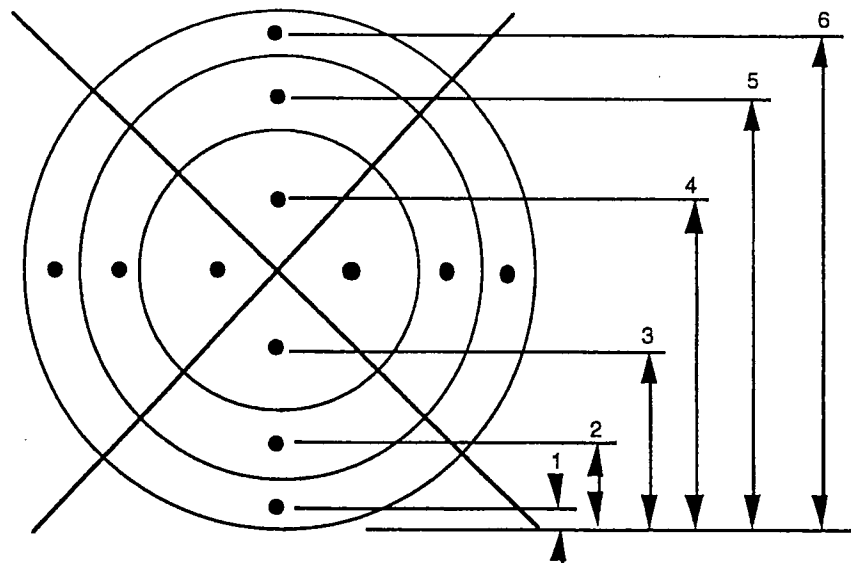


Figure A-3: Typical Circular Duct Sampling Grid

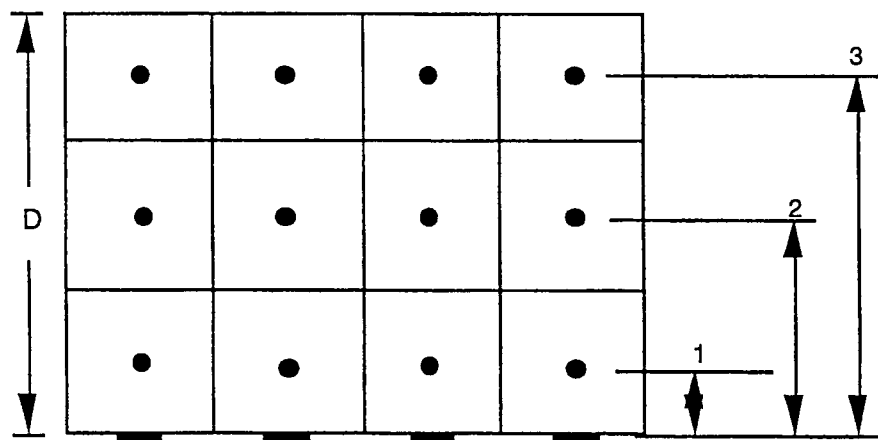


Figure A-4: Typical Rectangular Duct Sampling Grid

NUMBERING SAMPLING PORTS

1.0 PURPOSE

This procedure defines a numbering system to be used to identify sampling test ports.

2.0 SCOPE

This procedure should be used by anyone involved in collecting and presenting source sampling data. The port numbering system should be used to reference sampling ports on all data sheets, Job Plans, drawings, and reports associated with source testing. An exception to the system described herein would be locations which have a specific identification system designated by the client.

3.0 MATERIALS

The following materials are needed to properly execute this procedure:

- standard data sheet for applicable source testing methodology
- field log book
- black ink pen
- directional compass (to measure due North when appropriate)

4.0 DEFINITIONS

Flue - a term used to represent either a stack or duct which acts as a conduit for gas flow. A flue can be either circular or rectangular, and can be oriented in any direction.

Sampling Port - a passageway into a flue which can be used to sample or measure a property of the gas inside.

Cross-Section - a two-dimensional view of the flue at the location of the sampling ports oriented from inside the flue looking upstream into the flow. When drawn on planar paper, the cross-section will appear as an outline of the flue, having the same geometric shape as the flue, and with the same orientation of the flue as if the gas were flowing out of the page.

5.0 PROCEDURE

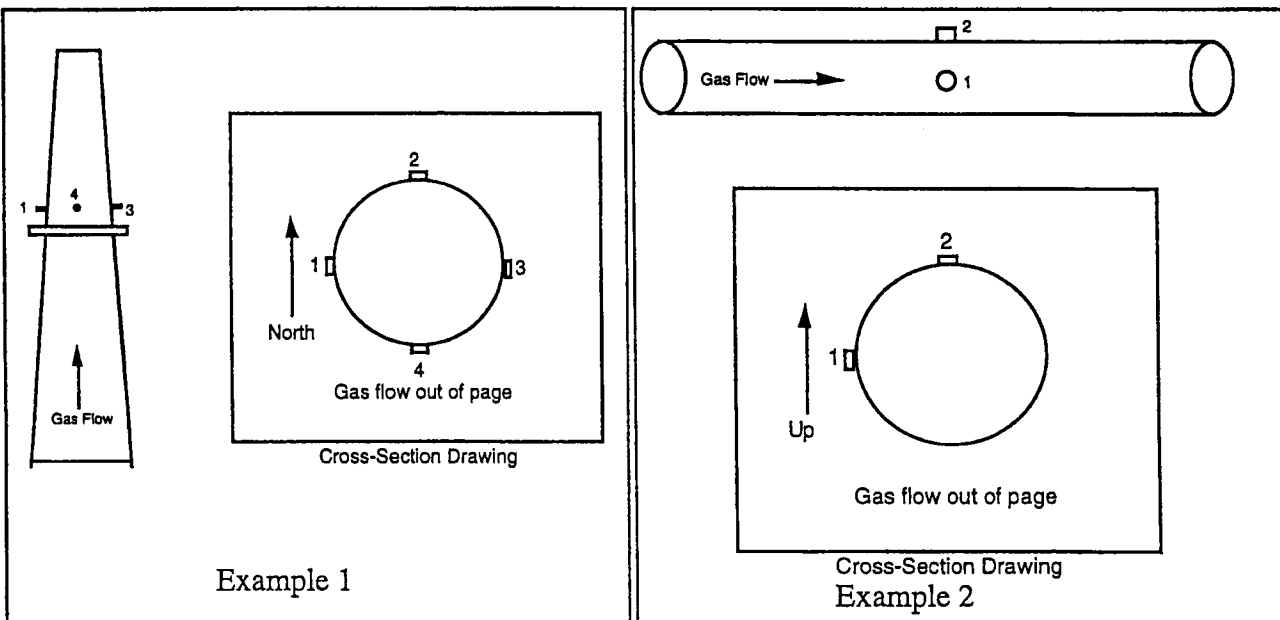
5.1 Identify the location of the sampling ports for the source. Only one cross-sectional plane of sampling ports should be included on a single drawing. If the testing involves multiple test planes, then a separate sketch should be prepared for each plane. In these cases, the planes should be identified appropriately to distinguish them apart (e.g., lower or upper plane.)

5.2 Make a sketch of the cross-sectional outline of the flue in a field log book or on the standard data sheet to be used for the testing. Mark on the drawing that the gas flow is "Out of the page". Also be sure to identify the source name which the drawing represents.



- 5.3 For vertically oriented flues, identify the direction for due North on the drawing with an arrow designation. For all other orientations (e.g., horizontal), identify the direction of Up on the drawing with an arrow designation. In both cases, make the North or Up designation from the perspective of looking into the gas flow of the cross-section.
- 5.4 Sketch all available sampling ports onto the cross-section diagram. Place the orientation of the ports from the perspective of looking into the gas flow, with the flow coming out of the page. Show all ports in the cross-sectional plane, even if one or more of the ports will not be used for sampling. However, do not show ports which are located in a different plane.
- 5.5 For circular flues, assign one of the ports the I.D. of being "1". Then, number all remaining ports on the cross-section diagram sequentially (e.g., 2, 3, 4) in clockwise order. Do not add any additional identifiers to the I.D. used on the diagram other than the port number itself (i.e., do not use such designations as P1, Pt-1, etc.)
- 5.6 For rectangular flues, port 1 is defined as either the left-most or top-most port *on the cross-section diagram*. Number all remaining ports sequentially in either a left-to-right or a top-to-bottom order. Do not add any additional identifiers to the I.D. used on the diagram other than the port number itself.
- 5.7 Any sampling points which utilize the ports should be referenced by the port number followed by a dash and the point number. For example, the first point of the third port would be designated as point "3-1".

The following sketches illustrate several examples of proper port designations:





Standard Operating Procedures

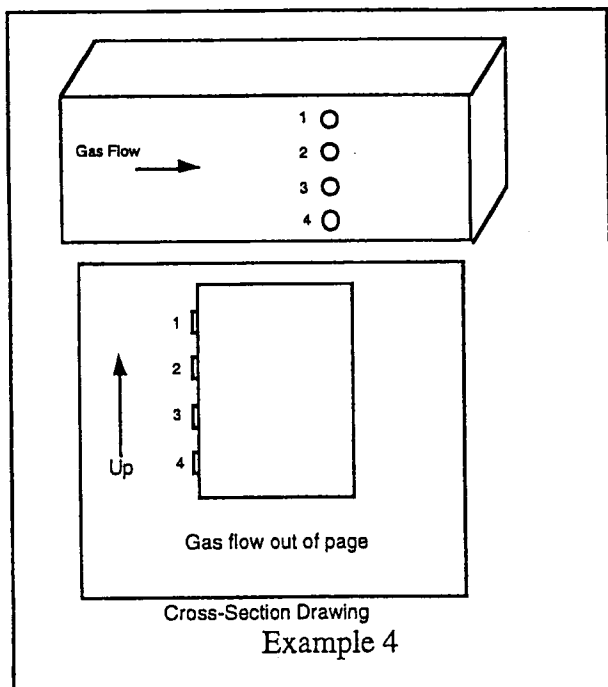
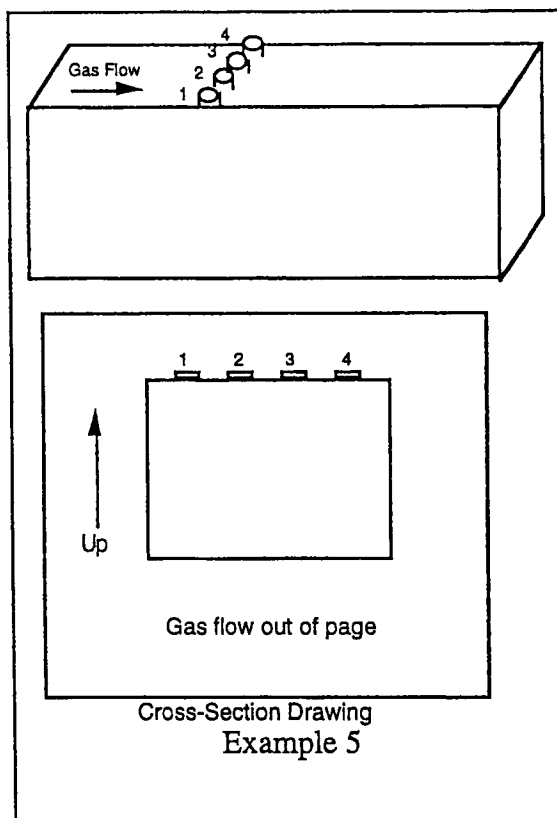
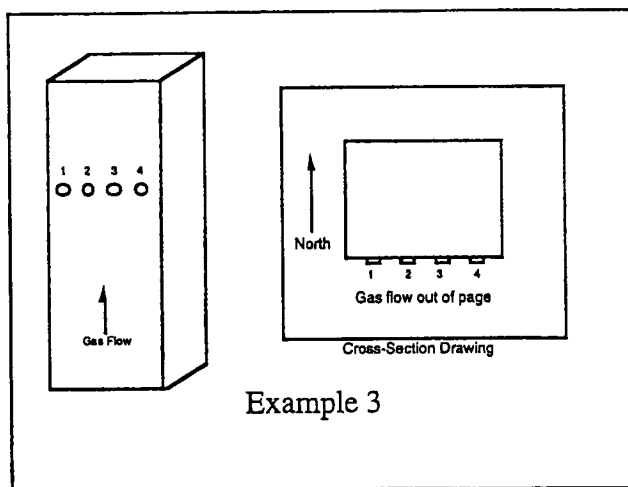
Clean Air Engineering Field Sampling Procedures

Effective Date: 10/15/95

Drafted By: JE Wright

Port Numbering
Field Project Management
SOP I.D.: EPA1-2
CTPM Section F Page 3

Approved By: Process Owners



CAE STANDARD OPERATING PROCEDURE

PITOT TUBE LEAK-CHECK

ISO NUMBER: EPA2-1

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

List of Materials: Sample Probe (or Pitot Tube), Umbilical, Meter Box Assembly, Tubing (Optional), Field Test Data Sheet

Definitions

Sample Probe - Device inserted into stack or duct in order to withdraw gas sample and monitor flue gas temperature and velocity within the stack. The probe consists of a glass or stainless steel lined tube with a heating element. The probe also contains a Stausscheibe or S-Type pitot tube for measuring flue gas velocity, a thermocouple for measuring flue gas temperature, and a thermocouple for measuring probe heater temperature.

Positive Pressure Side of Pitot - The side of pitot tube facing into the gas flow.

Negative Pressure Side of Pitot - The side of pitot tube facing out of the gas flow.

Umbilical - Flexible conduit between meter box assembly and probe/filter box/impinger bucket. Umbilical contains pitot lines, thermocouple lines, a gum rubber sample line, and amphenol line. The amphenol line is used to supply power from the meter box assembly to the sample train.

Meter Box Assembly - Consists of control console and pump box, see meter box assembly procedure. The meter box assembly controls and monitors operation of the sampling train.

Tubing - Clear, flexible, 1/4" tubing, usually used for pitot lines. When leak checking pitots a piece of Tygon approximately 2 feet long is often slid over one side of the pitot tip in order to make it easier to blow into the pitot. A pinch clamp is sometimes used with the Tygon to make it easier to seal the line after it is pressurized.

Field Test Data Sheet - Data sheet on which test parameters are recorded during a sample run.

Procedure Steps (See Figures 1 and 2):

1. Assemble the sample train per Method 5 sample train procedure.
2. Slide a piece of Tygon tubing over the positive pressure side of the pitot tip (Optional, see definitions)

Procedure Steps (Continued)

3. Gently blow into the positive pressure side of the pitot tip while at the same time watching the inclined manometer on the meter box. Blow into the pitot until the manometer is reading at least 3" of water (generally it would be better to be reading at least 6" of water.)
4. Quickly close off the pitot tip with your finger. If you are using a piece of Tygon with a pinch clamp close off the pinch clamp.
- 5.
6. Make sure the manometer is still reading over 3" of water. If not, repeat steps 3 and 4 again.
7. Keep the end of the pitot tip closed off until the oil in the manometer has stopped moving and remains unchanged for at least 15 seconds.
8. Make sure the manometer reading is still 3" of water or more. If not, check all connections in the pitot line and look for damage to the umbilical, then repeat steps 3 through 6 again.
9. With the positive pressure side of the pitot tip still closed off, place your finger over the negative pressure side of the pitot tip.
10. Release the positive pressure side of the pitot tip.
11. If the manometer reading is still greater than 3" of water keep the end of the pitot closed off until the oil has stopped moving and remains unchanged for 15 seconds.
12. If steps 6 and 10 were successful, check off the pitot leak check box on the field test data sheet; you have completed the leak check of the pitot. If step 10 did not yield a steady manometer reading of over 3" of water go to step 12.
13. If step 10 was not successful, slip a piece of Tygon tubing over the negative pressure side of the pitot tip (optional).
14. Gently draw through the negative pressure side of the pitot tip while at the same time watching the inclined manometer on the meter box. Draw through the pitot until the manometer is reading at least 3" of water (generally it would be better to be reading at least 6" of water.)
15. Quickly close off the pitot tip with your finger. If you are using a piece of with a pinch clamp close off the pinch clamp.
16. Make sure the manometer is still reading over 3" of water. If not, check all connections in the pitot line and look for damage to the umbilical, then repeat steps 13 and 14 again.

Procedure Steps (Continued)

17. Keep the end of the pitot tip closed off until the oil in the manometer has stopped moving and remains unchanged for at least 15 seconds.
18. Make sure the manometer reading is still 3" of water or more. If not repeat steps 13 through 16 again.
19. Check off the pitot leak check box on the field test data sheet; you have completed the leak check of the pitot.

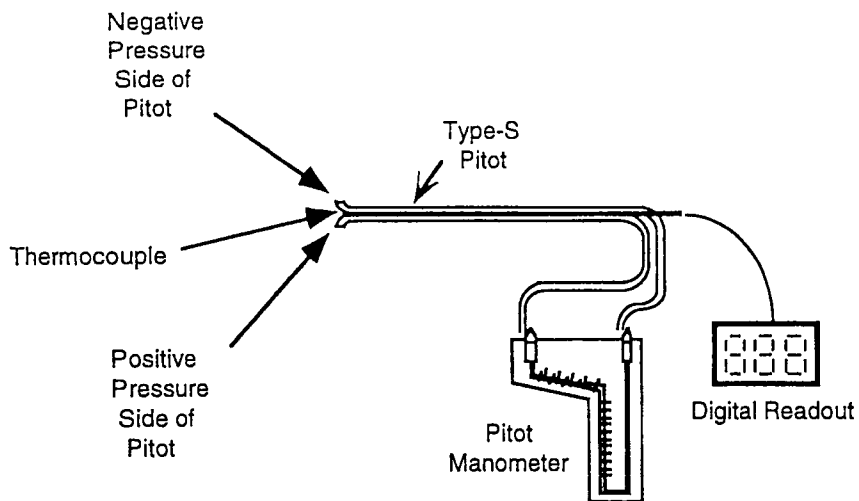


Figure 1: Type S Pitot Assembly

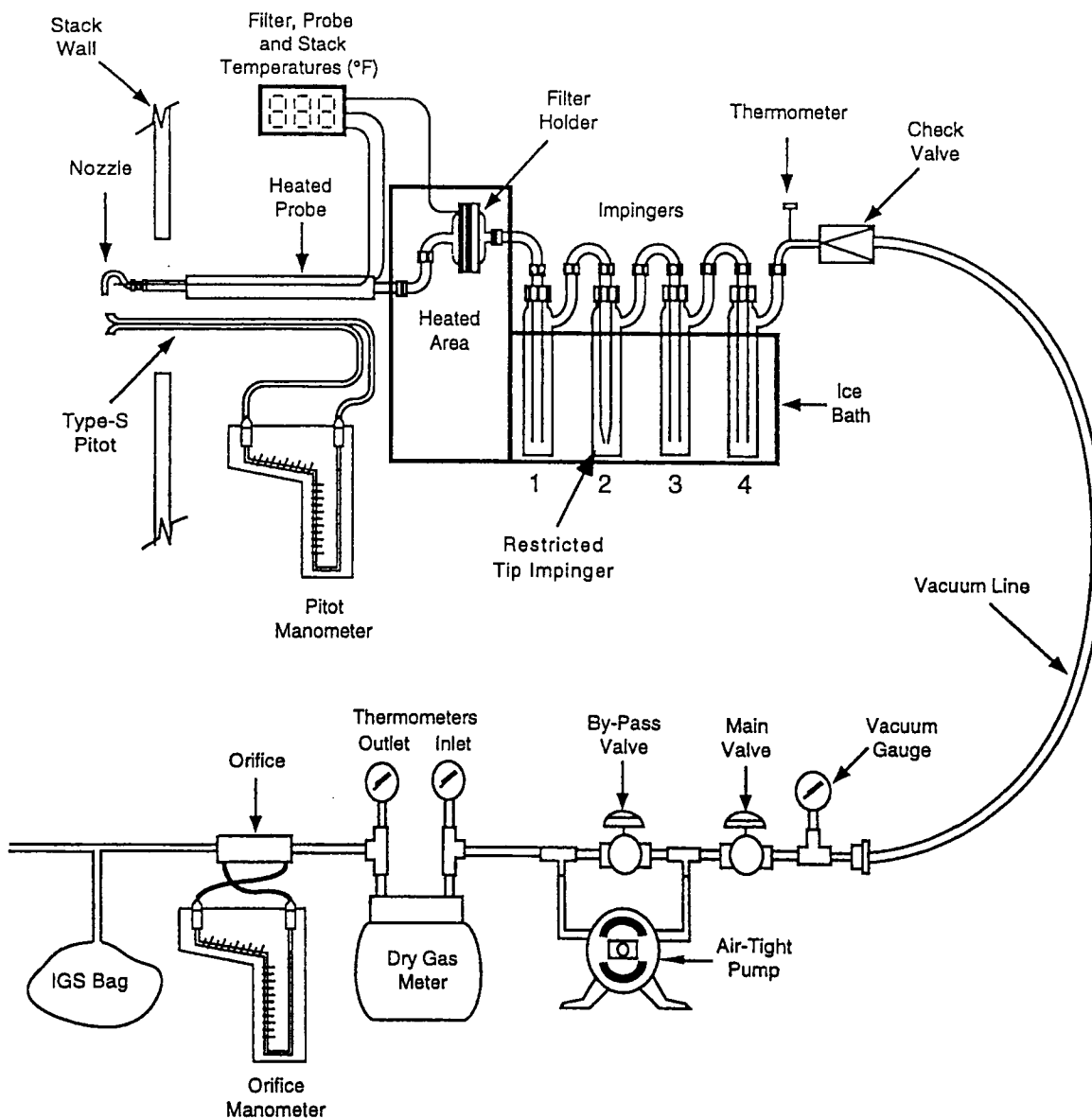


Figure 2: Standard M5 Sample Train

CAE STANDARD OPERATING PROCEDURE

METHOD 2 VELOCITY TRAVERSE (QUICK START)

ISO NUMBER: EPA2-2

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

List of Materials : Traverse probe of required length, umbilical line containing pitot lines and thermocouples, short piece of tygon tubing (3-4 inches), meter box console with inclined manometer or separate inclined manometer, gauge oil for manometer, pitot quick connects (connects to meter console, if used), pyrometer or temperature readout on meter console.

Definitions :

Procedure Steps :

1. After obtaining a static pressure from the duct, turn the traverse pitot 90- degrees from the direction of flow and quickly insert through the port and into the duct
2. Seal the port around the traverse probe with a towel or suitable sealing medium
3. Place the traverse probe at the first traverse point. The first traverse point is located closest to the opposite side of the duct or stack. The traverse probe should be oriented perpendicular to the direction of the flow. The orientation of the pitots inside the stack can be determined by observing the back end of the traverse probe. Allow the probe temperature to equilibrate with stack conditions, usually 3-5 minutes.
3. After temperature equilibration turn the traverse pitots into the direction of the flow, making sure that the impact side of the pitots are facing the direction of flow.
3. Record the traverse point number under the Traverse Point Number heading on the data sheet. These numbers should be recorded by labeling the port designation followed by the traverse point number (e.g. 1-1, 1-2 and 1-3 for points 1, 2 and 3 in port 1).
4. Allow the Delta P reading on the inclined manometer to stabilize. Once stabilized record the reading according to the following criteria
 - 0.005" if $\Delta P \leq 0.1$ "
 - 0.01" if $0.1 < \Delta P \leq 1.0$ "
 - 0.05" if $\Delta P > 1.0$ "
5. Record each traverse point temperature reading to the nearest whole degree.
6. If any deviations to EPA method 2 criteria or process upsets occur during the traverse make notations of these in the column marked notes.

7. Repeat this procedure for every port until all the points are traversed.
8. After the final point is traversed remove the probe from the duct. If the static pressure is extremely negative be sure to pull the probe through the port as quickly as possible.
9. Remove the probe from the duct and allow to cool. After the probe has cooled to the touch perform a post test leak check on the impact and static sides of the pitot tubes.(See Preliminary Traverse Procedure for a detailed description of performing a pitot leak test.).

METHOD 2 VELOCITY TRAVERSE (DETAILED PROCEDURE)

ISO NUMBER: EPA2-3

EFFECTIVE DATE: Pending

AUTHOR: Dept. 68

APPROVED BY: Pending

I Introduction

These instructions detail the procedures required to perform velocity traversing to US EPA acceptable standards. Velocity measurements taken in this manner will provide reliable results for a wide variety of applications, however it should be noted that the EPA's primary interest is in stack emissions. These procedures must therefore be evaluated for suitability prior to use for other applications.

II. METHOD SUMMARY

To determine the volumetric flow rate in a duct by the EPA method, the duct cross section is first divided into sub-sections of equal area, then the velocity pressure and temperature at the centroid of each sub-section are measured. The average volumetric flow rate in the duct is then determined using a calculation based on Bernoulli's Equation which combines the measured traverse parameters with the gas molecular weight and pitot calibration coefficient.

US EPA test methodology can be found in 40 CFR 60 Appendix A, Methods 1 and 2. These are reproduced in appendix 1.

III. TRAVERSE SYSTEM DESCRIPTION

The velocity measurement system consists of a calibrated pitot tube to sense gas velocity head, and a thermocouple assembly to sense gas temperature. These are attached by means of an umbilical to a manometer to indicate velocity head, and pyrometer to indicate temperature. Pitot tubes can be of either the 'S' type design (also called the stausscheibe), or the standard pitot design. 'S' type pitots are more compact and less prone to plugging with particulates, but require calibration against a pitot of the standard design. Thermocouples are typically type 'K' or 'J' and are fastened to the pitot tube in a manner which will avoid affecting the pitot reading.

III. TRAVERSE SYSTEM DESCRIPTION (Continued)

Figure 1

Refer to figure 1.

ITEM NO.	NAME	PURPOSE
1a.	TYPE 'S' PITOT	VELOCITY PRESSURE SENSOR
1b.	STANDARD PITOT	ALTERNATE VELOCITY PRESSURE SENSOR
2.	THERMOCOUPLE	TEMPERATURE SENSOR
3.	POINT MARK	INDICATES PROBE POSITION IN DUCT
4.	MANOMETER	INDICATES VELOCITY PRESSURE

5.	PYROMETER	INDICATES GAS TEMPERATURE
6.	UMBILICAL	CONNECTS PITOT TO MANOMETER AND THERMOCOUPLE TO PYROMETER
7.	PORT ADAPTER	ATTACHES PITOT TO DUCT, PREVENTS GAS LEAKAGE DURING VELOCITY MEASUREMENTS
8.	PITOT STAND	SUPPORTS PITOT DURING MEASUREMENTS

IV. PERFORMING A VELOCITY TRAVERSE

Traverse Location Selection/Determination of Traverse Points

Pitot gas velocity measurements are most accurate where the gas flow distribution is without distortion across the duct. This condition is most likely to occur in long runs of straight duct work. EPA Method 1 defines the ideal sample port location as being at least eight duct diameters downstream and two diameters upstream of any flow disturbance (see Figure 2).

Figure 2

Where sufficient duct length exists, a minimal number of traverse measurement points will provide reliable data, as the available duct length decreases from the ideal, the number of traverse points must be increased to obtain reliable velocity head data. Two duct diameters downstream and one half diameter upstream of any flow disturbance is the minimal acceptable location for velocity measurement.

Where only a short length of ductwork is available for obtaining velocity measurements, or where turbulent flow exists (such as exits a cyclone), EPA pitot tube methodology may be unreliable. Refer to section V for details on the limits of pitot tube velocity measurement.

IV. PERFORMING A VELOCITY TRAVERSE (Continued)

Traverse Location Selection/Determination of Traverse Points (Continued)

The following procedure is used to locate the traverse points, assuming that a suitable location for velocity determination exists:

Circular Ducts (Refer to Example 1)

1. Measure the duct cross section and the length from the sample location to upstream and downstream flow disturbances.
2. Determine the number of duct diameters from the sample location to upstream and downstream flow disturbances.
3. Determine the number of traverse points per test port using Method 1, Figure 1-2.
4. Determine the location of each traverse point within the duct using Method 1, Table 1-2.

Rectangular Ducts (Refer to Example 2)

1. Measure the duct cross section and the length from the sample location to upstream and downstream flow disturbances.
2. Determine the number of duct diameters from the sample location to upstream and downstream flow disturbances using the "equivalent diameter" as determined by Method 1, Equation 1-1.
3. Determine the number of traverse points per test port using Method 1, Figure 1-2 and Table 1-1
4. Determine the location of each traverse point within the duct as shown in the example.

The number of traverse points can be increased over the required minimum. All points must be located by dividing the duct into the desired number of equal areas using the procedures outlined above, then placing the traverse point at the centroid of each equal area. Do not locate sample points randomly within the duct.

IV. PERFORMING A VELOCITY TRAVERSE (Continued)

System Assembly

Before using any components, inspect each for visible signs of wear or abuse, and locate required calibration records. Do not use damaged components or equipment with no proof of calibration.

Connect the 'impact' side of the type 'S' pitot to the '+' side of the manometer, the 'suction' side of the pitot to the '-' side of the manometer, and the temperature sensor to the pyrometer using the umbilical. Locate the manometer on its base in a stable location. Insure that the manometer is level.

Leak check the system by blowing into the impact side of the pitot until at least 3" H₂O registers on the manometer. Seal the tube opening. The manometer reading must remain stable for at least 15 seconds. Repeat the procedure on the suction side of the tube by applying a vacuum to the opening. Verify operation of the temperature sensor by applying heat. Any problems identified must be fixed before traversing begins.

Mark the traverse points on the probe taking into account the sample port length and the port adapter as shown in Examples 1 and 2. High temperature situations will require suitable high temperature tape or paint markings.

Taking care not to touch the pitot tip to any hard surfaces, insert the pitot into the port adapter, assemble the adapter to the port then position the probe at the first traverse point mark. Determine that the pitot is oriented properly with the gas flow and verify that the manometer is indicating gas flow.

Data Recording

Record the traverse location designation, the pitot and pyrometer serial numbers, the date and the traverse start time on a data sheet.

Hold the pitot at the first traverse point for several minutes to allow the thermocouple to adjust to the gas temperature, insure that the pitot is still properly aligned, then record the gas temperature and velocity pressure. Move the pitot to the next point and repeat the procedure.

During the test period the duct static pressure and the ambient barometric pressure must be recorded. Obtain process operations data to help interpret the traverse results.

At the conclusion of the traverse record the stop time and note the operators name(s) on the data sheet.

IV. PERFORMING A VELOCITY TRAVERSE (Continued)

Calculation of Results (Refer to Example 3)

Average the traverse data as shown in the example. Note that the square root of each velocity pressure reading is first determined, then the average square root is calculated.

The amounts of each of the major gas constituents must be known in order to determine the gas molecular weight. US EPA Methods 3 and 4 (refer to Appendix A) are for determining gas composition. When velocity traversing is not associated with compliance testing, other means of determining the gas composition are acceptable. Process data, material balance or other means may be employed.

The gas molecular weight is determined from the gas composition using the example equation 1. Equations 2 through 7 determine the gas velocity and volumetric flow rate.

In most cases the primary gas constituents are nitrogen, oxygen, carbon dioxide, and water vapor.

Reporting Results

The minimum report should include:

- Facility name and traverse location designation.
- Traverse start time, stop time and date.
- Flue gas temperature, velocity, and volumetric flow rate.
- Flue gas composition.
 - An explanation of how the gas composition was determined.
 - Documentation of plant operating conditions during the traverse.
 - Copies of all traverse data collected.
 - Copies of equipment inspection and calibration forms.

The following additions to the minimum report are recommended:

- A diagram showing the location of traverse points in the duct.
- A diagram showing the location of flow disturbances in the duct.
- A description and schematic of the system traversed.
- An example calculation.
- A discussion of the traverse data.

V. LIMITS FOR APPLICATION OF EPA VELOCITY TRAVERSING METHODOLOGY

The techniques detailed in these instructions will provide unreliable results when applied to the following situations without modification.

SITUATION	SUGGESTED MODIFICATIONS
Gas velocity <15ft/sec	<ol style="list-style-type: none"> 1) Substitute a micromanometer for the standard manometer. 2) Perform a low velocity wind tunnel pitot calibration. 3) Evaluate non-EPA methodology such as vane and hotwire anemometers or velometers for applicability.
Gas velocity >200ft/sec	<ol style="list-style-type: none"> 1) Substitute an extended range manometer for the standard manometer. 2) Perform a high velocity wind tunnel pitot calibration. 3) Account for gas compressibility when calculating the gas velocity. 4) Evaluate non-EPA methodology such as the use of a flow orifice for applicability.
Wet or dirty gasses	<ol style="list-style-type: none"> 1) Use a pitot designed for use in dirty situations. 2) Incorporate provisions for compressed air blowback of the pitot. 3) Take data quickly to minimize blockage.
Cyclonic gas flow	<ol style="list-style-type: none"> 1) Use the "Alignment Approach" to take data. 2) Incorporate provisions for an angle finder on the pitot. 3) Account for the angular flow component when calculating velocity.
Turbulent gas flow	Use a directional pitot tip such as that in Appendix 2.
Variable gas flow	Use an electronic manometer interfaced with a data acquisition system.

VI. EQUIPMENT MAINTENANCE & CALIBRATION

Only calibrated equipment in good working order can provide consistently reliable information. Do not use equipment which appears to be damaged, or for which current calibration data cannot be found.

Pitot Tube

Details for geometric pitot and wind tunnel calibrations are in EPA Method 1 in Appendix A. Pitots should be inspected and dimensions checked prior to each use, record the inspection data on a suitable form.

Type 'S' pitot maintenance consists of repairing any dents, tube misalignment, broken welds or corrosion as required to maintain the proper dimensions, and cleaning. 'S' pitots which cannot be repaired to meet Method 1 dimensional specifications should be discarded.

Damaged standard pitot tubes should be discarded rather than repaired.

Manometer

Inspect the gauge for damage and for discolored or dirty fluid before each use. Replace fluid as required with fluid of the proper specific gravity as specified by the manufacturer. Disassemble to clean annually and replace o-rings and other components as required.

No additional calibration of the manometer is required.

Thermocouple/Pyrometer

A three point calibration check should be performed on the thermocouple/ pyrometer system before each use. Check the system against an ASTM reference thermometer in an ice bath, a boiling water bath, and an oil bath at a temperature above the highest expected gas temperature. Allow at least 3 minutes for temperature equilibrium when performing this calibration. The thermometer and the thermocouple should agree to within 1.5% when compared on an absolute basis. Record the calibration data on a suitable form.

EPA METHOD 3 ORSAT

ISO NUMBER: EPA3-1

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

List of Materials: Burrell Orsat, CAE Standard Data Sheet DS 012 ORSAT, Oxsorbent, Disorbent, IGS Bag, Buret Solution

Purpose: Orsat gas analyzers are used to determine carbon dioxide (CO₂) and oxygen (O₂) concentrations from gas streams.

CO₂ and O₂ concentrations are used to determine molecular weight, which is required to calculate volumetric flow rate.

Emission limits in air permits sometimes specify excess air (dilution) must be taken into account. This is done by adjusting emissions to dry standard conditions, then correcting the required CO₂ or O₂ concentration.

Description: A gas sample is extracted from a stack by single point grab sampling, single point integrated sampling, or multi-point integrated sampling. A measured volume of the gas sample is bubbled through successive chemical solutions which absorb CO₂ and O₂, resulting in a decrease in gas volume. The decrease can be measured as percent of CO₂ and O₂.

Definitions:

Oxsorbent	Dilute chromium chloride-hydrochloric acid, dark color.
Disorbent	Dilute potassium hydroxide solution in water, clear solution.
Buret Solution	Saturated solution of sodium acid sulfate in water.
MSDS	Material Safety Data Sheets - Identify chemical names and related hazards, Required for transportation of chemicals
Buret	Contain Buret Solution, marked for displacement measurements
Pipette	Contain Absorbing Solutions
Manifold	Contains Tube Connected to all Valves.
Capillary	Small chamber at the top of buret and pipettes
Collection Bag	Leak-free, flexible, plastic coated bag to contain the gas sample collected from the stack (IGS Bag)

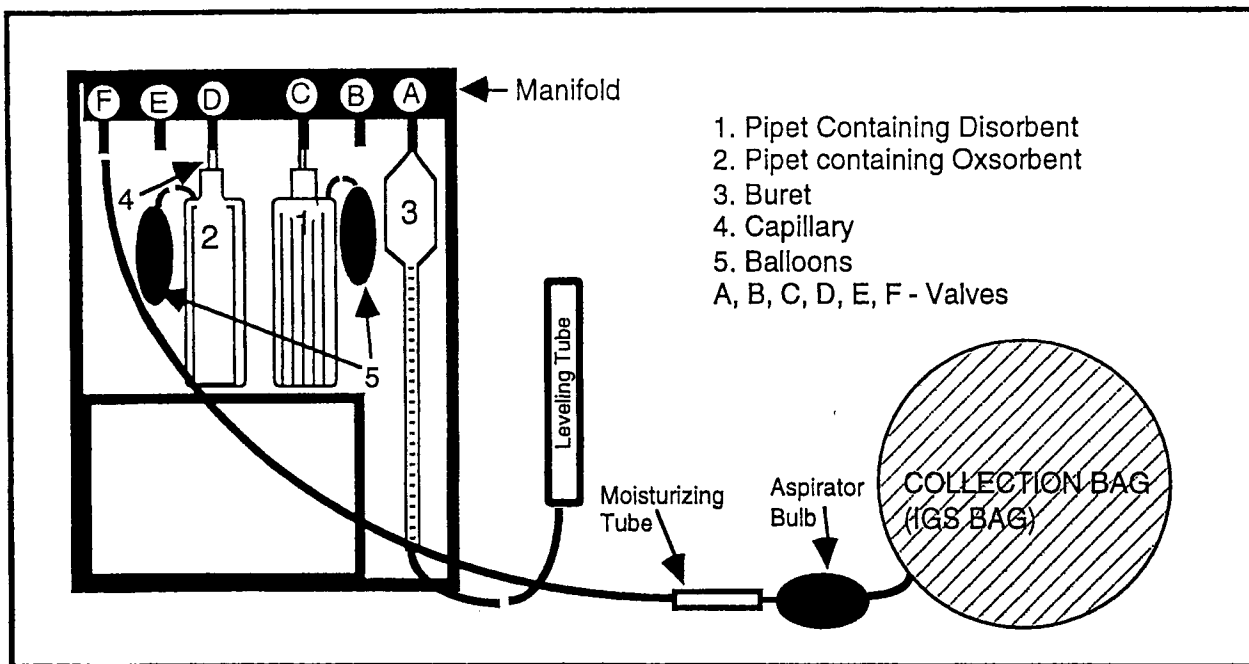


Figure 1

Procedural Steps for Preparing Orsat for Use:

Inspection

1. Check hoses, IGS Bag, and balloons for and connections between capillary's and manifold for cuts, abrasions or cracks
2. Replace damaged glassware

Cleaning

1. Disconnect Glassware (if required)
2. Wash all parts with soap and water, including the inside of the pipettes
3. Flush each valve and manifold with hot water, dry with pressurized air
4. If valves or manifold are plugged, first try to clear both with a thin wire. There is a screw in the manifold that can be removed. The second option is to take the manifold apart and clean with soap and water. *Do not remove valves unless all other options fail, they are hard to thread and set correctly and can easily damage the orsat.*

Assembly

1. Position the orsat so the valves face forward, close valves firmly - *don't over tighten.*
2. Attach graduated burette to valve A
3. Attach leveling tube (leveler) to graduated burette with a rubber hose
4. Attach disorbent pipette (contains tubes for increased surface area) to valve C
5. Attach oxorbent pipette (contains ball to seal glass tube) to valve D

The pipette opening for gas balloons should face the rear of the manifold

6. Fill leveler 2/3 with Buret Solution, hang on side frame
7. Remove material from moisturizing tube, wet, and return to holder
8. Fill pipette 2 with oxorbent (dark solution) and pipette 1 with disorbent (clear).
9. Fill balloons slightly with ambient air by blowing into opening, pinch rubber and quickly insert stoppers into pipettes. *Blow into opening, but do not touch surface to lips*

Note: The purpose of valve F on the Orsat is CO analysis, which, along with the optional wash bottle and associated metal bracket, is not typically utilized by CAE

Priming the Solutions

1. Open Valves A and B
2. Raise leveler until buret solution rises into capillary (1/8 inch from rubber tubing) see Figure 2.
3. Close Valve B
4. Open Valve C

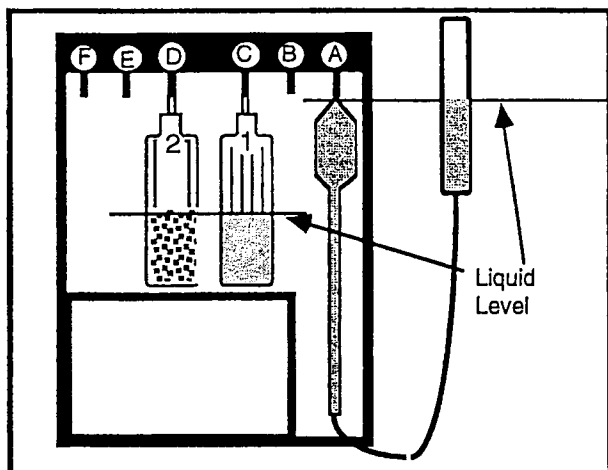


Figure 2

If liquid is allowed above the capillaries the orsat manifold will become restricted or plugged and will need to be cleaned as described above. By placing one finger on the rubber below the leveler, it is possible to pinch the hose while holding the leveler to stop the flow of fluids very quickly.

5. Lower leveler until disorbent in pipette 1 is 1/8 inch from rubber tubing, see Figure 3
If solution in pipettes will not raise when there is still solution in the burette, check to see if the balloon in completely deflated. If so, remove the balloon, allowing it to fill slightly with ambient air.

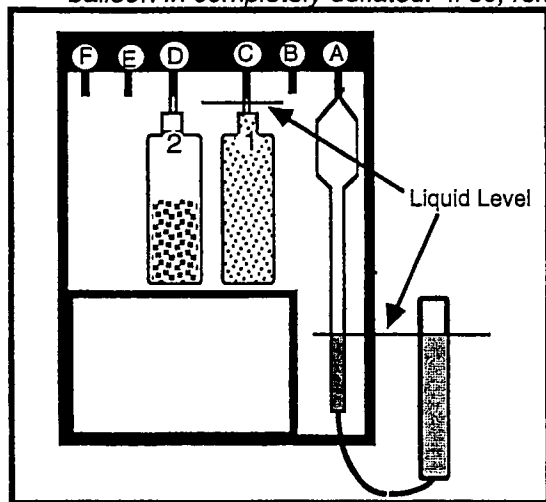


Figure 3

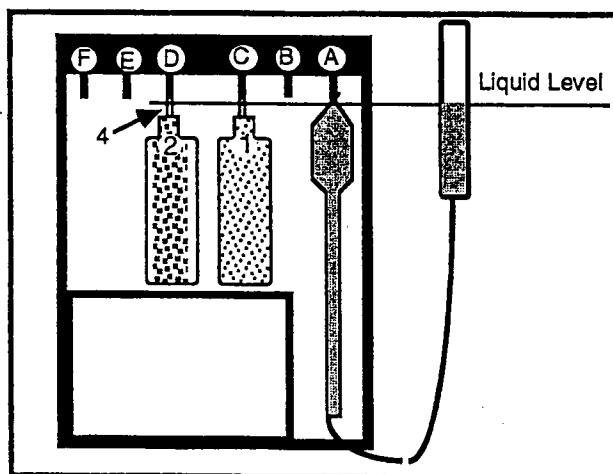


Figure 4

6. Close Valve C
7. Open Valve B
8. Raise the leveler until liquid in buret is in capillary, see Figure 2
9. Close Valve B
10. Repeat steps 4 through 9, substituting Valve D and oxisorbent for Valve C and disorbent.
11. Close all valves, liquids should be in capillaries, as shown in Figure 4

Leak Checks (quality control):

Orsat

1. Open Valves A and B
2. Lower leveler until solution in burette is down to zero.
3. Close Valve B while leaving Valve A open and hang leveler on orsat frame.
4. For an acceptable leak check over a four minute period:
 - a) the solution in the burette should not move more than 0.2 %
 - b) the pipette solutions shouldn't fall below the capillary

Collection Bag

1. Pressurize bag to 5 to 10 cm H₂O
2. Connect to water manometer
3. Allow to stand for 10 minutes
4. Any displacement in water indicates a leak

Alternative Collection Bag Leak Check

1. Pressurize bag to 5 to 10 cm H₂O
2. Allow to stand overnight
3. A deflated bag indicates a leak

Extraction of Stack Gas Sample:

Gas is extracted from the stack through a probe and particulate filter. The method of collection depends on the process being tested and the purpose of the test.

Single Point Grab Sample - not typically used by CAE

1. gas from the stack is pumped directly into the orsat (no collection bag)
2. Used for very short processes or to get immediate results
3. Probe should be located at the centroid of the stack cross section or no closer to the wall than one meter
4. purge the sample line and orsat with enough gas to allow at least five exchanges of gas
5. Analyze immediately and calculate the dry molecular weight
6. This method can also be used to determine if gas concentration is stratified (concentration of CO₂ or O₂ varies at different points within the stack)

Single Point Integrated Sample

1. This method used most often in conjunction with a moisture train to correct an emission rate or concentration of a gas pollutant to dry standard conditions
2. Gas is extracted from the stack and stored in a collection bag until analysis. In addition to the in-line filter, water is condensed and removed upstream of the bag
3. Purge the sample train and evacuate the flexible bag before attaching the bag
4. Probe should be located at the centroid of the stack cross section or no closer to the wall than one meter
5. Sample at a consistent rate simultaneous with the test period - try to collect at least 30 liters or one cubic foot.
6. Analyze the sample within eight hours.

Multi-Point Integrated Sample

1. This method used most often in conjunction with a sample train collecting pollutants in a particulate form which require a traverse of the stack. Results are needed to correct an emissions to dry standard conditions
2. Gas is extracted from the stack and stored in a collection bag until analysis. In addition to the in-line filter, water is condensed and removed upstream of the bag
3. Purge the sample train and evacuate the flexible bag before attaching the bag
4. The minimum number of points the probe should traverse during the test is eight for stacks having a diameter less than two feet, 9 for stacks having a equivalent diameter less than two feet, and twelve in all other cases.
5. Sample at a consistent rate simultaneous with the test period - try to collect at least 30 liters or one cubic foot.
6. Sample at each point for an equivalent length of time
7. Analyze the sample within eight hours.

Analysis of Stack Gas Sample:

Transferring the Sample into the Orsat

1. Make sure all valves are closed and liquids are 1/8 inch from rubber tubing (Figure 4)
2. Connect collection Bag to Orsat via rubber hose (Figure 1)
3. Open Valve B and F
4. Purge (replace) the gas in the Orsat with sample gas from the collection bag by squeezing bulb 10 to 15 times.

Draw an exact amount of the gas sample into the Orsat as follows:

5. Close valve B while orsat has positive pressure from the sample gas.
Prevents ambient air from being drawn into orsat.
6. Open valve A
7. Lower the leveler until liquid in the burette is **below** the lowest graduation
8. Close valve F
9. Force the solution in the burette to rise **slowly** to the zero mark
- Always take readings level with the bottom of the meniscus
10. **Tightly** pinch leveler tubing
11. Open Valve B to discharge the excess air, close valve B, and release the tubing
12. Holding leveler beside the burette, bring leveler elevation to a point where the liquid is even with the liquid in the burette. (All valves should be closed and the absorbing solutions in the pipettes should be in capillaries)
- If the above steps have been completed correctly, the liquid level in the burette will be at the lowest graduation.
- If not, repeat steps 3 through 12.

Determining the Carbon Dioxide and Oxygen Content

1. Raise the leveler and open valves A and C (clear solution)
Always keep your eye on the rising liquid, again, Do not permit solution to enter the manifold
2. Elevate the leveler until burette solution reaches the capillary
3. Carefully lower the leveler, drawing the gas sample back into the burette until the liquid in the pipette rises into the capillary at the top of pipette 1.
4. Repeat steps 2 and 3 three times
5. Close the valve above the pipette while the absorbent is in the capillary
6. Hold leveler beside burette at a point where the buret solution is at the same elevation in the leveler burette.
7. Record buret readings on the data sheet to the nearest 0.1 increment.
(Pipette 1 reading is % CO₂, pipette 2 is reading is % O₂ plus % CO₂)
8. Raise the leveler and open valves D (dark solution)
9. Repeat steps 2 through 7 for pipette 2
It may be necessary to jar orsat to dislodge ball in pipette 2 during step 3. If it remains in place, the absorbent may bubble into the manifold. Make certain ball is seated again before raising leveler.

Calculations

1. Repeat the gas sample analysis for CO₂ and O₂ shown above a total three times .
2. Three consecutive CO₂ and O₂ readings should not deviate more than 0.2 percent.
3. If deviation is greater than .2 percent, analyze more samples or replace chemicals.
4. Calculate dry molecular weights (M_d) as follows

$$M_d = 0.440(\%CO_2) + 0.320(\%O_2) + 0.28 (\%N_2 + \%CO)$$

$$\text{where } (\%N_2 + \%CO) = 1 - (\%CO_2 + \%O_2)$$

Note: Step 2 is a CAE QA limit. Method 3 states the analysis and calculations must be repeated until M_d differs from the mean by no more than 0.3 lb/lb-mole.

Comments and Trouble Shooting

- As chemicals become saturated with CO₂ and O₂, the rate of absorption decreases, along with absorption capacity. If saturation is suspected, take a reading after the 3 repetitive passes as normal, pass the gas through the chemical again, and take another reading. A different reading probably indicates saturated chemicals.
- Compare CO₂ to O₂ readings to the previous run. Depending on how stable the process is, a significant drop between runs indicates a
- Another indication of possible problem is the F₀ factor. If the F₀ factor is not within the expected range, check for saturated chemicals, a diluted sample, a loose balloon, or a orsat leak. F₀ factors can only be applied
- During use, disorbent will remain clear while oxsorbent will change from blue when fresh to green, but a color change does not necessarily mean the chemical is bad. Depending on the ratio of CO₂ to O₂, the chemicals will become saturated at different rates. Under typical conditions, oxsorbent must be changed several times before the disorbent becomes saturated.
- Remove chemicals from orsat before shipping.
- Buret solution can crystallize (freeze) at about 50° F
- Leak check IGS bag (beach ball) - Because orsats are
- Leak check after orsat has stabilized to ambient temperatures.
Temperature changes will cause gas to expanding or contract which will affect orsat readings.

The value of a standard procedures for everyone to follow was evident from the discussions which arose over procedures. The procedure for drawing the sample into the orsat wasn't resolved. Please comment on the differences between the procedures listed below and those in the document. Are they both acceptable? If so, the second method is probably easier and quicker.

Transferring the Sample into the Orsat

1. Make sure all valves are closed and liquids are 1/8 inch from rubber tubing (Figure 4)
2. If IGS bag (beach ball) is completely full, release enough gas to relieve pressure
3. Connect IGS Bag to Orsat via rubber hose (Figure 1)
4. Open Valve B and F
5. Purge (replace) the gas in the Orsat with sample gas from the IGS bag by squeezing bulb 10 to 15 times or by pressurizing (squeezing) sample bag.
6. Close valve B while orsat has positive pressure from the sample gas.
Prevents ambient air from being drawn into orsat.

Draw an exact amount of the gas sample into the Orsat as follows:

7. Open valve A
8. Lower the leveler until burette solution is **below** the lowest graduation (zero).
Always take readings level with the bottom of the meniscus
9. Raise leveler until the solution levels in both the leveler and the buret read zero.
10. Close valve F (all valves should be closed and the absorbing solutions in the pipettes should be in capillaries)

Also questioned was the need for a squeeze bulb and moisturizing tube. Several employees sited the frequency one of the two components will leak outweighs their value and will continue to remove them during use.

- How will not having a moisturizing tube affect the results
- Can you see any problems with simply squeezing the beach ball to force the sample into the orsat?

CAE STANDARD OPERATING PROCEDURE

DETERMINING NOZZLE SIZE AND K FACTOR

ISO NUMBER: EPA5-1

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

Materials:

Calculator

Paper & Pencil

Parameter Values (See example value in nomenclature table below)

Nomenclature Used:

Variable	Example Value	Units	Description
%O ₂	9.0 %	percent	percent of oxygen in the stack
%CO ₂	11.9 %	percent	percent of carbon dioxide in the stack
%N ₂	79.1 %	percent	percent of nitrogen in the stack
Bwo	0.0500	dimensionless	Moisture fraction (between 0 and 1)
Md	calculated below	pounds per pound-mole	Molecular weight of stack gas at meter (Dry Molecular weight)
Ms	calculated below	pounds per pound-mole	Molecular Weight of stack gas in stack
Ps	calculated below	inches of Mercury	Barometric Pressure in the stack
Pb	29.60 in. Hg	inches of Mercury	Barometric Pressure at the sampling location
St	-6.0 in. H ₂ O	inches of water	Static pressure in the stack
Ts	180 °F	degrees Fahrenheit	Stack temperature
/ΔP	0.9500 Root in.	square root of inches of water	Average of the square roots of delta P
ΔH@	1.800 in. H ₂ O	inches of water	Orifice reading when the meter pulls .75 cfm
θ	60 min	minutes	Duration of test
Tm	75.0 °F	degrees Fahrenheit	Temperature of the meter
Cp	0.84	dimensionless	Pitot calibration factor
RV	30.00 ft ³	feet cubed	Required Volume

Introduction:

K-Factor and Nozzle Sizing

The goal of isokinetic testing is to draw stack gas through the nozzle at a the same velocity as the gas in the sample stream. For example, if the stack gas velocity is 30 ft/sec, the velocity of the stack gas through the nozzle should also be 30 ft/sec.

The ratio of the nozzle gas velocity to the stack gas velocity equals the percent isokinetic. If the average velocities are the same for a test, the test is 100% isokinetic. To determine these velocities, you must know the following:

- the gas conditions in the stack (Ts, Ps, Ms, Bwo) and the ΔP reading
- the gas conditions at the meter box (Tm, Pb, Md) and the ΔH reading
- a few fixed constants (Dn, ΔH@, Cp)

Running an isokinetic test requires constant adjustment of the ΔH on the meter box (which controls nozzle gas velocity), based on the ΔP readings of the stack (which indicate the stack gas velocity). To simplify the calculations necessary in relating the two, a pretest calculation is performed. This calculation gathers all of the fixed constants, the gas conditions at both the stack and the meterbox, and simplifies them down to a single constant known as a K-Factor.

Once the K-Factor has been determined, simple arithmetic can provide you with the correct ΔH setting given any ΔP at the stack. The correct ΔH equals your K-Factor multiplied by the ΔP reading. Let's join uncle Jim in the Math room to find out how to calculate a K-Factor.

Step 1: Calculate the absolute pressure of the stack gas, Ps:

$$P_s = P_b + \frac{P_{static}}{13.6} \quad \text{Example: } P_s = 29.60 + \frac{-6.0}{13.6} = 29.159 \approx 29.16$$

Step 2: Calculate the dry molecular weight of the stack gas, Md:

$$M_d = 0.4(\% CO_2) + 0.32(\% O_2) + 0.28(\% CO + \% N_2)$$

$$\text{Example: } M_d = 0.4(11.9) + 0.32(9.0) + 0.28(79.1) = 30.264 \approx 30.26$$

Step 3: Calculate the molecular weight which includes the water present, Ms:

$$M_s = M_d(1 - B_{wo}) + 18(B_{wo})$$

$$\text{Example: } M_s = 30.26(1 - 0.050) + 18(0.050) = 29.651 \approx 29.65$$

Step 4: Calculate intermediate factor, K1:

$$K1 = 85(C_p)^2(\Delta H @ T_m) \left(\frac{T_m + 459.67}{T_s + 459.67} \right) \left(\frac{P_s}{P_b} \right) \left(\frac{M_d}{M_s} \right) (1 - B_{wo})^2$$

$$\begin{aligned} \text{Example: } K1 &= 85(0.84)^2(180) \left(\frac{75.0 + 459.67}{180 + 459.67} \right) \left(\frac{29.159}{29.60} \right) \left(\frac{30.264}{29.651} \right) (1 - 0.050)^2 \\ &= 818833 \approx 81883 \end{aligned}$$

Step 5: Calculate intermediate factor, D1:

$$D1 = 4934(C_p) \sqrt{\frac{P_s}{M_s(459.67 + T_s)}}$$

$$\begin{aligned} \text{Example: } D1 &= 4934(0.84) \sqrt{\frac{29.159}{29.651(459.67 + 180)}} \\ &= 15438 \end{aligned}$$

Step 6: Find the ideal nozzle size based on the V_{mstd} desired:

$$D_{n\ ideal} = \sqrt{\frac{V_{mstd\ desired}}{D1\Theta(\sqrt{\Delta P})_{ave}}}$$

$$\text{Example: } D_{n\ ideal} = \sqrt{\frac{30}{(1543)(60)(0.95)}} = 0.1846$$

Step 7: Find the closest larger nozzle and calculate the K-Factor.

$$KF = K1(D_{n\ actual})^4$$

$$\text{Example: } KF = 81883(0.187)^4 = 1.001 \approx 1.00$$

Step 8: Determine the highest ΔH based on the highest ΔP reading expected.

$$\Delta H_{highest} = KF(\Delta P_{highest})$$

$$\text{Example: } = 1.00(1.10) = 1.10$$

(If ΔH is too high, then reduce D_n to decrease the K factor, and increase the test time to pull the required V_{mstd} .)

Step 9: Calculate the estimated volume metered at STP, V_{mstd} :

$$V_{mstd} = D1\Theta(\sqrt{\Delta P})_{ave} D_n^2$$

$$\text{Example: } V_{mstd} = 1543(60)(0.95)(0.187)^2 = 30.77$$

Step 10: Calculate the estimated volume metered, V_m :

$$V_m = \left(\frac{2798(T_m + 459.67)}{P_b \left(P_b + \frac{\Delta H}{13.6} \right)} \right) C_p \sqrt{\frac{P_s}{M_s(T_s + 459.67)}} (\sqrt{\Delta P})_{ave} D_n^2 \Theta(1 - B_{wa})$$

Example:

$$\begin{aligned} &= \left(\frac{2798(75 + 459.67)}{1 \left(29.60 + \frac{0.9}{13.6} \right)} \right) 0.84 \sqrt{\frac{29.16}{29.65(180 + 459.67)}} (0.95)(0.187)^2 60(1 - 0.0500) \\ &= 31.44 \end{aligned}$$

CAE STANDARD OPERATING PROCEDURE

ISOKINETIC SAMPLING SET-UP

ISO NUMBER: EPA5-2

EFFECTIVE DATE: Pending

AUTHOR: Dept. 68

APPROVED BY: Pending

1. Perform preliminary velocity traverse. Calculate stack pressure, barometric pressure, average square root of ΔP 's and average stack temperature.
2. Using meter box data and assumed moisture, calculate intermediate parameter K_1 as follows:

$$K_1 = 850(C_p)^2(\Delta H_{\theta}) \left(\frac{T_m + 460}{T_s + 460} \right) \left(\frac{P_s}{P_b} \right) \left(\frac{M_d}{M_s} \right) (1 - B_{wo})^2$$

3. Determine the ideal nozzle size based on minimum required sample volume as follows:

$$D_{n,ideal} = \sqrt[4]{\frac{V_{mstd,desired} \sqrt{\frac{M_s(T_s + 460)}{P_s}}}{(493.4)C_p \theta (1 - B_{wo}) (\sqrt{\Delta P})_{avg}}}$$

4. Select the closest larger nozzle to the ideal nozzle size. Call this $D_{n,actual}$.

5. Using $D_{n,actual}$, calculate the K-factor as follows:

$$K = K_1 (D_{n,actual})^4$$

6. Calculate the maximum ΔH that would be expected from the selected nozzle as follows:

$$\Delta H_{max} = K(\Delta P_{max})$$

7. If ΔH_{max} is too high to reasonably obtain on the meter box, then select the next smaller nozzle size and recalculate the K factor and the ΔH_{max} . Repeat until a reasonable ΔH_{max} is obtained.
8. Calculate the estimated meter volume using the selected nozzle and the following equation:

$$V_{mstd,estimated} = (493.4)C_p \sqrt{\frac{P_s}{M_s(T_s + 460)}} (\sqrt{\Delta P})_{avg} (D_{n,actual})^2 \theta (1 - B_{wo})$$

If this meter volume is too low, then increase test duration until proper amount of volume can be obtained with the selected nozzle.

METHOD 5 SAMPLE TRAIN PRE-CLEANING

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AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

List of Materials: Sample Probe, Sample Nozzle, Impinger Bucket, Filter Holder, Cyclone Bypass, Connecting Glassware, Impingers, Nozzle Brush, Probe Brush, Glass Sample Bottles, Acetone, Kim Wipes, Lab Gloves, Safety Goggles

Definitions:

Sample Probe - Device inserted into stack or duct in order to withdraw gas sample and monitor flue gas temperature and velocity within the stack. The probe consists of a glass or stainless steel lined tube with a heating element. The probe also contains a Stausscheibe or S-Type pitot tube for measuring flue gas velocity, a thermocouple for measuring flue gas temperature, and a thermocouple for measuring probe heater temperature.

Sample Nozzle - The sample nozzle is attached to the inlet of the probe. The nozzle is made of glass or stainless steel in a button hook design with a sharp tape at the leading edge. The sample nozzles come in a variety of inner diameters in order to allow isokinetic sampling with a wide range of flue gas conditions.

Filter Holder - Contains and supports a glass fiber filter for collecting particulate matter in the sample flue gas. The filter holder consists of two round glass halves with a Teflon or quartz frit in between. The holder is held together by two phenolic rings which screw together compressing the glass halves and frit together. Filter holder is kept heated to 248 +/- 25°F in the filter heater box.

Cyclone Bypass - A piece of glass tubing that connects the exit of the sample probe to the filter holder inlet. It takes the place of a cyclone which is only used if there is a high concentration of large particulate or droplets in the sample flue gas stream.

Connecting Glassware - Consists of glass tubing with 90° or 180° bends to connect the filter holder to the 1st impinger and to connect the four impingers together.

Impinger - A glass bottle with a glass tube insert in the center. There are two types of impinger inserts, one with a restricted tip and one without a restricted tip. A series of four impingers is used to condense out any moisture present in the water each, the third is left empty, and the fourth contains a tared quantity of indicating silica gel as a desiccant. The impinger contents can also be analyzed for various pollutant compounds.

Definitions (Continued)

Nozzle Brush - A nylon bristle brush similar to a pipe cleaner or test tube brush used to clean and recover particulate from the sample nozzle.

Probe Brush - A nylon bristle brush similar to a test tube brush mounted onto a flexible Teflon rod sized for the length of the sample probe. The probe brush is used to clean and recover particulate from the liner of the sample probe.

Glass Sample Bottle - Wide mouth glass jar used to collect probe and nozzle rinses.

Acetone - A colorless organic solvent used for rinsing sample probes and nozzles. Acetone typically is shipped to job sites in 4 liter safety packs. Acetone is classified as a hazardous chemical and the Material Safety Data Sheets (MSDS) should be consulted before handling.

Acetone Squeeze Bottle - Teflon squeeze bottle clearly labeled to contain acetone.

Kim Wipes - Laboratory tissue similar to Kleenex, but with extremely low amount of lint.

Lab Gloves - Can be surgical type latex gloves or other latex or neoprene type glove designed to protect hands from hazardous chemicals.

Safety Goggles - Eye protection that seals around area of face surrounding the eyes.

Procedure Steps:

Note: This procedure should be performed before assembly of the sample train.

1. Wipe off outside of sample probe, impingers, cyclone bypass, and connecting glassware if needed.
2. Put on safety goggles and lab gloves.
3. Fill acetone squeeze bottle with acetone from safety pack if needed.
4. Rinse out several new glass sample jars and set them aside. These will be used to collect pretest impinger rinse blanks and probe and nozzle pretest rinse blanks.
5. Rinse the inside of all connecting glassware, the cyclone bypass, and the glass filter holder halves 3 times with acetone. Rinses should be performed over a sink or waste chemical bucket.

Procedure Steps (Continued)

6. Rinse all connecting glassware again as in step 4 except this time collecting the rinses in a glass sample jar.
7. Secure the lid tightly on the sample bottle and wrap Teflon tape around the edge of the lid to serve as a custody seal and to help prevent leakage.
8. Label the sample jar "Pretest Impinger Rinse Blank". Also include on the label the date, job number, client name, job site, test location name, and reagent used (Acetone in this case). Also mark the level of the liquid in the jar.
9. Rinse off the nozzle brush and probe brush thoroughly.
10. Squirt acetone onto several bunched up Kim Wipes and wipe down the length of the rod attached to the probe brush.
11. Place the sample probe at an angle so that the back or outlet of the probe is facing down. This will probably require a second person to hold the lower end.
12. Rinse out the inside of the probe thoroughly with acetone. Rotate the probe while rinsing to be sure to get all surfaces.
13. Insert a freshly rinsed probe brush into the inlet of the probe. Insert it just far enough so that all the bristles are an inch or two from the inlet of the probe. Use a Kim Wipe damp with acetone to hold onto the probe rod while you insert the brush.
14. Squirt a generous amount of acetone into the probe.
15. Insert the probe brush and probe rod down the full length of the probe until it reaches the other end. Manipulate the probe rod in the same manner as in step 13.
16. Again squirt a generous amount of acetone into the probe.
17. Slowly pull the probe rod back toward the inlet of the probe until the bristles are once again an inch or two from the inlet of the probe.
18. Repeat steps 14 through 17 two more times for glass line probes, five more times for steel lined probes.
19. Have the person at the lower end hold a new, pre-rinsed glass sample jar at the outlet of the probe.

Procedure Steps (Continued)

20. Rinse out the inside of the probe thoroughly with acetone, this time collect the rinse into the sample jar. Rotate the probe while rinsing to be sure to get all surfaces.
21. Place the lid back onto the sample bottle
22. Rinse out sample nozzle from both ends with a generous amount of acetone.
23. Squirt a small amount of acetone into the nozzle while holding it in such a way as to keep the acetone from running out.
24. Insert the nozzle brush into one end of the nozzle and gently brush in and out to loosen any possible particulate buildup that might be present.
25. Repeat steps 8 and 9 brushing from the other end of the nozzle.
26. Rinse the nozzle out thoroughly again with acetone.
27. Rinse the nozzle thoroughly with acetone, this time catching the rinse in the glass sample jar used for catching the probe rinse.
28. Secure the lid the same as in step 8.
29. Label the sample bottle "Pretest Nozzle and Probe Rinse". Include the rest of the information included on the label in step 7.

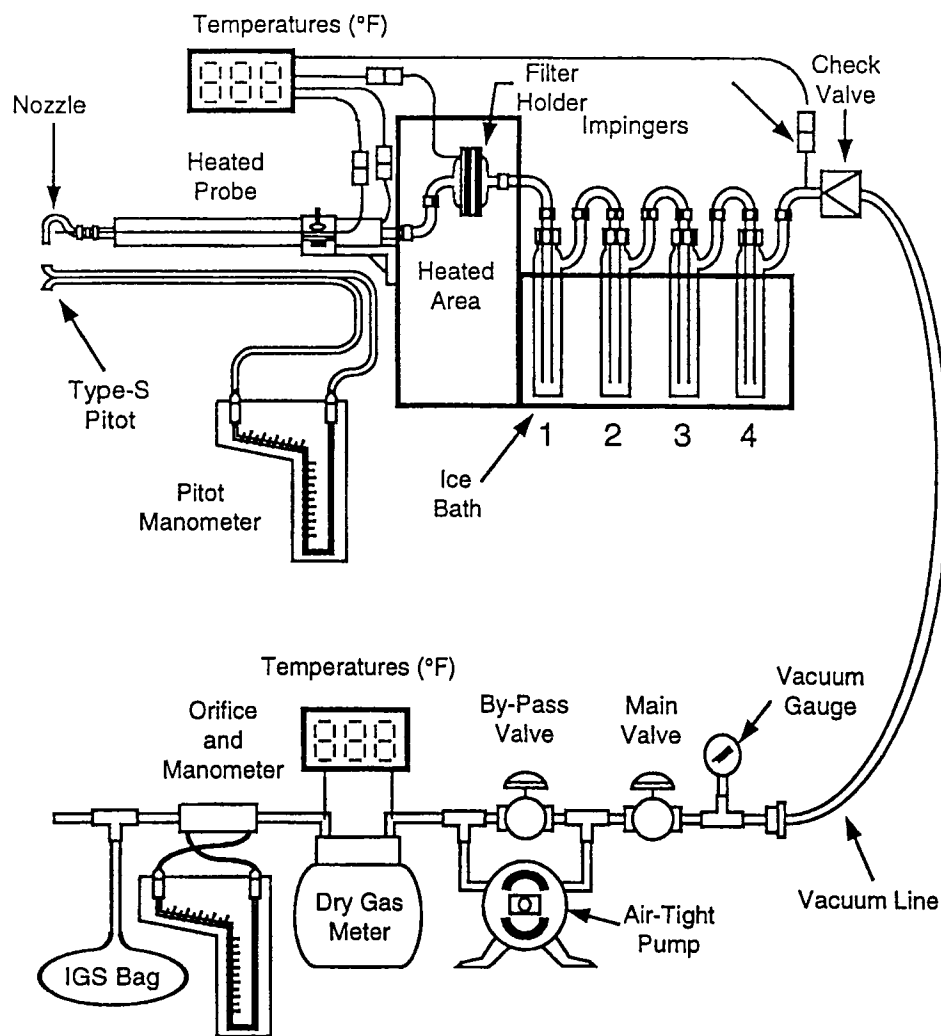


Figure 1: Standard M5 Sample Train



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METHOD 5 SAMPLE TRAIN ASSEMBLY

ISO NUMBER: EPA5-4

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

List of Materials: Meter Box Assembly, Sample Probe, Sample Nozzle, Filter Heater Box, Impinger Bucket, Impinger Outlet, Umbilical, Filter Holder, Cyclone Bypass, Connecting Glassware, Impingers, Silicone Grease

Definitions:

Meter Box Assembly - Consists of control console and pump box, see meter box assembly procedure. The meter box assembly controls and monitors operation of the sampling train.

Sample Probe - Device inserted into stack or duct in order to withdraw gas sample and monitor flue gas temperature and velocity within the stack. The probe consists of a glass or stainless steel lined tube with a heating element. The probe also contains a Stausscheibe or S-Type pitot tube for measuring flue gas velocity, a thermocouple for measuring flue gas temperature, and a thermocouple for measuring probe heater temperature.

Sample Nozzle - The sample nozzle is attached to the inlet of the probe. The nozzle is made of glass or stainless steel in a button hook design with a sharp taper at the leading edge. The sample nozzles come in a variety of inner diameters in order to allow isokinetic sampling with a wide range of flue gas conditions.

Filter Heater Box - The filter box contains the filter holder and a heating element. The filter box is the support point for the entire sample train. Both the sample probe and the impinger bucket attach to the filter box. The amphenol line in the umbilical connects to the filter box to supply power for the probe and filter heaters.

Impinger Bucket - Contains the impingers, also referred to as the back half of the sample train. Once the impingers are loaded the impinger bucket is filled with ice to chill the impingers in order to condense out any moisture present in the sample flue gas.

Impinger Outlet - Connects exit of last impinger to the sample line in the umbilical. It is also equipped with a thermocouple to monitor sample gas temperature.

Umbilical - Flexible conduit between meter box assembly and probe/filter box/impinger bucket. Umbilical contains pitot lines, thermocouple lines, a gum rubber sample line, and amphenol line. The amphenol line is used to supply power from the meter box assembly to the sample train.

Definitions (Continued)

Filter Holder - Contains and supports a glass fiber filter for collecting particulate matter in the sample flue gas. The filter holder consists of two round glass halves with a Teflon or quartz frit in between. The holder is held together by two phenolic rings which screw together compressing the glass halves and frit together. Filter holder is normally heated to 248 +/- 25°F in the filter heater box.

Cyclone Bypass - A piece of glass tubing that connects the exit of the sample probe to the filter holder inlet. It takes the place of a cyclone which is only used if there is a high concentration of large particulate or droplets in the sample flue gas stream.

Connecting Glassware - Consists of glass tubing with 90° or 180° bends to connect the filter holder to the 1st impinger and to connect the four impingers together.

Impinger - A glass bottle with a glass tube insert in the center. There are two types of impinger inserts, one with a restricted tip and one without a restricted tip. A series of four impingers is used to condense out any moisture present in the gas stream. The impinger contents can also be analyzed for various pollutant compounds.

Silicone Grease - Vacuum grease used to help seal between ground glass connections.

Procedure Steps:

Refer to Figures 1 through 4

1. Assemble the Meter Box per Meter Box Assembly Procedure.
2. Connect the umbilical to the Meter Box in the following manner
 - A. Find the end of the umbilical with the 5 male thermocouple plugs and male amphenol connector.
 - B. Connect the male thermocouple plugs numbered 1 through 4 into their respective receptacles on the meter box control console.
 - C. Connect the amphenol line to the blue and olive connection labeled "loosen" with a clockwise arrow above it. Align and insert the connectors then screw the collar counter-clockwise onto the meter box.

Procedure Steps (Continued)

- D. Connect the two tygon pitot lines in the umbilical to the connections labeled "Pitot". One of the pitot lines should be marked with a piece of tape. Connect this line to the pitot connection marked "+". Connect the other line to the pitot connection marked "-".

Depending on the meter box it may be necessary to use pitot line quick connects to connect the lines to the sample box. Simply push the tygon line over the longer side of the quick connect and push the quick connect into the quick connect socket on the meter box until it clicks.

- E. Push the sample line over the meter box fitting quick connect and then insert the quick connect into the quick connect socket labeled "Sample Inlet" until it clicks.
3. Clamp the sample probe to the support arm on the Filter Heater Box. If testing a vertical duct or stack align the pitot tips vertically. If testing a horizontal duct align the pitot tips horizontally.
 4. Plug the Probe power cord into the AC outlet on the Filter Heater Box.
 5. Connect the free end of the umbilical to the probe and filter heater box as follows:
 - A. Connect the amphenol line to the filter heater box. The amphenol line has a female connector with a collar which screws down around the male connector on the filter heater box. Be sure the collar is screwed down snugly.
 - B. Connect the pitot lines to the pitot tubes on the probe. Connect the line which is marked with tape to the positive pressure or impact side of the pitot. This is the side that will be facing into the gas flow during testing. When testing a conventional stack the positive pressure side is the side facing down.
 - C. Connect the female thermocouple plug #1 on the umbilical to the male stack thermocouple on the probe. The stack thermocouple plug is on the back or exit end of the probe. The stack thermocouple runs down the same side of the probe the pitot tube is located. It is usually a solid silver rod, but it may be a brown braided wire.
 - D. Connect female thermocouple plug #2 on the umbilical to the male probe heater thermocouple. The probe heater thermocouple wire comes out of the very end of the probe sheath at the exit end. It is always a flexible wire with a male thermocouple plug on the end.

Procedure Steps (Continued)

- E. Connect the male thermocouple plug #3 on the umbilical to the female filter heater box thermocouple jack. Plug #3 is the only male plug on the sample train end of the umbilical. The filter heater box thermocouple jack is the only thermocouple jack on the filter heater box.
- 6. Slide the impinger bucket onto the back of the filter heater box and secure the clasps.
- 7. Place four impingers into the cutouts in the bottom of the impinger bucket. Start with the cutout closest to the outlet of the filter heater box and call that impinger #1. Follow with impingers #2, #3 and #4 going clockwise around the bucket (See diagram below). Impingers #1, #3 and #4 have non-restricted inserts while impinger #2 has the restricted insert.
- 8. Place 100 ml of distilled water into impingers #1 and #2. Measure 100 ml with a glass graduated cylinder.
- 9. Place a tared quantity of silica gel into impinger #4 as follows:
 - A. Fill a 500 ml polyethylene storage bottle with silica gel until approximately half full.
 - B. Mark the storage bottle so that it can be identified.
 - C. Weigh the storage bottle with lid on an electronic balance or triple beam balance.
 - D. Add or remove silica gel until the bottle weighs exactly 300 grams.
 - E. Remove the stem from the fourth impinger.
 - F. Pour the silica gel from the storage bottle into the impinger. The storage bottle does not have to be completely emptied into the impinger; After the sample run the silica gel will be recovered back into the same storage bottled and re-weighed.
- 11. Place a 90° glass connecting piece on the top of the insert part of impinger #1. The top of the insert is always the impinger inlet and the tube or arm coming out of the side of the impinger is always the outlet. Slide the 90° piece into the screw cap and O-ring and screw onto the top of the impinger until snug. **DO NOT OVER-TIGHTEN SCREW CAPS!**
- 12. Connect the outlet of impinger #1 to the inlet of impinger #2 with a 180° glass connecting piece.

Procedure Steps (Continued)

13. Connect the rest of the impingers the same way. (i.e., impinger 2 to impinger 3 and impinger 3 to impinger 4)
14. Slide the impinger outlet into the bracket on the side of the impinger bucket. The outlet of impinger #4 should be adjacent to the bracket. Line up the inlet of the impinger outlet with the outlet of impinger #4 and secure with screw cap. Make certain that the inlet of the impinger outlet extends well below the O-ring and into the impinger outlet arm.
15. Slide the sample line onto the exit of the impinger outlet.
16. Insert the unthreaded end of the cyclone bypass to the filter holder inlet. The filter holder inlet is the side with the 90° bend. The cyclone bypass is attached to the filter holder with a screw cap.
17. Place the filter holder/cyclone bypass assembly into the filter heater box. The large, threaded end of the cyclone bypass should face the probe side of the box and the filter holder outlet should fit through the hole in the impinger bucket side of the box. A "sling" made from duct tape or glass tape may be useful in supporting the filter holder during assembly.
18. Connect the cyclone bypass to the outlet of the probe liner with a screw cap. Make certain the probe liner extends far enough into the cyclone bypass to clear the O-ring.
19. Connect the outlet of the filter holder to the 90° glass connector on impinger #1 with a screw cap.
20. Attach the appropriate size sample nozzle to the front or inlet of the sample probe as follows.
 - A. Cup and Socket style nozzle:
 1. Smear a sparing film of silicone grease around cup of nozzle. (Omit this step for high temperature applications)
 2. Insert the cup of the nozzle into the socket at the inlet of probe liner.
 3. Turn the nozzle so that points into the flow.
 4. Safety wire the nozzle to the probe by winding Ni-Cr wire around the small hooks on the nozzle and the rim of the socket on the probe liner.

Procedure Steps (Continued)

- B. Barrel (Nut and Ferrule) style nozzle:
 - 1. Insert nozzle barrel into union at end of probe liner.
 - 2. Finger tighten nozzle nut onto union on probe liner.
 - 3. Align Nozzle with pitots so that it faces into the flow.
 - 4. Finish tightening nut onto union using two crescent wrenches

- 21. The sample train is now assembled and ready to be heated up and leak checked.

PREPARATION AND HANDLING OF TARED GLASS FILTERS 8.26CM-11CM

ISO NUMBER: EPA5-5

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

Purpose: This procedure provides the steps for proper preparation and handling of tared glass fiber filters 8.26cm-11cm for use in EPA Method-5 and state equivalents.

Scope: This procedure applies to any CAE employee who works in the gravimetrics lab.

Materials:

- Powder free lab gloves
- Plastic or metal tweezers
- Filter Inventory Log
- Whatmans 934-AH glass fiber filters 8.26cm-11cm
- Disposable plastic petri dishes 10cm or 15cm
- Inked numbering machine with ink refill
- Stamping board
- Boekel desiccator or equivalent
- Drierite
- Anti-static brush
- Class S calibration weights 0.5g, 1.0g, 100g
- Calibration Log
- Ohaus GA110 balance
- Pad of particulate weight sheets
- Black marker
- Black pen
- Kimwipes
- Post-Its
- Small plastic desiccator
- Tared Media Log

Definitions:

- Balance:** Refers to the Ohaus GA110 analytical balance.
- Cal. weight:** Refers to Ohaus class S calibration weight.
- Clean:** Refers to a condition free of particulate or foreign substances that may contaminate a filter.
- Constant weight:** Two consecutive weights taken ≥ 6 hours apart with a difference $\leq \pm 0.0005$ g between the two weights
- Desiccator:** Refers to stainless steel Boekel desiccator.
- Drierite:** Refers to anhydrous calcium sulfate (CaSO_4).
- GFF:** Refers to Whatmans 934-AH glass fiber filter media 8.26cm-11cm.
- Gloves:** Refers to VWR brand disposable lab gloves.
- Lab:** Refers to the gravimetrics lab area.
- Petri dish:** Refers to disposable plastic petri dishes in 10cm or 15cm.
- Stamper:** Refers to the Bates Numbering Machine.

PREPARATION AND HANDLING OF TARED GLASS FILTERS

8.26CM-11CM

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General: In most particulate testing a preweighted GFF is required. EPA Method-5 is the most commonly used and referenced particulate test method. The following procedure is based on the guidelines set forth by EPA Method-5 for creating preweighted or "tared" GFF's. A copy of Method-5 appears in appendix A, this method should be reviewed prior to attempting this procedure.

IMPORTANT: In order to prevent contamination during the course of this procedure never handle GFF's with bare hands, always use clean tweezers or clean gloves. Always keep GFF's in their original box or in a clean clearly marked covered petri dish when not in use. "Remember to keep the workspace clean too!"

Procedure:

I. Untared GFF's: All GFF's are received in an unweighted state. These filters are stored in the glass cabinet on the west wall of the lab. The filters are organized according to type and size. Any additions or subtraction's to the stock should be noted in the Filter Media Inventory Log which is also located in the cabinet(see example in appendix B). Any subtraction's from a single box of filters should be also noted on that box. Affix a Post - It to the lid noting the number of filters removed from this box, also use to note any future subtraction's.

II. Numbering GFF's: All tared filters are assigned a unique and sequential number . GFF's are the only filters that can be stamped using an inked stamper. The step to stamp GFF's is as follows.

1. Obtain desired size of GFF's from glass cabinet and, using tweezers or gloves, transfer the needed amount to a 15cm petri dish.
2. Adjust inventory according to I and return unused filters to the cabinet.
3. Obtain the stamper from the drawer in the center aisle of the lab.
4. Adjust stamper to reflect appropriate number series for that filter.
Refer to appendix C for procedure on setting up and adjusting stamper.
5. Place stamping board white side up and clean surface with a kimwipe.
Note: stamping board is located in the glass cabinet on the west wall.
6. Using tweezers, remove filter from petri dish and place rough side down on the stamping board.
7. Carefully stamp the number in the center of the filter.

REMEMBER: Too much pressure can rupture the filter!!

8. Pick up filter with tweezers and check filter against the light to make sure no break through has occurred and that printing is legible.
 - A. If break through has occurred or printing is illegible, note the filter number and a brief reason for voiding, then dispose of that filter in the trash. This information will be used later to account for all filter numbers in this series.
 - B. If no break through has occurred and printing is legible, place the filter in the unused half of the petri dish.

PREPARATION AND HANDLING OF TARED GLASS FILTERS

8.26CM-11CM

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9. Continue repeating steps 6-8 until all filters have been stamped. Filters are now ready for the desiccator.
10. Check the Drierite in the dessicator.
 - A. If Drierite is blue/purple continue with this procedure.
 - B. If Drierite is pink, refer to appendix C and follow the procedure on changing and regenerating the Drierite.
11. Place the uncovered petri dish of stamped filters in the dessicator. Be sure to turn the handle clockwise till it can go no further, this will assure that the desiccator is tightly sealed.
12. Place a Post-It indicating the filter type, 0 weight, time, and date, on the door. Be sure to place Post-It directly in front of the filters, especially if there are other items in the dessicator.

IMPORTANT: STOP: These filters must be desiccated for ≥ 24 hours before continuing this procedure.

III. Taring GFF's: These filters will be used to measure small amounts of particulate at times less than .0010g, so it is critical that an accurate tare weight is recorded for each filter. This is achieved by weighting the filters to a constant weight which is defined as, two consecutive weights taken ≥ 6 hours apart with a difference $\leq \pm .0005$ g between the two weights. If this criteria is met, the filters are ready for use.

WARNING: Again, Do not continue this procedure unless filters have been desiccated for ≥ 24 hours.

1. Obtain a pad of Particulate Weight Sheets and black pen from the drawers in the balance table.
2. Place the small plastic desiccator between the two balances.
3. Remove the filters from the Boekel desiccator and transfer to small desiccator. Place the unused half of the petri on the bottom shelf and place the half with the filters on the top shelf. Make sure that the filters are in sequential order beginning with the first number in the series.

IMPORTANT: Do not expose desiccated filters to the lab environment for more than 2 minutes. Keep in a dessicator until needed.

4. Calibrate the balance and reset to 0. (Refer to appendix C for procedure for calibration and operation of balance.)
5. Remove the first filter from the small desiccator and place it on the center of the weight pan. Then make sure that all balance doors are tightly closed.
6. Allow balance to stabilize.

PREPARATION AND HANDLING OF TARED GLASS FILTERS

8.26CM-11CM

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7. Record the filter number in the Sample I.D. column and then record the weight in the first cell of the Tare Weight column followed by the date and time that weight was obtained. Time can be record in 5 minute periods. Usually 4 to 5 filters can be weighted in this time period, so each filter can be given the same time.(See example)
8. Remove filter from weight chamber and place in the petri dish halve on the bottom shelf.
9. Rezero balance if necessary, then repeat steps 5-8 until all filters have a first weight.
10. Transfer the first weight filters back to the Boekel desiccator.

IMPORTANT: STOP: These filters must desiccate for ≥ 6 hours before continuing this procedure.

11. Repeat steps 2-7, step 4 may be skipped if the balance was calibrated earlier in the day. Record the second weight along with the corresponding information.(see example) These two weights must have a difference $\leq \pm 0.0005g$ to be a valid tare weight.
 - A. If this criteria is met, place the filter in a individual petri dish and write the filter number on the lid with a black marker. Repeat process until all filters have been weighted. Note: To save space, completed filters may be stored in a single petri dish and put into individual petri dishes at a later time
 - B. If this criteria is not met place those filters back in the Boekel desiccator and desiccate for ≥ 6 hours and then repeat this step until a consecutive weight is obtained. If more than three weights are necessary consult lab supervisor before continuing.
12. Tape the lids of the petri dishes with scotch tape, a small piece on each side is sufficient.

IV. Relinquishing Tared GFF's: Tared filters are stored in the Glassware Preparation Lab. Only tared filters meeting the criteria of this procedure should be relinquished to this storage area. In order to achieve this the analyst is responsible for recording the completed filters in the Tared Media Log which is maintained in the Gravimetrics Lab.

IMPORTANT: If there any uncertainty about the filters, contact the lab supervisor. Do not remove any filters until problem is resolved and you have verified that filters have a constant weight.

1. Record filters in the Tared Media Log (see example) This information includes media type, number series, date completed, analysts initials, and receiving lab personnel's initials. It is important that this information be completed to more accurately track tared media.
2. Take the completed filters and the Tared Media Log over to the Glassware Preparation Lab. Relinquish filters to lab personnel and have them initial in the appropriate space in the log.
3. Return the Tared Media Log to its appropriate space in the gravimetrics lab

CAE STANDARD OPERATING PROCEDURE

CARB 501 ANDERSON IMPACTOR

ISO NUMBER: CARB501-1

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

The Anderson impactor should be kept clean, dry and assembled when not in use. Before using, examine the jet plates by holding them up to light. If any hole is plugged, first try to blow out any dirt with compressed air, then wash with a detergent-water solution and sponge then air dry. The holes are precision drilled and must not lose their size or shape through cleaning with abrasives or corrosive materials. Handle the plates only by their edges.

PRE TEST PROCEDURE:

1) Choose a representative sample point based on a preliminary traverse, preferably near the middle of the gas stream. The gas velocity should be relatively stable and close to the overall average.

2) Just prior to starting to sample, determine ΔP , T_s , M_s and P_s at the sample point chosen. Calculate the following:

- Gas velocity (ft/sec) at sample point:
$$\text{Velocity} = 85.49 \times 0.84 \times \sqrt{\Delta P \times [(T_s + 460)/(M_s \times P_s)]}$$
- Required flow rate (acfm) through impactor:
$$Q_i = 60 \times \text{vel} \times A_n$$

Test this calculation with several nozzles to arrive at a nozzle that will achieve Q_i of 0.2 to 0.5 cfm. It may be acceptable to exceed this range under some circumstances as explained later, but Q_i must never exceed 0.7 cfm.

After determining the nozzle required, calibrate your closest available nozzle and determine Q_i specifically for that nozzle.

- Flow rate (dcfm) through dry gas meter:
$$Q_m = (Q_i) \times Y_d \times (460 + T_m / 460 + T_s) \times (P_s / P_m) \times (1 - B_{wo})$$

3) The sample period must be based on the particulate loading. A preliminary particulate test (M17) will be required if the loading is unknown. If you proceed to sample without knowing the grain loading, the first particle size test run will probably be void because the amount of sample collected will not be in the required range.

All impactor stages are limited to a maximum weight gain of 10 mg. To obtain a measurable sample, and at the same time avoid overloading any individual stage, use 0.5gr (32 mg) total particulate catch as your target.

$$\text{Sample period (min)} = 0.5 \text{ gr} / (Q_m \times \text{Estimated gr/dscf})$$

EXAMPLE:

Loading = 0.01 gr/dscf, Ts = 350° F, Ps = 29.5" Hg, Ms = 28, Bwo = 0.1, ΔP = 0.2" H₂O.

- Velocity = $85.49 \cdot 84 \cdot \sqrt{.2 \cdot [(350+460)/(28 \cdot 29.5)]}$
= 31.8 ft/sec
- Impactor flow rate (Qi) = $60 \cdot 31.8 \cdot A_n$
= 0.366 cfm (3/16" nozzle, $A_n = 1.918 \times 10^{-4}$)
= 0.65 cfm (1/4" nozzle, $A_n = 3.409 \times 10^{-4}$).
Since 0.65 cfm is beyond the preferred range for Qi (0.2 to 0.5 cfm), use the smaller nozzle.
- Meter flow rate (Qm) = $.366 \cdot (460 + 75/460 + 350) \cdot (29.5/29.6) \cdot (1 - 0.1)$
= 0.22 cfm
- Sample time (min) = $0.5 \text{ gr} / (0.01 \text{ gr/dscf}) / (0.22 \text{ cf/min})$
= 227 min
- Sample volume at meter (cf) = $0.22 \text{ cf/min} \cdot 227 \text{ min}$
= 49.94 cf

For this example, sample for 227 minutes @ 0.22 cfm on the meterbox using a 3/16" nozzle. The flow rate through the impactor will be 0.366 cfm. The total sample collected should weigh approximately 0.5 grains or .032 grams.

As a check on these calculations:

- 0.5 grains of sample @ 0.01 gr/dscf loading will require: $0.5/0.01 = 50 \text{ dscf}$
- 50 dscf @ 0.22 cfm will require: $50/.22 = 227 \text{ minutes}$

SAMPLING:

Assemble the sample train without the impactor. Leak check the train from the probe tip to insure an acceptable leak rate (<.02 cfm @ 15" Hg). Install the impactor, then leak check at low vacuum (≤5"Hg). Small leaks are acceptable, do not tighten the impactor in an attempt to stop all leaks. Over tightening will destroy the substrates by cutting off their outer edges.

Position the impactor in the duct with the nozzle capped and directed out of the gas flow. Leave it in this position for least 30 minutes, allowing the impactor to thoroughly pre-heat. When sampling after a wet scrubber, preheat the impactor in an oven (sample box oven) to above the stack temperature, then let it cool in the stack for 15 minutes before beginning sampling. This will assure that the impactor is at the gas temperature, if droplets are not present in the gas, no condensation problems should occur. Gasses with free moisture droplets will still cause a problem and require other strategies to be successfully sampled.

SAMPLING (continued):

At any time that the impactor is in the duct but sampling is not occurring, the nozzle must be aimed out of the gas flow. It is also good practice to also keep the nozzle capped. Keep in mind that since the sample is extremely small, virtually any amount of accidentally captured particulate will skew the test results.

Sample at the meter rate (Q_m) determined as shown in the example. A K-factor can be used to approximate Q_m , however Q_m should also be timed on a stopwatch and adjusted as required. Maintain Q_m until the run is completed. Only one point should be sampled per run unless the gas flow is very uniform. If uniform, several points may be sampled out of ONE port, all at the same sampling rate. Do not attempt to change ports with the same impactor. **Be extremely careful not to bump impactor during or after sampling.**

At the end of the required sample time, close the meter valve, then turn the nozzle out of the gas flow and withdraw the impactor from the sample port. **DO NOT leak check after the sample run.** Return the impactor and the data sheets to the field lab area immediately after the sample run. **Handle the impactor very carefully, do not bump or shock it in any way, and do not turn it upside down!!**

At least two sample runs must be performed at each point location to obtain repeatable data.

SAMPLING SUMMARY:

Insure that the probe and sample train leak check without the impactor on the probe before installing the impactor on the probe. Then leak check the system including the impactor at a low vacuum (~5"Hg). Once the impactor is installed on the probe, don't worry about small leaks. Over tightening the impactor will cut the substrates and introduce large errors. Use Teflon tape on the impactor threads and hand tighten only until snug.

Pre-heat the capped impactor out of the flow for at least 30 minutes. Un-cap the nozzle, turn on the pump with the nozzle aimed out of the flow. Quickly turn the impactor around to point the nozzle into the flow and set the sampling rate to (Q_m) using a K factor. Once set, time the meter rate with a stop watch and adjust as necessary to maintain Q_m . Do not change flow rate through impactor during the test. Turn the impactor out of the flow at the end of the run after closing the pump valve.

Extreme care must always be taken to avoid knocking the sample off of the substrates. Insert and remove the probe carefully without bumping the impactor on the port. Even the "click" of a vice grip can disturb the collected piles of particulate. Hold the impactor upright after use and carefully transport it to the lab.

NEVER PERFORM A LEAK CHECK FOLLOWING A SAMPLE RUN. Leak checks after the sample run will disturb the collected sample, changing the particulate distribution inside the impactor, destroying the size distribution information.

WEIGHING:

The laboratory must be clean, vibration free and temperature controlled. Careful weighing of the substrates is very important because the amount of sample collected is extremely small. Weights are taken to 0.00001 g, the maximum acceptable individual sample weight is 0.001 g. Ideally, the CAHN electro-balance should be turned on and warmed up over night prior to use. Zero and calibrate the balance before weighing each substrate set. Desiccate substrates in open petrie dishes WITH their foil sheets for at least 24 hours prior to weighing. Weigh each substrate with its aluminum foil sheet. It is critical that the same match of each substrate with its' foil sheet be maintained for post test weighing. The foil sheets can be numbered BEFORE obtaining tare weights.

LOADING THE IMPACTOR:

Refer to the diagram attached for the order of assembly. There are two types of substrates, designated 'even' and 'odd', and a backup filter disk. Place each substrate on top of it's plate with the substrate number up. Insure that no jet holes are covered by a substrate. Line up each plate notch with one edge of the substrate plate holder. Store the aluminum sheets in the dessicator while sampling.

POST-TEST:

Post-test weights must be performed on site as soon as possible. Minimize handling and any possible disturbance to the samples.

Disassemble the impactor as soon as possible. Immediately place the whole plate / substrate holder assembly in the dessicator to cool. Brush out the preseparator (if used) onto a separate tared sheet and place this in the dessicator also.

Let the substrates cool and dry in the dessicator for an hour or so before weighing. Weights can be obtained as soon as the substrates have equilibrated to room temperature.

Individually remove each substrate from the holder and put on the proper foil sheet, then brush any dust on the plate onto the same foil sheet. Weigh the matched substrate and foil sheet together before proceeding to remove the next substrate from the holder. It is not necessary to obtain multiple weights for each substrate, one weight per substrate is sufficient.

Zero and calibrate the balance before weighing each substrate set.

POST TEST DATA REDUCTION:

On site data entry is required for particle size sampling, since this is the only way to insure that the sampling was been successful. Use only an approved spreadsheet for data reduction and interpretation.

Insure that the gas composition data is entered in the proper format (wet or dry basis), for the spreadsheet that you are using. Use gas composition data from the actual sample run whenever possible (measure the water collected and do orsat analysis). Short runs (≤ 30 min.) may require a parallel sample train or other means to obtain gas composition data.

Particulate captured in the preseparator, the nozzle, and inside of the cone are considered a part of the first impactor stage's particulate catch.

POST TEST DATA REVIEW:

To determine if a given run is acceptable or if a replacement run will be required, examine the following items:

1. % isokinetic 100 \pm 10%
2. Flow (Q_i) through impactor was \leq to 0.5 cfm.
3. No single impactor stage gained over 10 mg. of weight.
4. The particulate was distributed through the impactor with the heaviest stages towards the middle and the lightest stages being towards the ends.
5. A visual inspection of the substrates reveals no evidence of rough handling or of particulate bouncing. The particulate on each substrate should be accumulated in discrete dots or cone shaped piles, if rough treatment occurred, the dots will be broken or smeared.
6. The run compares well in terms of general size distribution and mass mean particle diameter with the other runs made at the same location. Any significant changes in the size distribution between runs must have an explanation associated with the change. Dissimilar runs must be repeated until at least two repeatable samples have been collected.

ADDITIONAL NOTES:

The first test run should be considered an experiment. The safest procedure is to recover and complete data entry for the first test prior to performing additional runs. Make any required adjustments for the second sample run after checking the first run for proper substrate loading and isokinetics. At least two additional runs will still be required if the first sample run did not work out.

Sample times can vary from 30 seconds to several hours, depending on the particulate loading, gas velocity and sample rate. Use the results of the first run to adjust the sample time.

The range of diameters classifiable by the impactor is typically about 0.5 to 10 μ , the actual cut sizes changes with each sample run. Cut size is a function of gas viscosity (composition & temperature) and sample rate (meter operation, calibration & nozzle diameter).

The actual cut points can be manipulated by changing the flow rate through the impactor Q_i . Accomplish this by changing the nozzle diameter. A higher flow rate (larger nozzle) will result in smaller cut diameters on each stage. This is useful if the particulate is very fine. A lower flow rate (smaller nozzle) will increase the cut diameter on each stage. Isokinetic sampling must be maintained, regardless of the nozzle size.

0.5 cfm is normally the upper sample rate (Q_i) limit, however Q_i may be increased up to 0.7 cfm when sampling an extremely fine particulate. Flow rates in the range of 0.5 to 0.7 cfm may result bouncing within the impactor. If these high rates are used, the substrates will require careful inspection to insure that bouncing has not occurred. If bouncing has occurred, the rate must be reduced and the sample re-run. Q_i in excess of 0.7 cfm is never acceptable.

CAE STANDARD OPERATING PROCEDURE

EPA METHOD 22

ISO NUMBER: EPA22-1

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1. Determine the potential emission point(s).
2. Select an observation position which meets the following criteria:
 - Clear unobstructed view of the emission point(s)
 - At least 15 feet away from source
 - Not greater than 440 yards from source
 - Sun is positioned behind observer.
3. Estimate the following ambient conditions:
 - Wind speed
 - Wind direction
 - Sky description (e.g., color, clouds, etc.)
4. Fill in the appropriate company, source, and ambient conditions data on the "Fugitive or Smoke Emission Inspection" data sheet.
5. Sketch the plan view of the source on the data sheet. Indicate the following on the sketch:
 - Observer location relative to source
 - Location of potential and/or actual emission point(s)
 - Sun orientation
 - North directional arrow
6. OPTIONAL - Sketch an elevation drawing to more clearly identify location and orientation of emission point(s).
7. Verify that the source is operating according to predetermined conditions, and will be at these conditions throughout the observation period.
8. Begin observations:
 - Record beginning clock time on data sheet for first series of observations.
 - Start stopwatch No. 1.
 - If and whenever emissions are observed, start a second stopwatch (stopwatch No. 2). Stop this watch whenever emissions cease.
 - If process shuts down or observations cease anytime during the period, stop both watches. Restart watch No. 1 when this break ends.
 - Read no more than 20 continuous minutes at any given period. Take at least 5 minutes break between each 20-minute set of observations.
9. End Observations:
 - Stop Stopwatch No. 1 and, if applicable, Stopwatch No. 2.
 - Record ending time.
10. Record time indicated by Stopwatch No. 1 on the datasheet as the Observation Period.
11. Record time indicated by Stopwatch No. 2 on the datasheet as the Accumulated Emission Duration.



Clean Air Engineering

Standard Operating Procedures

Equipment Calibration

TNMHC Calibration
CEM/FID
SOP I.D.: EPA25A-1
CTPM Section J Page x-1

Effective Date: x/x/96

Drafted By: J. Wright

Approved By: FDC Process Owners

CAE STANDARD OPERATING PROCEDURE

EPA METHOD 25A CALIBRATION OF TOTAL NON-METHANE HYDROCARBON ANALYZERS

ISO NUMBER: EPA25A-1

EFFECTIVE DATE: Pending

AUTHOR: Dept. 68

APPROVED BY: Pending

EPA METHOD 25A CALIBRATION OF TOTAL NON-METHANE HYDROCARBON ANALYZERS

1.0 PURPOSE

This document provides guidelines for selection of calibration materials for total non-methane hydrocarbon analyzers which use a flame ionization detector (FID).

2.0 SCOPE

These guidelines pertain specifically to the J.U.M. Engineering Model 109A dual-channel non-methane hydrocarbon analyzer. However, these guidelines may also generally apply to any similar analyzer which meets the definition of a Total Non-Methane Hydrocarbon Analyzer as described below.

3.0 BASIS

Currently, there are no federal or state regulatory guidelines which apply directly to this topic. The guidelines contained herein are based on adaptation and extension of EPA Method 25A to non-methane hydrocarbon analyzers.

4.0 DEFINITIONS

Total Hydrocarbons (THC) - any and all organic compounds which are detected by a flame ionization detector.

Total Non-Methane Hydrocarbons (TNMHC) - any and all organic compounds other than methane which are detected by a flame ionization detector.

Total Non-Methane Hydrocarbon Analyzer - an analyzer which uses one or more flame ionization detectors and a selective organic filter or converter to determine the separate concentrations of methane (CH_4), THC, and total non-methane hydrocarbons. The total non-methane hydrocarbons are usually determined mathematically as the difference between the measured levels of THC and CH_4 .

THC Channel - the channel on a total non-methane hydrocarbon analyzer which measures THC. The sample delivered to this channel is representative of the actual gas stream being sampled.



Standard Operating Procedures

Equipment Calibration

TNMHC Calibration
CEM/FID
SOP I.D.: EPA25A-1
CTPM Section J Page x-2

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CH₄ Channel - the channel on a total non-methane hydrocarbon analyzer which measures methane only. The sample delivered to this channel is passed through a filter or converter, usually charcoal or a molecular sieve-type material, to remove all organic species except methane prior to analysis.

TNMHC Channel - the calculated channel on a total non-methane hydrocarbon analyzer which indicates the concentration of non-methane THC.

EPA Method 25A - EPA reference method for measuring total hydrocarbons in stationary sources. This method is contained in detail in 40 CFR 60, Appendix A.

Calibration Gas - a known concentration of a gas in an appropriate diluent gas.

EPA Protocol No. 1 Gas - a calibration gas prepared in accordance with the EPA Traceability Protocol for Establishing True Concentrations of Gases Used for Calibrations and Audits of Continuous Source Emission Monitors: Protocol No. 1.

Applicable Span Value - The upper limit of a gas concentration measurement range that is either a) specified for affected sources in the applicable part of the regulations as 1.5 to 2.5 times the facility emission limit, or b) 1.5 to 2.5 times the expected maximum concentration for unregulated sources. The span value is nominally equal to the instrument data acquisition range.

5.0 STANDARD CALIBRATION GUIDELINES

5.1 Calibration Gas Selection

5.1.1 Determination of Applicable Span Value. Separate span values should be determined for both the THC and the CH₄ channels of the analyzer. Following EPA Method 25A specifications, the following guidelines apply:

- THC Channel Span = 1.5 to 2.5 times the facility emission limit expressed in terms of ppm as methane. If no emission limit exists, then the span should equal 1.5 to 2.5 times the expected maximum concentration of the source.
- CH₄ channel Span = 1.5 to 2.5 times the expected maximum CH₄ concentration of the source.

(Note: different span values for the THC and the CH₄ channels will make the TNMHC channel calculation incorrect. In these cases, the TNMHC channel should be ignored.)

5.1.2 Calibration Gas Specifications. All primary calibration gases should be methane mixed in air. An additional propane gas is also needed for quality control purposes. EPA Protocol No. 1 certification of all methane and propane gases is required for compliance tests. Otherwise, gases certified to $\pm 2\%$ accuracy may be acceptable on a project-specific basis.



Clean Air Engineering

Standard Operating Procedures

Equipment Calibration

TNMHC Calibration

CEM/FID

SOP I.D.: EPA25A-1

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Effective Date: x/x/96

Drafted By: J. Wright

Approved By: FDC Process Owners

Depending on the relative magnitudes of the applicable span values for the THC and the CH₄ channels, three to six different methane blends are required. A zero gas of high purity air with less than 0.1 ppm of carbon is also required for each channel.

The required calibration gas materials and concentration ranges are listed in Table 1.



Table 1: Calibration Gas Specifications

Calibration Level	Gas Composition ¹	Concentration
Zero	Zero Air	less than 0.1 ppm of carbon
Low Level	Methane in Air	25-35% of applicable span value (THC & CH ₄)
Mid Level	Methane in Air	45-55% of applicable span value (THC & CH ₄)
High Level	Methane in Air	80-90% of applicable span value (THC & CH ₄)
Converter Check	Propane in Air	10-30% of applicable THC span value

¹ Note: Calibration gases may also be mixed in nitrogen. It is important, however, that all gases used be mixed in the same gas (e.g., air or nitrogen).

5.2 Calibration Requirements

5.2.1 Calibration Error Tests

- 5.2.1.1 Calibrate both the THC and the CH₄ channels of the analyzer at four points using the calibration gases specified in Section 5.1.2.
- 5.2.1.2 Follow the calibration error test procedures of EPA Method 25A, Section 6.4 for both the THC and the CH₄ channels using the above gases.
- 5.2.1.3 The calibration error for each gas must be less than $\pm 5\%$ of the respective calibration gas value before testing may proceed.

5.2.2 Channel Conversion Check

- 5.2.1 Check the non-methane channel conversion by introducing a propane calibration gas into the analyzer. The gas should have a cylinder concentration of approximately 10-30% of the applicable THC span value.
- 5.2.2 During the conversion check, the methane channel should read very near to zero, while the THC and TNMHC channels should both read approximately 2.5-3.5 times the concentration of the propane cylinder value. If these criteria are not met, efforts should be made to identify and rectify the problem before testing proceeds (e.g., adjust converter temperature.)



Clean Air Engineering

Standard Operating Procedures

Equipment Calibration

TNMHC Calibration
CEM/FID
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5.2.3 Data Reporting

5.2.3.1 CH₄ Channel - this channel reports methane in ppmv.

5.2.3.2 THC Channel - this channel reports total hydrocarbons in ppmv as methane equivalent.

5.2.3.3 TNMHC Channel - this channel reports the total non-methane hydrocarbons in ppmv as methane equivalent. It is the direct subtraction of the THC Channel minus the CH₄ Channel. (Once again, the results of this channel are incorrect if the THC and the CH₄ channels spans are different.)

5.2.4 Analyzer Drift

5.2.4.1 Follow the Drift Determination procedures described in Section 7.2 of EPA Method 25A for both the THC and the CH₄ channels of the analyzer.

5.2.4.2 In order to validate a test run, both the THC and the CH₄ channels of the analyzer must meet the zero and calibration drift specifications of $\pm 3\%$ of the applicable span value.

6.0 **ALTERNATIVE CALIBRATION PROCEDURE**

Use this procedure only when situations prevent adherence to the above standard procedure.

This alternative procedure is similar to the standard procedure, except that the THC channel is calibrated with propane gas mixtures instead of methane. The methane channel is still calibrated with methane, and the channel conversion check is still performed. The TNMHC channel output is ignored. Instead, the TNMHC (as methane) must be calculated separately as follows:

$$\text{TNMHC} = 3 \times \text{THC} - \text{CH}_4$$

CAE STANDARD OPERATING PROCEDURE

OPERATING THE CEM TRUCK

ISO NUMBER: CEM-1

AUTHOR: Dept. 66

EFFECTIVE DATE:

APPROVED BY: Pending

This procedure is intended to instruct people in the proper operation of the CEM truck. The new truck has many capabilities that some people may not be aware of. This document will describe the new truck and provide information for its operation.

This vehicle is plated for a Gross Vehicle Weight (GVW) of 21000 lbs. A normal Illinois driver's license only allows drivers to drive up to 16,000 lbs. Therefore, if you do not have a special Illinois driver's license you cannot legally drive this truck. Some out of state driver's licenses do not make a distinction below 28,000 lb so that some out of state licenses may be valid for the truck.

A logbook must be kept by any driver of this vehicle regarding how many hours that can be driven during any one day as well as how many hours total during a week that can be driven.

This truck will be carrying hazardous goods in the form of compressed gas cylinders. As long as there are less than 14 aluminum cylinders in the truck, the truck does not have to be placarded. Be careful when driving through some tunnels as there are restrictions as to what type of compressed cylinders can be carried through especially if there is compressed H₂ on board.

The truck is powered by a Cummins turbo diesel. Make sure only diesel fuel is put into the truck. The fuel tank is located on the right hand side of the truck and is capable of holding 75 gallons. However, do not try and fill the tank up passed the fuel port because there is room built into the tank for expansion. The truck gets about 10 miles per gallon so one tank of gas should last for several hours.

The oil level should be checked at every fill up. This can be accomplished by releasing the two tie down straps on both sides of the hood. Then go to the front of the truck and pull the hood towards you. All necessary checks can be made from the left hand side of the truck. Check the oil, the belts and the anti-freeze.

Before driving the truck several important things need to be checked. First of all, there are stabilizing jacks on the bottom of the rear of the truck. These jacks have locking pins that hold them in place. To lower the jacks, first take the crank that is located in the power cord toolbox and raise the crank as far as it will go. Secondly, pull the pin out of the jack and raise it all the way up, make sure the pin locks the jack into position.

Secondly, check to make sure that all power cords and external lines have been disconnected from the truck. There is a small grounding line that is connected to the frame of the truck make sure this line is disconnected before moving.

Lastly, make sure all toolboxes and doors are closed and locked.

When sitting in the truck you will notice that there is no standard gear shift. The truck has automatic transmission, this transmission is operated electronically. To start the truck, make sure the parking brake is engaged. The transmission does not have a park selection so when the vehicle is parked it is in neutral with the parking brake on. Check all indicators to make sure that everything is running correctly. You will notice that the transmission control box that is to your right should be reading N and N. This means that Neutral has been selected and the vehicle is actually in Neutral. When you want to go forward you would put your foot on the brake and the push the D button on the control box. When you push Drive the computer will automatically shift the transmission for you. This should be the only drive setting that you will ever have to push. After pushing the D button, with your foot on the brake release the parking brake and you are ready to drive. When switching into reverse, stop, put the transmission back into Neutral and then hit Reverse. Make sure someone is guiding you when you are reversing.

When you are driving be careful to watch for low clearances. The truck height is 12 feet. Also, when driving on the highway, watch for weigh stations. States differ as to what weight trucks have to be weighed. Pull over for all weigh stations unless they only require trucks over 22,000 lb GVW.

CEM SYSTEM SET-UP AND OPERATION

ISO NUMBER: CEM-2

EFFECTIVE DATE: Pending

AUTHOR: Dept. 66

APPROVED BY: Pending

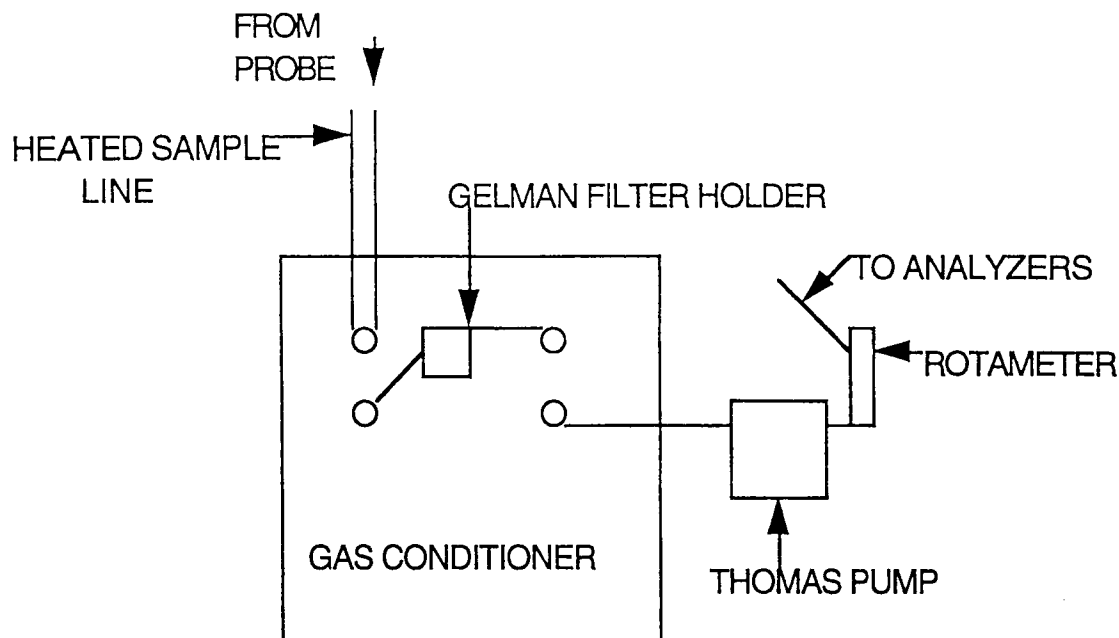
Typically the monitors should be set up with the minimum required length of heated sample line (i.e. As short as possible to reach any points that need to be sampled) There are, of course, exceptions to this rule. Some of which follow.

1. When ambient temperatures are below 40 deg. The gas conditioner should be placed in a warm location to prevent freeze up.
2. When using an FID. A heated sample line must be run to the back of the analyzer to prevent condensation of water and volatile organic compounds. Where possible the HSL should be kept at a temperature approximating that of the stack. Ideally the FID should be run off of its own HSL and any other analyzers set up separately.
3. When space or other restrictions prevent the setup of the gas conditioner. At the test location.

If, in setting up, you are using a ball joint adapter to the probe end then put the ball joint adapts on in the lab and leak check the HSL with a meterbox to insure that all flare and swage fittings are well tightened and leak free. Think ahead and use an angle iron or pipe to secure the ball joint adapter to the probe in a way that does not allow the vacuum. Grease /screw clamp fitting to work itself loose during point or port moves. At the back side of the ball joint adapter (or if you have a monitor probe with a 1/4" liner t directly to the probe) "t" off with a straight t or a 3 way valve. In either case the HSL should exit the "t" from the bottom with a calibration line entering from the leg opposite the probe. This is the only way to properly set up a three way valve. If you are using a straight "t" you will have to be sure that the calibration line is leak free during sampling so that no ambient air is pulled through the system along with the gas sample.

It is probably a good idea to take a Thomas pump to the location at this point and leak check the probe/HSL assembly.

Once this is complete tie the heated line into the gas conditioner. If it is a dual pass conditioner use both passes to insure complete drying of the sample stream. (see drawing)



The via MAK ii is rated for four (4) lpm per pass so if you have only a single pass conditioner 4 lpm is the absolute maximum sample flow rate that you should use. If using a dual path I wouldn't go any higher than 5 lpm to insure a dry sample gas stream.

The Gelman may be placed at the end of the heated sample line at the testers discretion. Ideally it should be placed at the pressure side of the Thomas pump but often in high particulate concentrations the gas conditioner will gum up with mud as a result of long term usage. Be sure that Gelman filter holder has all "o" and thrust rings intact. It may also be leak checked prior to set up to assure sample integrity.

Be sure to place the rotameter at the outlet side of the Thomas pump. This will insure positive pressure operation of the pump and any leakage will be outleakage instead of inleakage. The pump may not want to start turning due to backpressure in this configuration so you may either have to open the rotameter as much as possible or leave the outlet fitting loose until the pump begins turning. At this point be sure to either tighten the fitting or reduce the rotameter flow to test settings (if you over-close the rotameter you will feel and hear the pump begin to "bog down").

ANALYZER OVERVIEWS

At this point in time you should be ready to run Teflon line from the gas conditioner/sample pump setup to the analyzers. How to best accomplish this is dependent on the type of analyzers you are using. A brief overview of the most commonly analyzers and how they operate follows.

NOX (TECO 10A,10S)

The TECO model 10 is a multi-range chemiluminescent analyzer. It pulls its own sample at approx. 800cc/min using the high vacuum and bypass pump flows limited by critical orifices (the capillaries). It has no sample outlet tap and must be isolated in its setup it is also a destructive analyzer and may not be used in line with other analyzers. It is a two channel (NO, NOx) and should always, unless specified otherwise, be used in the NOx mode. There are also two output voltage jack on the back panel (0-10v, and 0-100mv) **always** use the 0-10v output. It does not matter that this may be listed on the no output in the back. **Always** use the 0-10v lead. The mv output will result in a dirty signal using the chart recorders and will not work at all using Labtech notebook. To set the 0-10v scale at any range turn the analyzer to zero (using the scale knob on the front panel) open the top drawer and at the front, mounted on a divider, are two output leads. Using a multimeter, hook into the leads and get a voltage (dc) reading. Adjust the output to zero using the screw set on the front panel of the analyzer (just above the range knob). (use this time to also zero the meter on the front panel of the analyzer by turning the set screw on the bottom panel of the meter face.) Leave the multimeter connected to the leads and turn the range knob to full scale. At this point your multimeter should be reading approx. 10v dc. Inside the top drawer on the back of the range knob there is a small white set screw. Use this screw to adjust to output voltage to 10 volts. (again set the analog meter on the front panel to full scale using the meter cal knob inside the top drawer on the opposite side of the divider from where the output leads are located.) The NOx analyzer should now be ready electrically for sampling. Turn on the pumps (vacuum/bypass) and check the bypass flow (2scfh) and chamber vacuum (28-30"hg dependent on altitude 24" is cool in Denver) be sure that the NOx converter is set at a temp of 650 deg c and is heating. (the thermostat light will flash on and off and it can be checked by touch by opening the bottom drawer and feeling for warmth. If all these criteria are met the analyzer should be ready for sampling.

TECO 10A, 10S OVERVIEW

RANGE(S) 0-10,25,100,250,1000,2500,10,000

OUTPUT VOLTAGE HIGH 0---10VDC; LOW 0--100mv DC

SAMPLE FLOW (SELF LIMITED AT APPROX. 750-1000cc/min.)

PRINCIPAL OF OPERATION:

Chemiluminescent/destructive cannot be manifolded in series with other analyzers "t" off the sample stream

SO2 (WESTERN RESEARCH 721 AT2)

This is a dual range (0-500,0-5000ppm) non-destructive ultra violet analyzer. It has both sample inlet and outlet ports and may be used in series with other analyzers. It does not have an internal pump and utilizes sample flow from the Thomas pump integral to the sampling system. The flow range for this analyzer is typically 1-5 lpm with around 2 being ideal(2 lpm adequately meets flow requirements for all the non-destructive analyzers we use so if they are manifolded together they should all operate properly using this flow and will leave a measurable bypass stream remaining to allow for flow adjustments without overwhelming the gas conditioner with the need to increase total sample flow.). This analyzer will also "overrange", that is, will read off scale especially if you exceed 500 ppm. While it will overrange on the led at the front of the instrument it will not overrange on the voltage output and will be stuck at full scale. You will have to change ranges on the back of the analyzer for chart or LTNB ratings if this occurs. There are two voltage range outputs for each scale (high and low). Check the manual as to which leads on the back of the analyzer are for which. (I believe that for a 0-1volt output you should be using jacks 3 and 4 on the board on the upper right hand corner on the back panel) this can easily be checked using a multimeter and placing the analyzer in zero and span modes while checking output voltages. The only special considerations there are with these analyzers are warm up time and variable readings due to non-equilibrium of the sample passing through the gas conditioner. This latter problem will cause drift (possibly excessive) until the pH of the sample gas reaches equilibrium with the pH of the condensate in the gas conditioner. Usually this alleviates itself after pulling stack gas for a half hour to an hour.

721 AT2 OVERVIEW

RANGE(S) HIGH 0-5000ppm; LOW 0-500ppm

VOLTAGE OUTPUTS 2 PER RANGE

HIGH 0---1VOLT DC; LOW 0--20 mvDC

SAMPLE FLOW 1----5LPM

PRINCIPAL OF OPERATION

Non-destructive ultra-violet; may be manifolded together with other analyzers

CO (TECO 48)

The TECO 48 is a multi ranged non-destructive analyzer using gas filter correlation to analyze for co. This is a really simple analyzer to use in the field and is usually trouble free if it is set up correctly. If you do have any trouble with it is usually something that merits replacing it with another analyzer. This analyzer has its own internal pump to regulate sample flow. This coupled with the fact that it is non-destructive makes it ideal for manifolding in series with other non-destructive. Analyzers. The internal flow rate is usually set at around 2 lpm so that it works great with other analyzers with 1-5 lpm flow requirements. Ranges are controlled by thumbwheels on the front panel. Two ranges can be used at the same time as there are two outputs at the back of the analyzer. This analyzer is different from others in that it takes a running average of the gas sample as

opposed to an instantaneous analog readout of the sample. For nearly all of our applications you should use the time thumbwheel setting zero. This gives you a running average updated every 10 seconds. Upon setup be sure that the output (a or b on the back panel) jives with the thumbwheel setup (a left wheel, b right wheel) both the a and b outputs are 0---10volts dc. These may be checked using the z/fs button of the front of the analyzer. To get back into run mode use the run button on the front of the analyzer. This returns to run mode from any of the diagnostic checks that can be made from the front panel. Please note that the run mode also activates the span and zero solenoids at the back of the analyzer. For CEM purposes cal gases can be plumbed directly into the TECO 48 from the regulators to span and zero ports at the back of the analyzer. We do not use any of these ports for our calibrations. For our purposes, almost exclusively, cal gases must be run through the entire sample system i.e. From the sample probe. There are two common problems with the teco48. The first is evident immediately upon powering up the analyzer. Symptoms are usually that the led is dim and the pushbutton and thumbwheel controls do not respond when the are used. This is usually due to the IC boards immediately behind the control panels coming loose during shipment. Remove the top of the analyzer and try to reseal the IC boards by jiggling them gently into the sockets. If they do reseal the led should brighten and the pushbuttons and thumbwheels will function properly. The second problems is of a more minor but irritating nature. The analyzer will over-pressurize if the sample flow becomes too great. This will result in an error on the led and put the output voltage to 10 volts. To eliminate the problem the monitor must be plumbed with a bypass and allowed to draw sample at its own rate. At times, the sample pressure will go low and result in an error of the same effects. This occurs when sample flow through the system drops to a point that the internal pump to the analyzer is pulling a net vacuum. It is important to monitor the bypass flow so that any problems with pluggage or lines popping off will be noticed as quickly as possible and corrected.

TECO 48 OVERVIEW

RANGE: 0- ; 0--1000ppm DUAL CHANNEL WILL OVER RANGE TO 10,000ppm BUT ONLY ON LED NOT OUTPUTS. REMAINS LINEAR +/-2% WHEN OVER RANGING.

OUTPUT: 1--10VOLTS DC

SAMPLE FLOW: 1-5LPM REGULATED BY INTERNAL PUMP

PRINICPAL OF OPERATION:

NDIR, gas filter correlation can be manifolded in series with other analyzers will pump enough flow to provide sufficient sample to other analyzers in series.

HORIBA/FUJI CO

Operate with NDIR principals they do not have internal pumps and are typically dual range i.e. 0--500 and 0--1500 ppm. Voltage output is usually 0--1 volt dc unless they have been modified. Can be used in series with other analyzers and works well but they are not as nice as the TECO 48. Simple range switch and zero and span adjustments on the front panel.

HORIBA/FUJI OVERVIEW

RANGE: VARIABLE BUT TYPICALLY 0--500; 0--1500ppm

OUTPUT: 0--1VOLT DC

SAMPLE FLOW: 1-5 LPM

PRINCIPAL OF OPERATION:

NDIR can be manifolded in series with other non-des. Analyzers

HORIBA/FUJI CO2

Operate the same as the co monitors but for co2 dual range typically 0-15%; 0-25%, voltage output 0--1volt dc. Can be put in series with other non-des. Analyzers. Front panel the same as the co analyzer with a range switch and zero/span adjustment.

HORIBA/FUJI CO2 OVERVIEW

RANGE: DUAL 0--15%,0--25%

OUTPUT: 0--1VOLT DC

SAMPLE FLOW: 1--5LPM

PRINCIPAL OF OPERATION:

NDIR can be used in series with other NDIR analyzers

TELEDYNE O2 (320,326)

The Teledyne 320 and 326 are electrochemical oxygen analyzers. Oxygen reacts chemically in the sample cell and sends current to the output to give an O₂ reading. The two models operate on the same principal but have different applications. The 320 is a good diagnostic tool but inadequate for continuous operation. It operates on a battery/charger system and is not made for CEM applications its output is only 0--100mv dc. It is good for times when the analog meter values can be handwritten i.e. Traverses with a m-17 train or sample pump. Leak detection etc. Both the 320 and 326 are multi ranged instruments (0-5%,0-10%,0-25%) and each uses a fuel cell in operation although the cells are **not** interchangeable. The 320 uses a b-1 fuel cell and the 326 and a3 fuel cell. These are Teledyne items and each analyzer is set up for one or the other. They can be altered and other cells used by altering the internal electronics of the analyzer. Something that we do not do. When the model 320 is not in use it should be plugged into a 120 v power source and allowed to recharge.

Unlike the 320 the 326 is a good tool for continuous long term applications. It utilizes a longer lived fuel cell as well as providing a 0--1 volt dc output that can be used with charts or LTNB. It is provided with inlet and outlet taps but is a destructive reaction so **if the Teledyne 326 is manifolded with other analyzers it must come last!** It is also good practice to have the o2 analyzer last in the series as it is the best diagnostic tool for finding leaks. If you o2 will not zero while nitrogen or another constituent gas is being run then chances are that there is a leak in your sampling system. The 326 has a built in rotameter reading in scfh so ideally 2 scfh should provide adequate sample (1lpm). The cell will overpressurize and when this occurs erroneous results will be generated (usually high). The cell holder will also pop open due to pressure and should be checked periodically to be sure that it is well seated. (do this by opening the front hinged panel and pressing the holder into the socket. To replace cells, open analyzer in the same manner and remove cell holder from the socket unscrew the cap and lift out and replace the sample cell being sure to place the brass side of the cell toward the elec. Contacts in the cell holder. Drawbacks of the analyzer are that the cell must be replaced. (while this is not time consuming if it happens in the middle of a run the new cell must be calibrated and you end up losing sample time and may be forced to restart a run. In the same vein it seems that new cells must be "conditioned" before they begin to settle down and maintain steady readings. New cells tend to drift. The other major drawback of the Teledynes is that the fuel cell is very sensitive to water. Any entrained water in the sample line that reaches the cell will have detrimental effects. These are usually readily noticeable and typically the meter begins to bounce erratically all over the scale. Sometimes the cell can be removed and the holder blown out to easily resolve the problem and other times the cell must be allowed to dry overnight to begin to read properly. **It is vital to have at least one preferably two extra sample cells when going into the field with Teledynes.** In order to prolong cell life on both models when not in use the cell holder should be closed to ambient air by either capping (model 320) or closing off the 1/4" sample ports (model 326) in all instances the analyzers should be **turned off** when not in use. There is no warm up time for these analyzers so it will not cost any time to turn them off and on as needed. For the model 326 this is the power switch on the front panel, for the 320 be sure that the pump switch (either toggle, or push button switch on top right of the analyzer) is off and leave it plugged in to charge overnight.

TELEDYNE 320/326 OVERVIEW

RANGE: 0-5%,0-10%, 0-25% LINEARITY BETWEEN RANGES VARIES

OUTPUT: MODEL 326 0--1VOLT DC, MODEL 320 0-100 mv DC

SAMPLE FLOW: 2.0 SCFH (1-2LPM)

PRINCIPAL OF OPERATION:

Electrochemical fuel cell, destructive cannot be used in series unless it is last.

SERVOMEX O2

The Servomex o2 analyzer uses a non-destructive paramagnetic operation. It is a dual range analyzer (0--25%,0--100%) with output voltage of 0--1 volt dc. Again as a non-des. Analyzer it can be manifolded with other analyzers but should be last in the series to assure sample integrity. This analyzer requires some warm up time and the instrument status light on the front panel will begin blinking when it is ready for operation. This is a simple analyzer to use in that it is a no fault type. The only thing to do is be sure that there is sufficient flow (a flow status light is just below the inst. Status light and will blink if flow is low). If there appears to be flow to the analyzer and still the low flow light is blinking then check the in line filter at the back of the analyzer for pluggage. There is a type of ball and float system in the back if you open the case but I would call rentals and have some one talk you through this repair if it becomes necessary. The only other things of interest or adjustments that need to be made are zero and span pots located under the flap on the front panel. Of course, adjust these to the appropriate cal gas values and begin sampling.

SERVOMEX O2 OVERVIEW

RANGE: (DUAL) 0--25%,0--100% NOT ALL HAVE THE 100% RANGE

OUTPUT: 0-1 VOLT DC

SAMPLE FLOW: 1-5 LPM

PRINCIPAL OF OPERATION:

PARAMAGNETIC, NON-DESTRUCTIVE

HYDROCARBONS (JUM/RATFISH FIDS)

FIDS analyze for total hydrocarbons using flame ionization. They are unique to the other monitors mentioned in that they analyze on a wet basis and, as mentioned earlier, must be plumbed with a heated sample line directly to the back of the analyzer. They contain integral pumps and draw their own sample at the proper flow rates. FIDS are destructive and although the JUM has a sample outlet port they cannot be used in series with other analyzers. The heated sample line may be "teed" at the back of the FID and a portion of the sample gas sent through a gas conditioner to the other analyzers, but ideally these monitors should be set up separately.

Another difference in the FIDS are that they require a fuel gas to operated. For the JUM, this is hydrogen only. It supplies its own combustion air from ambient. The Ratfish, on the other hand, uses hydrogen and zero air under pressure to operate. Both have a warm up time and must be ignited. While this may not always be easy, if you follow the instructions in the manual, with persistence, they will light.

These are multi-ranged instruments usually from 0--10 to 0--100,000 ppm. The output voltage is 1--10volts dc. **They cannot be used with LTNB** because of the nature of their wiring they are not compatible due to common/ground

configuration. So isolate them with a chart recorder. They are not linear from range to range and if they should over range the analyzer should be calibrated with higher range cal gases. No special external pumps etc. (with the exception of the fuel gases) are required. Jums are now more common with rentals so it would be rare to have to use a Ratfish. Jums are a little easier to use and seem to be more reliable for field work.

JUM/RATFISH OVER VIEW

RANGE: MULTI (0-10,100,1,000,10,000,100,000)

CAN BE ADJUSTED INTERNALLY UP OR DOWN (RENTALS ONLY)

OUTPUT VOLTAGE: 0---10VOLTS DC

INCOMPATABLE WITH LABTECH NOTEBOOK

SAMPLE FLOW: INTEGRAL PUMP CONTROLS FLOW INTERNALLY

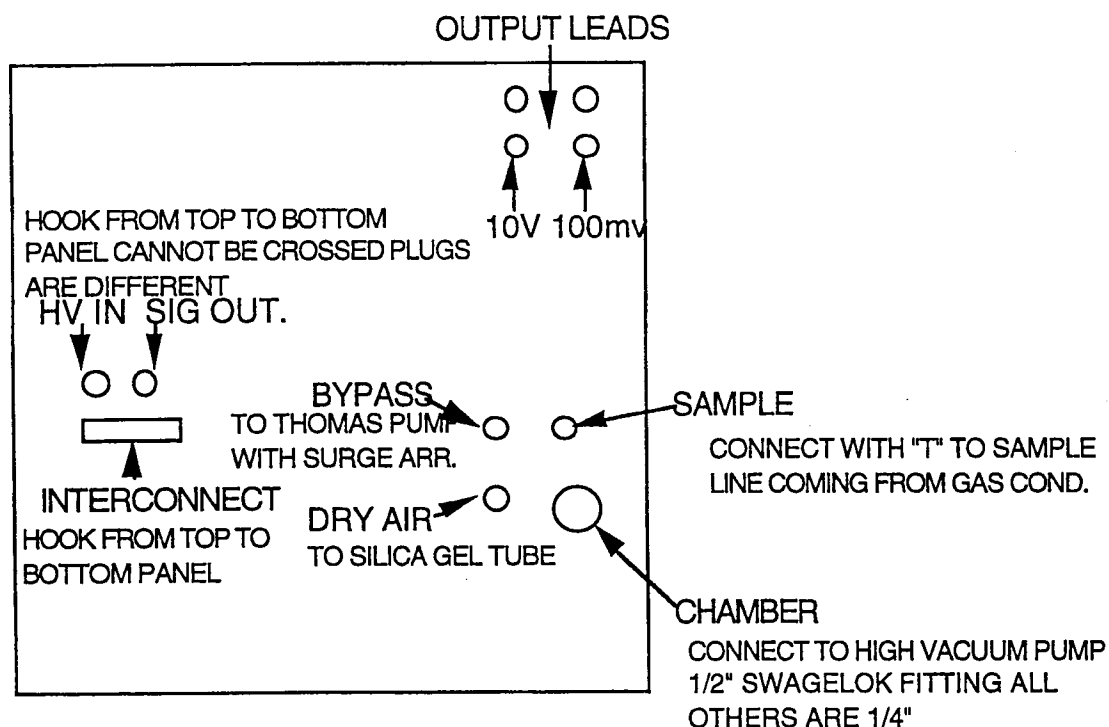
PRICIPAL OF OPERATION: flame ionization detection of a wet sample.

Destructive method using fuel gas.

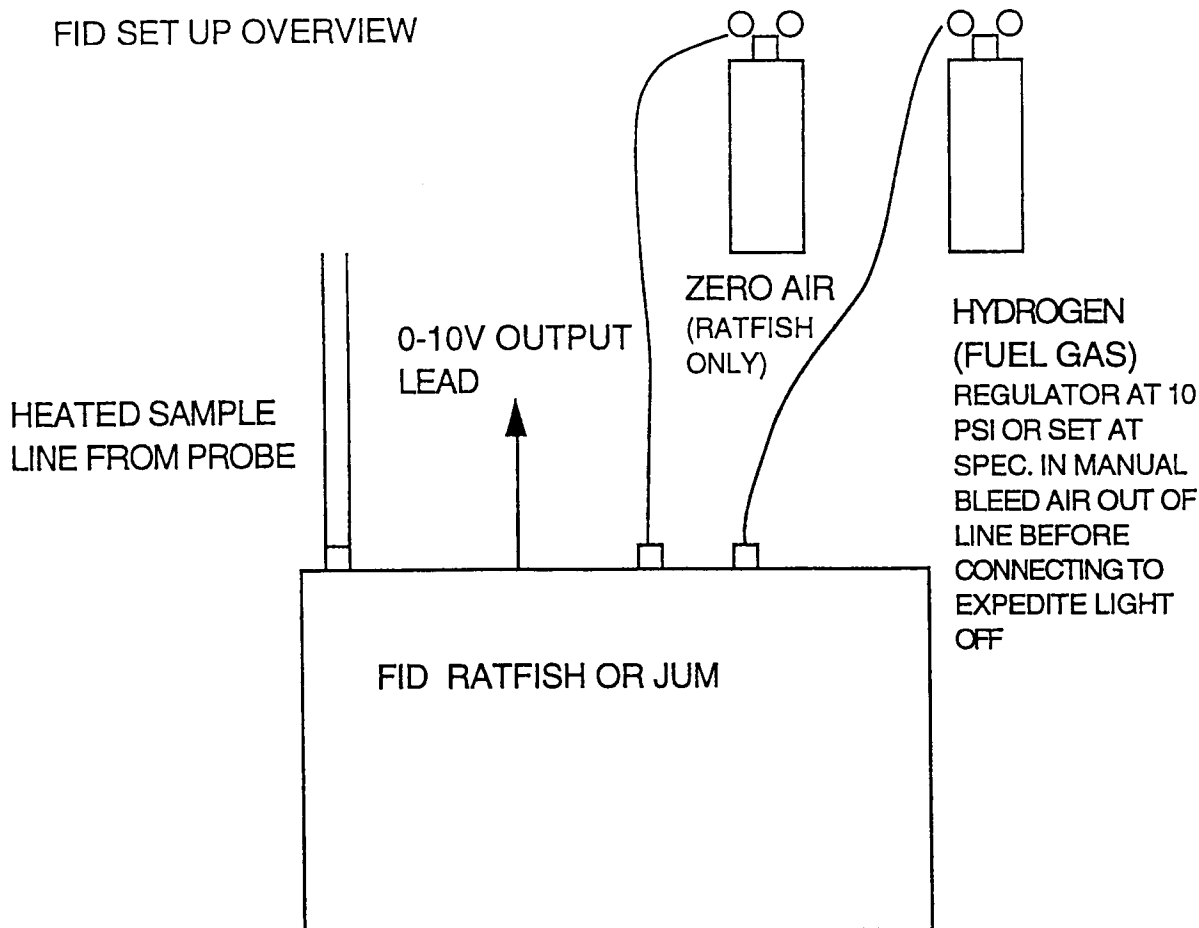
ANALYZER SET UP

After completing the gas conditioner/heated sample line/probe set up, you're ready to plumb the monitors typically all that is needed is several short lengths of Teflon tubing, "tees", rotameters, and a couple of small (1/2", 9/16") wrenches. There are drawings provided for some typical set up schemes below as well as drawings for analyzers that are not self-contained (NOx, FID)

NOX MONITOR BACK PANEL DETAIL



This panel set up is straightforward all that must be kept in mind is the following. A surge tube (1/2" Diameter pvc pipe) must be put in line between the Thomas pump and the analyzer or else the "pulse" of the Thomas pump will affect the bypass flow rates. The high vacuum pump that is hooked into the chamber needs to have an ozone scrubber (pvc tube filled with charcoal) mounted on the pump. This prevents degradation of the vacuum oil from ozone in the chamber. Be sure that the silica gel in the dry air tube is in good shape. If it is letting water pass through (pink) your readings will become very erratic. Hook up the electrical lead to the 0-10v output jacks (these may be marked no and NOx but it does not matter, typically the 0-10v lead is the no output.).



The FID set up is an easy one. There are a couple of hints to make life easier. First, be sure to bleed hydrogen to the end of the Teflon tubing so that it does not have to travel the length of the hose before reaching the analyzer. This will result in a much quicker light off. The analyzer does not use much hydrogen so it may take minutes for it to reach the analyzer through the tubing. Second, use a butane lighter or a beach ball with cal gas and introduce raw gas at the sample port at the back of the monitor. Response to the hydrocarbons should be immediate and off scale. This is just a simple way to be sure that the analyzer is lit and that it is responding to hydrocarbons.

BASIC SET UP DIAGRAMS

FID WITH NOX AND NDIR ANALYZERS WITH INTERNAL PUMP
SEE FIGURE 1

NOX, AND NDIR ANALYZERS WITH NO INTERNAL PUMP
SEE FIGURE 2

NDIR (PUMPED) ANALYZERS
SEE FIGURE 3

SAMPLE SYSTEM LEAK CHECK PROCEDURE

If the procedures for set up that were outlined earlier have been followed, the probe and heated line system should be leak free. As a QA check you should at this point leak check the system to the Thomas pump at the gas conditioner outlet. If you are using a three way valve at the probe end, turn it to the stack side and plug the end of the probe. The rotameter at the Thomas pump outlet should go to zero. There may, however, be some pulse action from the pump evident. If you are using a three way "tee" at the probe end be sure that it is capped or tightly attached to a cal gas regulator with the valve closed to prevent ambient being pulled through the cal line to the probe and conduct the leak check in the identical manner as with the three way valve. All lines from the Thomas pump outlet to the analyzers should be under positive pressure if the setup has been done properly, therefore all leakage should be outleakage and unless significant enough to cause a loss of sample flow to the analyzers can be ignored.

CALIBRATION ERROR DETERMINATION

To determine calibration error perform the following checks (they are outlined in EPA m6c section 3) at a point immediately upstream of the analyzers introduce the cal gases (zero<.25% of span, mid 40-60% of span, and high 80-100% of span). Run all the gases through the analyzers one at a time and set the analyzers to the tagged tank values. Repeat this procedure making no adjustments to the system, other than necessary to maintain sample flow, and record the results either on the chart recorder, LTNB, or in the figure provided. If any reading is greater than 2% of the span value corrective action must be taken and the cal error check
REPEATED.

EXAMPLE:

You have a NOx monitor setup and ready to go at 0-1000 ppm range. You run your zero gas ("0" n2) and get a zero response, you then run the span gas (850ppm) and get a response of 846ppm (this is .4% of span (1000ppm)) when you run the mid range gas (500ppm) you get an analyzer response of 479 ppm (2.1% of span) this is unacceptable calibration error. And you must take

corrective action. In this case all that is necessary is to increase the span reading to a response of 860ppm (1% of span and the mid range response should rise accordingly and come within the limits specified (2% of span value).

There will be times when the analyzer linearity (the reason for a cal error check) is way out of control due to analyzer problems. These are typically beyond our capability to repair and the analyzer should be replaced. Call rentals first as they may be able to offer a quick and easy solution to the problem.

SYSTEM BIAS CHECK PROCEDURES

The bias check is performed to assure the fact that the sample system (hsl,gas cond.,pump) is not creating a change (bias) in the sample integrity. The details of the bias check are outlined in EPA m6c section 4. The basics of the bias check are as follows. Procedures are the same as the cal. Error check except that the cal gas is introduced at the valve or "tee" at the end of the sampling probe and that the cal gas used should closely approximate the stack gas concentration (mid or full scale whichever is closer). The gas then passes through the entire system to assure that there is no error in concentration produced by the sample system. **(if the system has leak checked properly bias should be minimal!!!!!!)**

Determine the system bias by the following equation

$$\frac{\text{ANALYZER CAL RESP. (CAL ERROR)} - \text{SYSTEM CAL RESP. (BIAS CHECK)}}{\text{SPAN}} \times 100 = \text{BIAS}$$

Record the results of the bias check using chart recorders LTNB or by hand using the chart that is printed later in the appendix. If the bias check yields a result of less than 5% of span the system is ok and sampling may begin.

SYSTEM RESPONSE TIME DETERMINATION

System response may be determined simultaneously with the bias check by recording the time it takes to achieve a stable response to the zero and full scale calibration gases. All that is required is that the duration to stable response is timed and recorded. Any sampling done must be test time plus twice the system response time. I.e. A 21 min. So2 cert. Run would be 23 min. Long if the system response time was 1 minute.

CALIBRATION AND ZERO DRIFT DETERMINATION

Cal and zero drift determinations are done at the beginning and end of each test run. Procedure is identical to the bias check, the bias check may be used for the initial cal. But to assure linearity etc. It is recommended to run all cal gases at the onset of testing and then use the gas closest to stack concentrations for all calibrations thereafter. The sole difference between zero and cal. Drift and the bias check is that the limit for both the cal. And zero drift is three (3) percent of span and not five. Use the same equation listed previously for bias determination to calculate drift. If the zero and/or cal. Drift exceeds 3% the run must be repeated. If the cal. And/or zero drift exceeds 5% then the cal. Error and bias check must be done over and the test rerun.

HELPFUL HINTS

Although it does not specify in method 6c what order to run the cal gases in it is most sensible to run zero, full scale, and then mid range last. This sets the endpoints of the calibration curve and the linearity. Keep in mind that all instrumental methods refer to the procedures outlined in method 6c. With some exceptions. (example: co cal gases are to be 60 and 30% of span respectively and are not required to be protocol 1). Refer to the method that you are to be using for any specific details that may deviate from EPA 6c.

Method specific details will be outlined in the appendix

APPENDIX

METHOD SPECIFIC REQUIREMENTS

EPA METHOD 3A (O₂/CO₂ INST)

CAL GASES: SAME AS EPA 6C

ZERO (<.25% OF SPAN) O₂ <.0625%; CO₂<.04%
MID (40-60% OF SPAN) O₂ 10-15%; CO₂ 6-9%
HIGH (80-100% OF SPAN) O₂ 20-25%; CO₂ 12-15%
EPA PROTOCOL 1 GASES

ALL OTHER METHOD PARTICULARS ARE THE SAME AS OUTLINED IN EPA METHOD 6C.

EPA METHOD 6C (SO₂ INST.)

CAL GASES:

ZERO (<.25% OF SPAN) O₂ <.0625%; CO₂<.04%
MID (40-60% OF SPAN) O₂ 10-15%; CO₂ 6-9%
HIGH (80-100% OF SPAN) O₂ 20-25%; CO₂ 12-15%
EPA PROTOCOL 1 GASES

EPA METHOD 7E (NO_x INST.)

CAL GASES:

ZERO (<.25% OF SPAN) O₂ <.0625%; CO₂<.04%
MID (40-60% OF SPAN) O₂ 10-15%; CO₂ 6-9%
HIGH (80-100% OF SPAN) O₂ 20-25%; CO₂ 12-15%
EPA PROTOCOL 1 GASES

EPA METHOD 10 (CO INST.)

CAL GASES:

ZERO NITROGEN
LOW 30% OF SPAN VALUE
MID 60% OF SPAN VALUE
HIGH SPAN VALUE (NOT TO EXCEED 1.5 TIMES APPLICABLE SOURCE
STANDARD EMISSION RATE)
MAN. CERTIFIED +/- 2% OF TAGGED VALUES. EPA PROTOCOL 1
REQUIRED FOR BIF PERMITTED SOURCES

EPA METHOD 25A (THC INST. BY FID)

CAL GASES:

ZERO OR PURIFIED AIR

LOW 25-35% OF SPAN VALUE

MID 45-55% OF SPAN VALUE

HIGH 80-90% OF SPAN VALUE

SPAN IS NOT TO EXCEED 1.5 TO 2.5 TIMES THE SOURCE PERMITTED
LIMIT OR EXPECTED CONCENTRATIONS.

GASES TO BE EPA PROTOCOL 1 OR IN CASES OF HIGH RANGE (1-10% BY
VOLUME) DILUTION OR ALTERNATIVES MUST BE APPROVED BY THE
ADMINISTRATOR.

GASES MAY BE PROPANE IN AIR OR NITROGEN OR AN ORGANIC
COMPOUND OTHER THAN PROPANE PROVIDED THAT APPROPRIATE
ADJUSTMENTS ARE MADE TO THE MEASUREMENT SYSTEM.

(I.E. METHANE, ETHANE, BUTANE) CORRECTIONS ARE AS FOLLOWS

METHANE ---1

ETHANE-----2

PROPANE---3

BUTANE-----4

ALL HYDROCARBON EMISSIONS WILL BE REPORTED AS THE
CONSTITUENT IN THE CAL GAS. SO 1000 PPM THC AS PROPANE EQUALS
3000 PPM THC AS METHANE ETC. (SEE EQUATION 25A-1)

CEM QA/QC PROCEDURES AND REQUIREMENTS

ISO NUMBER: CEM-3

EFFECTIVE DATE: Pending

AUTHOR: Dept. 66

APPROVED BY: Pending

Introduction:

This document contains the standard operating procedures for running all CEM work. All information is directly out of 40 CFR Part 60 and Part 75 and the BIF regulations. The monitors referenced here are the usual effluents encountered during testing. They include; NO_x, SO₂, CO, O₂, CO₂ and THC. Any other effluent, such as H₂S and HCl, will not be addressed here.

Scope:

All the effluents that CAE usually encounters have their own specific method detailing how the monitors for that effluent should be setup and run. The regulations are very precise as to exactly how the monitors should be operated and calibrated. Below is a list of these reference methods:

40 CFR Part 60**Method:****Effluent:**

3A	O ₂ and CO ₂
6C	SO ₂
7E	NO _x
10	CO
25A	THC

Most of the procedures reference method 6C for the bulk of their information. All of these methods should be read completely before doing any CEM work.

When performing a monitor certification there are procedures detailing how to perform a RATA (Relative Accuracy Test Audit). These methods are in Appendix B in

the 40 CFR. Performance Specification 2 (PS 2) contains the bulk of the information for performing a monitor certification. NO_x and SO₂ are contained in PS 2. O₂ and CO₂ procedures are contained in PS 3. CO procedures are contained in PS 4. PS 3 and PS 4 reference PS 2 extensively. The new Part 75 regulations have different requirements for performing a monitor certification.

The BIF regulations have still another procedure for performing a monitor certification. These regulations are in section 2.1 for O₂ and CO and in section 2.2 for THC.

Methods:

Method 6C:

This method is for the determination of SO₂. Method 6C will be listed first because Methods 3A, 7E and 10 reference this method extensively. For all of these methods it is assumed that a sample is withdrawn from the stack, conveyed to a moisture removal system and then to a series of pumps and gas manifolds to direct the gas to the correct analyzer at the correct flow rate.

The span of the instrument should be set so the average pollutant concentration equivalent to the emission standard is not less than 30% of the span (i.e. the span is less than 10/3 times the emission limit). The span is defined as the upper limit of the gas concentration measurement range displayed on the data recorder. Therefore, the span is not necessarily the range set on the monitor but at what range the data acquisition system is set. There is also the requirement that the minimum detectable limit should be less than 2% of the span. This requirement is usually not a problem with the analyzers that CAE uses. There are three different calibrations that need to be done in order to run these monitors.

1) Calibration Error

An analyzer Calibration Error check is the first calibration that needs to be done. This check flows the calibration gas directly into the back of the analyzer, bypassing all the sample lines, pumps and gas conditioners (This Calibration Error (CE) check is different than the CE check a plant is required to perform. A plant's CE check has to introduce the gas at the probe not at the back of the analyzer). Three different calibration

gases are needed for this procedure: A zero gas which should be less than 0.25% of span; a mid-level gas which should be between 40 and 60% of span; and a high-level gas which should be between 80 and 100% of span. The upscale calibration gases must be EPA Protocol 1 gases. These gases can be flowed in any order and any adjustments to the analyzers can be made at this time. A good procedure is to run the zero gas to the analyzer and adjust for the zero and then to run the high-level gas and adjust the analyzer. To check the linearity, the mid-level gas should be flowed without any adjustments being made. Once this procedure is completed, all the gases should be re-run to the analyzer and at this point no further adjustments can be made.

The method states that for the Calibration Error the difference between the calibration gas and the analyzer response cannot be greater than plus or minus 2% of the span. For example, if the span is 200 ppm the analyzer response cannot differ from the calibration gas by more than plus or minus 4 ppm. This applies for all three of the calibration gases. Once the Calibration Error is complete no adjustments to the system can be made at any time without redoing the Calibration Error, except for regulating the flow.

2) Bias Check

Once the Calibration Error is complete, a Sampling System Bias Check needs to be performed. The Bias Check involves running the calibration gas up to the probe and back down through the normal sampling line and through all gas conditioners, pumps, manifolds and flow meters. For this check, only two gases need to be run. The zero gas must be run and either the mid-level or the high-level gas depending on which gas is closer to the actual gas concentration in the stack. The requirements for the Bias Check are less restrictive than for the Calibration Error. For the Bias Check, the difference between the analyzer response for the Bias Check and Calibration Error response (for that gas) divided by the span, multiplied by 100, cannot be greater than plus or minus 5%. This 5% holds for both the zero gas and the upscale calibration gas.

3) Post Bias Check and Calibration Drift

Once the initial Bias Check is completed, actual runs can begin. During the run, the sample rate coming down from the stack must be maintained within 10%. The run times can vary depending on the testing being done and what kind of data the client

requires. Usually a 21 minute run is performed for RATA testing and 60 minute runs are performed for compliance testing. Once the run is complete, another Bias Check needs to be performed. The same procedures will be used as the initial Bias Check. The Bias Check must still meet the 5% criteria. For the run to be valid, both the initial and final Bias Checks must be less than 5% and the Calibration Drift between the Bias Checks must be less than 3%. This holds true for both the zero gas and the upscale calibration gas.

The Calibration Drift is determined by taking the difference between the final Bias Check and the initial Bias Check, dividing by the span and multiplying by 100. If either the Bias Check before or after a run, or the Calibration Drift over the course of the run is greater than the acceptable limits, the run is invalid and the Calibration Error and Bias Checks need to be repeated. The average ppm concentration needs to be corrected for the pre and post test Bias Checks. Equation 6C-1 in Method 6C needs to be applied to the raw average over the course of the run.

The response time for all the analyzers should be noted for each location. The response time is defined as the amount of time required for the measurement system to display 95% of a step change in gas concentration on the data recorder. In other words, it is the amount of time from when the zero calibration gas is switched to the high calibration gas, until the analyzer reads 95% of the stable high calibration gas value and vice versa. The greater of these two times is the response time. There is a different procedure in the BIF Regulations and also in Method 20.

For this method, a heated sample probe and heated sample line must be used on all surfaces in contact with the gas until the gas conditioner. SO₂ is readily scrubbed by H₂O so that the gas must be kept from coming into direct contact with any H₂O in the sampling system. There is also a requirement for an interference check to be performed at the initial field test of a new analyzer on each particular source category. This interference check is conducted by running a regular Method 6 train at the sample bypass for three tests using midget impingers. The results must be within 7% for each run. This check must be performed on each source category that is going to be tested. This data must be reported on all jobs where the analyzer is used.

Method 3A:

Method 3A is for the determination of O₂ and CO₂. This method follows the exact same procedures as Method 6C except for the following differences.

The average concentration for determining the span of the analyzer for this method should not be less than 20% instead of the 30% of span specified in Method 6C. Non-protocol gases can be used for O₂ and CO₂ analyzers as long as an Orsat analysis is done on the tank. The Orsat analysis must be within 5% of the calibration gas or 0.2% by volume. If the concentrations are less than 5% than the tank value should be used. O₂ and CO₂ are not scrubbed by H₂O so an unheated sample line can be used. An interference response must be performed according to Method 20.

Method 7E:

Method 7E is for the determination of NO_x. This method follows the exact same procedures as Method 6C except for the following differences.

The NO_x method calls for the gas sample to be conveyed to a chemiluminescent analyzer which is equipped with a NO₂ to NO converter. The NO₂ to NO converter is not necessary if data is presented to demonstrate that the NO₂ portion of the gas is less than 5% of the total NO_x concentration. The NO₂ to NO converter must undergo a conversion efficiency test in accordance with Section 5.6 of Method 20.

Method 20, which is for gas turbine engines, requires 4 calibration gases for NO_x analyzers. It also contains the requirements for the NO₂ to NO conversion check. To perform the efficiency test a mid-level NO calibration gas is added to a clear, evacuated, leak-tight Tedlar bag. Dilute this gas approximately 1:1 with 20.9% O₂. Immediately attach the bag outlet to the inlet of the NO_x analyzer and begin operation of the sampling system. Operate the sampling system, recording the NO_x response for at least 30 minutes. If the NO₂ to NO conversion is 100%, the instrument response will be stable at the highest peak value observed. If the response at the end of 30 minutes decreases by more than 2% of the highest peak value, the system is not acceptable and corrections must be made before repeating the check.

An interference response check must be performed before the initial field use for each analyzer. The interference response check involves running CO, SO₂, O₂ and CO₂ gases directly to the analyzer to make sure that there are no interferences by any other constituents. Once the interference response check is complete, the NO_x analyzer can be run according to the same procedures as Method 6C, except ambient air may be used as a zero gas if necessary. A heated sample line is necessary to keep from scrubbing NO_x in the sample line.

Method 10:

Method 10 is for CO analysis. Method 10 also follows method 6C closely except that four calibration gases are needed at different ranges. A zero gas is needed, and the low-level gas should be approximately 30% of span, the mid-level gas should be approximately 60% of span and the high-level gas should be around the span value. The span range shall not exceed 1.5 times the applicable standard. The calibration gases shall be certified by the manufacturer to be within plus or minus 2% of the specified concentration. In other words, a EPA Protocol 1 gas is not necessary, but should be used for all compliance testing. CO is not scrubbed by water so that an un-heated sampling line can be used. If an NDIR type CO analyzer is used (ACS or Fuji) an ascarite tube is required to eliminate CO₂ interference. The removed CO₂ volume must be accounted for in calculating the CO concentration.

Method 25A:

Method 25A is for total hydrocarbon determination. In this method, the span value usually established in applicable regulations is 1.5 to 2.5 times the emission limit. If no span value is provided, use a span value equivalent to 1.5 to 2.5 times the expected concentration.

This method also states that the probe should be located within 10% of the center of the stack. In most cases, THC's are measured on a wet basis. This means that the THC analyzer shall be in front of all gas conditioning systems. The heated sample line will connect directly into the back of the analyzer. Obviously, the probe and the sample line need to be heated.

In this method, four calibration gases are needed. A zero gas (which is either less than 1 ppm or 0.1% span); a low-level cal gas (25 - 35% of span); a mid-level cal gas (45 - 55% of span); and a high-level cal gas (80 - 90% of span) are needed. The Calibration Error and Bias Check should be run as normal. Since the THC monitor is ahead of all gas conditioning systems, there is no difference between the CE and the Bias Check. Therefore, all 4 gases can be run up to the probe instead of directly to the analyzer.

For this method, the zero can only drift by plus or minus 3% of span, the Calibration Drift can only be less than plus or minus 3% of span, and the Calibration Error can only be less than plus or minus 5% of the calibration gas value (NOTE: that 5% is of the calibration gas not the span, this is different than Method 6C). The

Calibration Error test must be completed within 2 hours of the start of the tests. CAE usually calibrates the THC monitor with Propane gas, so make sure that the plant calibrates to Propane also or that the emission standard is in THC as propane. If the plant does calibrate to something other than propane a correction factor must be applied. This correction factor equation multiplies the measured value by a constant. The constant is equal to 2 for ethane, 3 for propane and 4 for butane. This gives the organic concentration as carbon. See method 25 equation 25A-1 for more information.

Monitor Certification Procedures: (Part 60)

Performance Specification 2: (Relative Accuracy Testing)

The methods listed above were for running analyzers for compliance runs. There are more regulations that show how a plants monitors should be certified. This procedure is usually referred to as a Relative Accuracy Test Audit (RATA). This test is used to make sure that the plants monitoring system is reading the actual concentration of gas coming out of the stack. The RATA is just a series of tests in which a separate monitoring system is setup at a plant and the results are compared between the monitors over a period of time. A RATA consists of at least 9 runs. Each run has to be at least 21 minutes long. Up to 12 runs can be performed and three runs can be eliminated, but all of the 12 runs have to be reported. Actually more than 12 runs can be performed with only three runs being thrown out, but if they don't pass after 12 runs they're probably not going to pass at all.

The measurement location for the Reference Method probe (CAE's probe) has to be at least two diameters downstream from the nearest control device and at least .5 diameters upstream. It does **not** have to be at the same location as the CEM probe (Plant's probe). Three traverse points must be used for the Reference Method probe. These points need to be **16.7%, 50% and 83.3%** of the stack diameter. If the stack diameter is greater than 2.4 meters (7.87 feet) you can use points that are **.4, 1.2 and 2** meters (15.8, 47.2 and 78.7 inches) from the stack wall. However, you must use the percentage points after a wet scrubber or where two streams with different pollutant concentrations are combined, unless a stratification test is done. All sampling must be conducted within 3 cm (1.2 inches) of the traverse points.

The plant must operate at greater than 50% of maximum load during these tests. The plant must also conduct a Calibration Drift test. The CD test is conducted over a 7

day period usually by plant personnel. The RATA test may be conducted during the CD test period. Protocol gases are not required for the CD test. The drift over each of the 7 days must be less than 2.5% of span for pollutant monitors or less than .5% O₂ or CO₂ for diluent monitors.

PS 2 contains all of the information necessary for certifying NO_x and SO₂ analyzers. The difference between the RM and CEM analyzers must be no greater than 20% relative accuracy for NO_x and SO₂. The relative accuracy number is based on the differences between the runs, a standard deviation and a confidence coefficient. All the calculations will be presented in the Lotus spreadsheets. A 10% relative accuracy can be used if the numbers are based on a emission standard instead of the raw averages. For SO₂ emissions between .3 and .2 lb/10⁶Btu, a 15% relative accuracy, based on the emission standard, can be used. For SO₂ emissions less than .2 lb/10⁶Btu, a 20% relative accuracy, based on the emission standard, can be used.

For some plants, a moisture and/or a diluent correction may need to be done. Moisture and diluent determinations must be within a 60 minute period for each run.

The monitors will be run in the same way as is stated in each of their respective methods. A Calibration Error will be performed and then an initial Bias Check. To be consistent on Lotus spreadsheets, the Calibration Error will be called CALE and the initial Bias Check will be called CAL00. A Bias Check will be performed before and after each run. The runs will be called RUN01, RUN02 and RUN03 etc. The Bias Checks will be named after the run in which they follow. For example, the Bias Check after RUN01 will be called CAL01 and so on. All of these files will be under a sub-directory for that date of testing. The date sub-directory will be under another directory specifying which unit and what location that is being tested. Finally, the location sub-directory will be under the job number directory. For example, the data files will be turned into reports as follows: c:\6652\un1\5-20\run11.wk1.

The averages over the course of each run must be corrected for the calibrations before and after each run. A copy of a Lotus spreadsheet that performs this calculation will be included with this packet. A note on data taken from Labtech. When Labtech starts a run it prints an instantaneous average that is labeled time zero. This number should not be used in any of the calculations since it is not a minute average. Also, when Labtech does print a minute average it prints it for the period preceding the time marked. For example, a data point that says 9:03 is based on the 60 seconds between 9:02 and 9:03. So if there is a 21 minute run that starts at 9:15 to 9:36 the data that would be used off of Labtech would be 9:16 to 9:36, for a total of 21 data points. Care should be taken

when comparing the RM data to the CEM data. Some data acquisition systems have the opposite convention, a data point labeled 9:03 is the average between 9:03 and 9:04.

A copy of the Lotus spreadsheets that are included in this packet should be turned in with the data from every job. The first spreadsheet is simply called RUN.wk1. This spreadsheet will have all the individual runs stored in it. In addition, it will contain the equation for correcting the data for the Bias Checks. It will also contain any equations that may be necessary to correct the data for diluent or moisture factors and for converting the ppm results into lb/10⁶Btu or into lb/hr. The second spreadsheet is called CAL.wk1. This spreadsheet contains the equations for determining the bias check calibrations and calibration drift that may have occurred over the run. This spreadsheet will contain all the cals for all the runs. The final spreadsheet is called RA.wk1. This spreadsheet contains the calculations for determining the Relative Accuracy. This file should be renamed for each Relative Accuracy that is done. For example, the SO₂ relative accuracy should be called SO2RA.wk1. The NO_x RA will be called NOXRA.wk1. At the end of a 9 run RATA for SO₂ and NO_x the following files should be handed into reports: RUN.wk1, CAL.wk1, CALE.wk1, SO2RA.wk1, NOXRA.wk1, RUN00.wk1... RUN09.wk1, CAL00.wk1 ... CAL09.wk1. A 7 day Calibration Drift that is performed by the plant should also be included in the report.

Performance Specification 3:

PS 3 is for certifying O₂ and CO₂ monitors. The only difference in this method is that the relative accuracy must be within 20% or 1% by volume O₂ or CO₂ whichever is greater.

Performance Specification 4:

PS 4 is for certifying CO monitors. The only difference in this method is that the relative accuracy must be within 10% or within 5% of the emission standard, whichever is greater.

Cylinder Gas Audit: 40 CFR (Appendix F)

This test is to determine that the monitor is linear over the entire range of the monitor. The cylinder gas audit is similar to the BIF Calibration Error Check. But in

Appendix F, the CGA calls for only two spans to be run to the analyzer (no zero gas is required). The ranges of the pollutant calibration gases for the CGA should be **20 - 30%** of span for the low range and **50 - 60 %** of span for the high range. For CO₂ the ranges are **5 - 8%** by volume for the low range and **10 - 14%** by volume for the high range. For O₂ the ranges are **4 - 6%** by volume for the low range and **8 - 12%** by volume for the high range. The requirement for passing this test is that the accuracy is less than plus or minus **15%** or less than plus or minus **5 ppm** whichever is greater. This test must be performed quarterly. This test is usually done by plant personnel but CAE performs them occasionally. Protocol gases must be used for this test.

Relative Accuracy Audit: 40 CFR (Appendix F)

The RAA may be performed three of four calendar quarters. The RAA is used when certifying a time share CEM system. The RAA is setup and run exactly like the 40 CFR RATA except only 3 sets of measurement data are required. The requirements for the RAA is less than plus or minus 15% of the 3 run average or less than plus or minus 7.5% of the applicable standard. A full monitor certification must be performed on one of the units.

Boiler and Industrial Furnaces Methods:

These methods apply to Boilers and Industrial Furnaces that burn hazardous waste. The certification process is a little bit different than the Performance Specifications. The BIF regulations call for certifying CO, O₂ and THC only.

All of these monitors still follow the same procedures in each specific regulation. The BIF calls for Calibration Error, Calibration Drift and Response Time tests for all three of the plants analyzers. It also calls for a RATA for the CO and O₂ monitors.

The Calibration Error test involves running our Protocol 1 gases to the plants CEM system. The gas must flow up to the probe and through all of the sampling system. Usually a plant has a 0-25% span for O₂; 0-300 ppm for Low CO; 0-3000 ppm for High CO and 0-100 ppm for THC. Three calibration gases are needed for the CE check. 0 - 20% for the zero gas, 30 - 40% for the mid level gas, and 70 - 80% for the high level gas. The method specifies that the gases should be run in the following order:
Z,M,H,M,Z,H,Z,M,H (Z=ZERO, S=STACK, M=MID, H=HIGH). For all three ranges the CE must be within **5 %** of span for CO and for THC. Therefore, for low CO the CE

must be within 15 ppm of the calibration gas, for high CO the CE must be within 150 ppm and for THC the CE must be within 5 ppm. For O₂, the gases must be 0 - 2% for the zero, 8 - 10% for the mid, and 14 - 16% for the high by volume. The CE for O₂ must be within .5% by volume. This test must be performed quarterly and must use protocol gases.

The Response Time Test involves running the zero and high range gases to the plants analyzers and then waiting until the output stabilizes and then switching to stack gas. The time it takes the analyzer to go from the high and low range gas to 95% of the final stack gas is the Response Time. Each gas must be run 3 times. The average of these three runs is compared for the high range and for the zero gas. The greater of these two averages is the response time. Run the gases in the following way:

Z,S,H,S,Z,S,H,S,Z,S,H,S. This test is subject to interpretation because it is hard to reach 95% of the final stable value if the plant is not running very stable. Therefore, you need to use your best judgment to determine the Response Time.

The Response Time test and the Calibration Error test can be combined to save a few steps. The gases can be flowed in the following order:

Z,S,M,H,S,M,Z,S,H,S,Z,S,M,H,S.

The Calibration Drift is usually performed by the plant. Each monitor must have a zero and a high gas run to it. The CD must be less than 3% of span for CO and THC and within .5% by volume for O₂. The CE, RT and RATA tests must be conducted at the same time as the CD test, according to the method.

The RATA is run basically the same way as a PS 2 test. Only CO and O₂ need to be certified however. The same three points should be used to traverse the stack for a total time of 21 minutes. Moisture determinations (if needed) must be within 60 minutes for each run. The RA calculation is based on one comparison for CO corrected at 7% O₂. There is no separate calculation for CO or O₂ by themselves.

Monitor Certification: 40 CFR Part 75

The new Part 75 regulations have different requirements for performing a monitor certification. Part 75 is for power plants only at this time. The main difference between Part 60 and Part 75 is that there is a requirement that flow monitors be installed and certified at every sight.

A daily Calibration Error Check must still be performed on the CEM's by plant personnel. The CE must be within 2.5 % of the span and they must use protocol gas. If

the span is less than 200 ppm the absolute value of the difference between the monitor response and the cal gas has to be less than or equal to 5 ppm. The CE for flow monitors must be less than or equal to 3%.

A Linearity Check (what 40 CFR Part 60 calls a CGA) must be conducted quarterly. Part 75 calls for a low, mid and high range gas to be used for each monitor. (The CGA only calls for the mid and high). The Linearity Check must be less than or equal to 5 % of the reference value. Note this is 5% of the reference value not the span value as it was in the CE check. The check is also acceptable if the absolute value of the difference between the average of the monitor response and the average of the reference values is less than or equal to 5 ppm. For CO₂ and O₂ monitors the L.C. must be less than 5% or less than or equal to .5% CO₂ or O₂ whichever is less restrictive. The gas must be run through all filters, gas conditioners etc. Challenge the monitors three times with each reference gas (the calibration gases must be Protocol 1). Do not use the same gas twice in succession. For each concentration use the average of the responses to determine the linearity.

A Cycle Time/ Response Time test must also be completed. A low level and a high level gas are used for this test. Run one gas up to the probe and time the response to 95 % of the final stable value. When one gas is done run the other gas up to the probe and time that value. The Response Time test passes if both readings are less than 15 minutes.

A RATA and Bias test must also be completed.. The relative accuracy is run the same way as Part 60 except a flow monitor RATA must also be completed at three different load levels. The RATA limits have changed in Part 75. For SO₂ and NO_x the RA must be less than 10%. For SO₂ if the average is below 250 ppm then the mean value cannot exceed 15 ppm of the RM mean value. For NO_x, if the mean value on a LB/10⁶BTU level is less than .2 LB/10⁶BTU then the measurements cannot exceed .02 LB/10⁶BTU if the 10 % RA is not achieved. For CO₂, the RA must be less than 10 % or within 1% CO₂, only if the CO₂ is run on a separate system than the NO_x monitor. If the NO_x and the CO₂ are measured using the same sampling system than the CO₂ RATA will be combined with the NO_x RATA and reported as a LB/10⁶BTU number. If any of the RA's are less than 7.5 % then the plant only has to perform a RATA every year instead of every six months. For the CEM data if there is a moisture correction necessary, 1 moisture run must be performed for each wet to dry conversion run. If the moisture # is only being used to determine the molecular weight. Then 1 moisture run must be completed for every 3 flow traverses.

After three runs, it is a good idea to look at the runs to see if they are going to pass or not. If 4 runs are completed and then they look like they are going to fail. That is considered an attempt at a RATA. Therefore, they can only have one attempt left to try to avoid a bias in their numbers.

For the flow monitor, the RA must be less than 15 % or if the flow is less than 10 fps then the mean values should be within 2.0 fps. Part 75 states that all runs must be within a 30 to 60 minute time period. This means that the flow and CEM runs must be completed in a period no shorter than 30 minutes long and no longer than 60 minutes. This does not mean that the runs have to be 30 minutes in length. The CEM runs can be no shorter than 21 minutes. The flow RATA will last as long as it takes to complete the traverse. Therefore, only one CEM run and one flow run can be completed in a 30 minute period. The flow traverse must be performed at three load levels: 1) between minimum safe operating level and 50 % load, 2) between 80 % load and maximum safe operating level and 3) at the normal operating level. The pollutant analyzer RATA and the normal flow RATA must be performed at the same time. The flow RATA must pass at all three load conditions.

The Bias test is used to see if the CEM monitors read consistently lower than the RM tests. If the mean difference is greater than the absolute value of the Confidence Coefficient then the monitor has failed the Bias test. If it fails the Bias test, a correction factor must be applied to the monitors until another RATA is completed. This correction factor is defined as one plus the absolute value of the average difference divided by the average CEM concentration.

For flows, the higher bias is the one used and is then applied to the monitor. Calculate the Bias for all 3 conditions and choose the highest one. If there is one load that is greater than 10% but less than 15%, then the flow traverse 6 months later will only have to be performed at one level.

A Data Acquisition Test must also be performed. This test is a check to make sure that the DAS performs all the correct emission calculations and has all the right procedures for reporting missing data etc.

MONITOR SETUP PROCEDURES AND HELPFUL HINTS:

When arriving on site, the first thing that should be done is to turn on the monitors. The monitors need at least a couple of hours to warm up before they will run normally. Don't try to run calibration gas to the monitors before they warm up because they may not read accurately.

Once the monitors are turned on, the test locations need to be setup. Usually, due to the location of the trailers, a hot, wet sample is brought down from the stack and a cold, dry sample down from the inlet. If a cold sample is brought down, make sure that in cold weather (less than 40F) the sample line is heat traced. If it is not heat traced, moisture can condense in the line. When running cold line, the gas conditioner has to be at the test location. The gas conditioner can get plugged in cold weather when the water freezes in the passes. The conditioner should be kept inside if at all possible.

The sampling probe consists of a heated probe hooked up to a hot box. There should be a tee at the end of the probe. One part of the tee is hooked up to the probe, one part is hooked up to the heated sample line and the other part is hooked up to the calibration line. The cal line will be opened and closed from the trailer so that a valve is not necessary at the end of the probe.

Attached to the probe is a frit which is the filter for the sampling system. This frit should be at the end of the probe so that the probe itself does not get plugged with particulate. Make sure the frit is clean before the start of testing. A dirty frit has the possibility of scrubbing SO₂. Also, these frits can get clogged easily, especially at an inlet location, so they should be replaced after every day. The heated probe and the hot box should be kept at 250F to prevent condensation. If these are not heated then the stack gas will condense and not only will SO₂ be scrubbed but the stainless steel fittings will be eaten away by the acids in the gas. Out of the hot box runs a heated sample line which is also kept at 250F. The heated sample line connects to a gas conditioner. The gas conditioner temperature should be kept at about 35F. Make sure that the drain's underneath the gas conditioner are clear or water can get backed up in the gas conditioner. The sample line can then be hooked up to the outlet of the gas conditioner. The sample line and the cal line will then be run to the trailer. If the ambient temperature is below 40F a heat traced line must be used or condensation may form and freeze in the sample line. When running a long sample line (greater than 100 feet) it is usually better to keep the sample pump in the trailer. If the pump is at the location and the flow is

being regulated in the trailer, the response time for the system will be significantly increased because the sample line will be pressurized with extra gas.

Once the sampling system is completely hooked up, a leak check must be done. The leak check should be done from the tip of the probe. A leak check would be considered good if there is no flow going through the sample flow meter. When the leak check is completed and everything is up to temperature, stack gas can now be pulled down to the trailer.

At the outlet location, where a heated sample line goes all the way to the trailer, there is a similar setup procedure, but the gas conditioner will be in the trailer. There will still be a heated probe connected to a hot box with a heated sample line coming out of it. It is important to note that the heated sample lines are not designed to support their own weight. If a heated sample line has to be run down a long distance it should be connected to a rope and mesh support so that the rope and mesh can support the weight. Also on the heated sample line, there is a minimum bend radius of 12 inches. Make sure the heated sample line always lays straight or there is the possibility that the tubes can get kinked. If there is excess heated sample line (H.S.L.) laying on the ground make sure the H.S.L. doesn't overlap itself or hot spots can develop in the H.S.L. and melt it.

If there is a THC analyzer being run, the H.S.L. will be connected directly to the back of the THC analyzer. If other analyzers are being run put a tee at the inlet to the THC analyzer. One end will be connected to the H.S.L., the second end into the THC analyzer and the third end will be run to the gas conditioner. Make sure the line going to the gas conditioner is also heated or moisture can condense in this. When performing a leak check on a system which has a THC analyzer in it, the THC analyzer must be taken out of line, because some THC analyzers can leak backward and give an indication of a bad leak check. The cold dry lines are then connected to the flow panel which will regulate the flow and direct it to the right place.

The THC monitor takes in between 1 & 2 liters per minute (lpm). Some THC monitors bleed air into the sample so that this sample cannot be used by any of the other analyzers. The NO_x monitor also bleeds air into the sample so that its sample cannot be used by any of the other analyzers. The NO_x has its own pump which usually pulls about 1 lpm. The TECO 48 CO monitor does not destroy the sample, but it does have its own pump. The monitors that have their own pump need to have a tee somewhere upstream of the sample inlet so that the gas coming down from the stack does not over or under pressurize the analyzers. The SO₂, CO₂ and paramagnetic O₂ analyzers do not have pumps and do not destroy the sample, so they can be run in series with each other. The

Servomex O₂ and CO₂ analyzers have a flow sensor that measures the flow using restrictors. These restrictors can pressurize the flow upstream of the analyzers. If there is an analyzer directly upstream of these analyzers it can cause higher readings than it should. The Teledyne O₂ analyzers use a chemical cell to detect O₂. They can destroy a small amount of the sample so they should be the last analyzer in the series.

When using a flow panel, the pumps on the NO_x and CO monitors need to be taken out. The flow panels will provide positive pressure to each analyzer so that the pumps are not necessary.

FIELD RECOVERIES

ISO NUMBER: LAB-12

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

- a. general train recovery
 1. disassemble train
 2. wipe each impinger with a paper towel to remove excess moisture which will interfere with determining volumes gravimetrically.
 3. Weigh each impinger and record gross weight (volume)
 4. Follow appropriate recovery techniques for the method in use.
1. EPA Method 5 recovery and analysis
 - a. Remove tared Quartz filter from holder using tweezers. Place in appropriate container (petri dish) This should be labeled with filter ID number, unit and run number.
 - b. Rinse probe liner, nozzle, cyclone bypass, and front half of filter with acetone. Collect rinse in 250 ml glass container. Label appropriately.
 - c. Weigh impingers and record volumes
 - d. Empty contents of impingers 1-2-3 into 500 ml plastic container. Rinse each impinger and corresponding U bends three (3) times with DI water. Place rinses in the same sample container as the catches.
 - e. Note volume of total sample now in container. Record this volume on the lid or label of container. (This volume will be needed when filling out chain of custodies and Fed-Ex forms)
 - f. Note volume level by marking a liquid level line on the sample container with permanent marker.
 - g. Using teflon tape, seal each sample container around lid.
 - h. Make sure each sample container is properly labeled. Place in plastic bag and close with a zip tie.
 - i. Fill out client weight sheet. Include identification numbers of filters and beakers, description of sample, volumes, etc.

- j. Remove lids from filters (place underneath to maintain easy identification)
 - k. Transfer Acetone front half wash to a tared beaker. Document beaker number, unit and run number. Evaporate under the hood. After beaker is completely dry, place in desiccator. Desiccate samples 24 hours before first weighing. Wait at least 6 hours before taking second weight. Weigh until two weights are within 0.0005 g of each other. Record data on client weight sheet.
 - l After weights are recorded, filters are archived on appropriate shelf in lab. Front half acetone beakers need to be reconstituted with acetone. (Scrap sides and bottom of beakers with a rubber policeman or teflon spatula to remove all residue) Rinse several times with acetone and return this wash to the original sample jar. This jar is then archived along with the filters.
2. EPA Method 5/29 in house lab analysis procedure
- Follow the procedure for Method 5 samples; with the following changes.
- a. Front half acetone beakers are to be reconstituted with the front half 0.1 N HNO₃ rinse of the corresponding unit and run number.
 - b. The front half 0.1 N HNO₃ and the filters are then sent to the appropriate independent lab for analysis.

**FOR INDIVIDUAL TRAIN SET-UP AND RECOVERY
TECHNIQUES, REFER TO FLOW CHART CHEAT SHEETS**

3. NJATM5 in house analysis
- a. Recover filter as usual, desiccate and weigh.
 - b. Measure volume of acetone and evaporate to dryness in tared beaker. Desiccate and weigh.
 - c. Measure volume of back half water catch and rinse.
 - d. Filter impinger solution (back half catch and rinse) through a tared Gooch crucible, fitted with a glass fiber filter disc. Dry the crucible/filter at 220-230°F. Desiccate and weigh.
 - e. Extract organic particulate from the impinger solution first with 3 (25ml) portions of chloroform and then with 3 (35ml) portions of ethyl ether. Combine the chloroform and ethyl ether extracts in a tared beaker. Evaporate at about 70°F until no solvent remains. Desiccate and weigh.
 - f. ***Extractions are to be done under the hood with proper PPE.

- g. The impinger solution remaining after the extractions should be evaporated in a tared beaker at 220-230°F. Desiccate and weigh.

**The total particulate weight collected, corrected to 12% CO₂ is the sum of the weights of particles as determined in sections (b) through (f).

CAE STANDARD OPERATING PROCEDURE

PREPARATION

OF 8" X 10" GLASS FIBER FILTER

ISO NUMBER: PFIL-1

EFFECTIVE DATE: Pending

AUTHOR: Dept. 64

APPROVED BY: Pending

1.0 Purpose- This procedure provides the steps for proper preparation and handling of tared 8" x 10" glass fiber for use in high volume particulate testing.

2.0 Scope- This procedure applies to glass fiber filters tared in the CAE Gravimetrics Lab.

3.0 Materials-

- Powder free lab gloves
- Plastic or Teflon Tweezers
- 934-AH 8" x 10" glass fiber filters
- Kimwipes
- Bates Numbering Machine
- Teflon Coated Stamping Board
- Desiccator
- Drierite (Calcium Sulfate)
- Anti-static Brush
- Four Place Analytical Balance (Mettler AJ100)
- Filter Tare Sheets
- Black Pen
- File Folders
- Envelopes (11" x 13")
- Two 13" x 9" Glass Baking Pans (Cleaned and Acetone Rinsed)

4.0 Definitions-

g - Abbreviation for gram(s)

8 x 10 - Refers to the size of the filter; eight inches by ten inches

GFF - Abbreviation for glass fibre filter

5.0 Procedure-

- 5.1. Obtain untared 8" x 10" glass fiber filters from glass cabinet in the Gravimetric Lab and, using tweezers or gloves, transfer the needed amount to a clean acetone rinsed 13" x 9" glass baking pan.
- 5.2. Obtain the Bates numbering machine from the drawer in the center aisle of the lab.
- 5.3. Adjust the Bates numbering machine to reflect next set of filters in the 8" x 10" series.
- 5.4. Place stamping board teflon side up and clean the surface with a Kimwipe.
- 5.5. Using tweezers, remove the filter from the baking pan and place it rough side down on the stamping board.
- 5.6. Carefully stamp the number in the upper right hand corner of the filter.

Note: Too much pressure can rupture the filter!!

- 5.7. Pick up the filter with tweezers and check it against the light to make sure that no break through has occurred and that the printing is legible.
 - a) If a break through has occurred or the printing is illegible, note the filter number and a brief reason for voiding, then dispose of the filter in the trash. This information will be used later to account for all filter numbers in this series.
 - b) If no break through has occurred and the printing is legible, place the filter in the second clean, acetone rinsed baking pan.
- 5.8. Continue to repeat Steps 5.5-5.7 until all filters have been stamped. Filters are now ready for the desiccator.
- 5.9. Check the Drierite in the dessicator.
 - a) If Drierite is blue/purple proceed to Step 5.10.
 - b) If Drierite is pink, complete the procedure for changing and regenerating drierite before proceeding.
- 5.10. Place the uncovered baking pan of stamped filters in the dessicator. Close the dessicator door and dessicate for no less than 24 hours before proceeding.
- 5.11. After dessicating for at least 24 hours, place the unused baking pan on the bottom shelf of the dessicator and place the baking pan with the filters on the top shelf. Make sure that the filters are in sequential order beginning with the first number in the series.

Note: Do not expose desiccated filters to the lab environment for more than 2 minutes. Keep in a dessicator until needed.

- 5.12. Calibrate the analytical balance. Complete the procedure for the calibration of the Mettler AJ100 balance before continuing.
- 5.13. Using tweezers, remove the first filter from the baking pan and place it on the center of the weight pan. Close the balance lid and wait for the balance to stabilize.
- 5.14. Record the weight on the first line of the filter tare sheet and fill in all the required information
- Note:* Time can be recorded in 5 minute increments. Usually a number of filters can be weighed in this time period, so more than one filter can have the same time.
- 5.15. Remove the filter from the balance and place it in the baking pan on the bottom shelf.
- 5.16. Re-zero the balance, if necessary. Repeat steps 5.13-5.15 until all filters have a weight.
- 5.17. Transfer all filters back into the dessicator. Close the dessicator door and dessicate the filters for no less than six hours.
- 5.18. After dessicating for at least six hours, repeat Steps 5.12-5.17 until two consecutive weights are within ± 0.5 mg of each other.
- 5.19. Once the conditions in Step 5.18 are met, place each filter in its own file folder and write the corresponding filter number on the folder with a black marker.
- 5.20. Repeat Step 5.19 until all the filters have been placed in file folders and labeled.
- 5.21. Place each file folder into its own labeled envelope.
- 5.22. Bring the tared filters to the Field Support Lab and place them in the tared media cabinet located in the chemical room.
- 5.23. Fill out the date and filter numbers on the media tracking form on the cabinet.

6.0 References-

- 6.1. Standard Test Method for High Volume Sampling for Solid Particulate Matter and Determination of Particulate Emissions
- 6.2. 40 CFR Part 60 Appendix A Method 5

7.0 Attachments-

- 7.1 Filter Tare Sheet

CAE STANDARD OPERATING PROCEDURE

CALIBRATION
OF THE ANALYTICAL BALANCES

ISO NUMBER: PFIL-2

AUTHOR: Dept. 64

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1.0 Purpose- The purpose of this procedure is to ensure that the analytical balances used in the Gravimetrics Laboratory are properly calibrated.

2.0 Scope- This procedure applies to any employee performing the calibration of the analytical balances used in the Gravimetrics Laboratory: the Ohaus GA110, the Ohaus GA200D and the Mettler AJ100.

3.0 Materials-

- Ohaus GA110
- Ohaus GA200D
- Mettler AJ100
- Tweezers
- Class "S" Calibration Weights
- Calibration Log
- Black Pen

4.0 Definitions-

g - Abbreviation for gram

mg - Abbreviation for milligram

5.0 Procedure 1 - Ohaus GA110

Note: The balances should be calibrated every day that they are in use.

5.0.1. Turn the balance on by pressing the "ON/OFF" button.

5.0.2. Level the balance. This is done by adjusting the knobs underneath the balance so that the liquid bubble is in the middle of the circle.

5.0.3. Zero the balance by pressing the "RE-ZERO" button.

- 5.0.4. Slowly flip the switch on the right hand side of the GA110 to the position marked "CAL". (180°) (If "CE" is displayed, flip the switch back to its original position and repeat this step.)
- 5.0.5. After about 15 seconds the digital display will blink "C" several times and then will read "CC". At that point, the internal calibration is complete. Flip the switch back to its original position (180°).
- 5.0.6. Take out the class "S" calibration weights and the calibration log located in the right hand drawer of the balance desk.
- 5.0.7. Using tweezers, place the 500 mg weight on the balance pan, close the balance doors and wait for the balance to stabilize.
- 5.0.8. If the balance indicates that the weight is within ± 0.2 mg of 500 mg, proceed to Step 5.0.9.
If the balance indicates that the weight is not within ± 0.2 mg of 500 mg, repeat Steps 5.0.3 through 5.0.7.
- 5.0.9. Using tweezers replace the 500 mg weight in the kit and place the 1.0 g weight on the balance pan, close the balance doors and wait for balance to stabilize.
- 5.0.10. If the display indicates that the weight is within ± 0.2 mg of 1 g, proceed to Step 5.0.11.
If the display indicates that the weight is not within ± 0.2 mg of 1 g, repeat Steps 5.0.3 through 5.0.9.
- 5.0.11. Record the date, time and weights in the calibration log. Initial the entry.
- 5.0.12. Calibration of the Ohaus GA110 is complete when steps 5.0.1 through 5.0.12 have been performed.

5.1 Procedure 2 - Ohaus GA200D

- 5.1.1. Turn balance on by pressing the "ON/OFF" button.
- 5.1.2. Level the balance. This is done by adjusting the knobs underneath the balance so that the liquid bubble is in the middle of the circle.
- 5.1.3. Zero the balance by pressing the "RE-ZERO" button.
- 5.1.4. Slowly turn the knob on the left side of the GA200D from the position marked "Weighing" two clicks downward to the position marked "CAL 200". (If "CE" is displayed return the knob to its original position and repeat this step.)
- 5.1.5. After about 15 seconds, the display will flash "C" and then will read "CC". At this point the internal calibration is complete. Turn the knob back to its original position marked "Weighing".
- 5.1.6. Take out the class "S" calibration weights and calibration log from the right hand drawer on the balance desk.

- 5.1.7. Using tweezers place the 1 g weight on the balance pan, close the balance doors and wait for the balance to stabilize.
- 5.1.8. If the display indicates that the weight is within ± 0.2 mg of 1 g proceed to Step 5.1.9.
If the display indicates that the weight is not within ± 0.2 mg of 1 g repeat Steps 5.1.3 through 5.1.7.
- 5.1.9. Using tweezers, replace the 1 g weight in the kit and place the 100 g weight on the balance pan, close the doors and wait for the balance to stabilize.
- 5.1.10. If the display indicates that the weight is within ± 0.2 mg of 100 g proceed to Step 5.1.11.
If the display indicates that the weight is not within ± 0.2 mg of 100 g repeat Steps 5.1.3 through 5.1.9.
- 5.1.11. Record the date, time and weights in the calibration log. Initial the entry.
- 5.1.12. Calibration of the Ohaus GA200D is complete when Steps 5.1.1 through 5.1.11 have been performed.

5.2 Procedure 3 - Ohaus GA200D (Five Place)

- 5.2.1. Turn balance on by pressing the "ON/OFF" button.
- 5.2.2. Level the balance. This is done by adjusting the knobs underneath the balance so that the liquid bubble is in the middle of the circle.
- 5.2.3. Zero the balance by pressing the "RE-ZERO" button.
- 5.2.4. Switch the balance to five place mode by pressing the "RANGE" button. Check to make sure that the balance goes into five place mode before proceeding.
- 5.2.5. Slowly turn the knob on the left side of the GA200D from the position marked "Weighing" one click downward to the position marked "CAL 40". (If "CE" is displayed return the knob to its original position and repeat this step.)
- 5.2.6. After about 15 seconds, the display will flash "C" and then will read "CC". At this point the internal calibration is complete. Turn the knob back to its original position marked "Weighing".
- 5.2.7. Take out the class "S" calibration weights and calibration log from the right hand drawer on the balance desk.
- 5.2.8. Using tweezers, place the 0.2 g weight on the balance pan, close the balance doors and wait for the balance to stabilize.
- 5.2.9. If the display indicates that the weight is within ± 0.05 mg of 0.2 g proceed to Step 5.2.10.
If the display indicates that the weight is not within ± 0.05 mg of 0.2 g repeat Steps 5.2.5 through 5.2.8.

- 5.2.10. Using tweezers, replace the 0.2 g weight in the kit and place the 0.1 g weight on the balance pan, close the doors and wait for the balance to stabilize.
- 5.2.11. If the display indicates that the weight is within ± 0.05 mg of 0.1 g proceed to Step 5.2.12.
If the display indicates that the weight is not within ± 0.05 mg of 0.1 g repeat Steps 5.2.5 through 5.2.10.
- 5.2.12. Using tweezers, replace the 0.1 g weight in the kit and place the 0.01 g weight on the balance pan, close the doors and wait for the balance to stabilize.
- 5.2.13. If the display indicates that the weight is within ± 0.05 mg of 0.01 g proceed to Step 5.2.12.
If the display indicates that the weight is not within ± 0.05 mg of 0.01 g repeat Steps 5.2.5 through 5.2.12.
- 5.2.14. Record the date, time and weights in the calibration log. Initial the entry.
- 5.2.15. Calibration of the Ohaus GA200D for five place weighing is complete when Steps 5.2.1 through 5.2.14 have been performed.

5.3 Procedure 4 - Mettler AJ100

- 5.3.1. Turn the balance on by pressing down on the bar once.
- 5.3.2. Open the balance and rotate the clips so that the pan is held in the down position. Close the balance.
- 5.3.3. Hold the bar down until "-CAL-" is displayed. Release the bar.
- 5.3.4. "- - - - -" will be displayed.
- 5.3.5. "100.0000 g" will flash on the display. Open the balance and place the circular flat 100 g weight in the center of the wire pan. Close the balance.
- 5.3.6. "- - - - -" will be displayed again.
- 5.3.7. "0.0000 g" will flash on the display. Open the balance and remove the 100 g weight. Close the balance.
- 5.3.8. "- - - - -" will be displayed again.
- 5.3.9. "0.0000 g" will remain displayed. The internal calibration is complete. Rotate the clips so that the pan is released and returns to the up position.
- 5.3.10. Open the balance and place the 100 g weight on the center of the wire pan. Close the balance.
- 5.3.11. If the display indicates that the weight is within ± 0.2 mg of 100 g proceed to Step 5.3.12.
If the display indicates that the weight is not within ± 0.2 mg of 100 g, remove the weight and repeat Steps 5.3.3 through 5.3.10.

5.3.12. Open the balance and replace the 100 g weight in the kit. Place the 1 g weight on the center of the wire pan. Close the balance.

5.3.13. If the display indicates that the weight is within ± 0.2 mg of 1 g proceed to Step 5.3.14.

If the display indicates that the weight is not within ± 0.2 mg of 1 g, remove the weight and repeat Steps 5.3.3 through 5.3.12.

5.3.14. Record the time, date and weights in the calibration log. Initial the entry.

5.3.15. Calibration of the Mettler AJ100 is complete when Steps 5.3.1 through 5.3.14 have been performed.

6.0 Attachments-

6.1 Ohaus GA110 Calibration Sheet

6.2 Ohaus GA200D Calibration Sheet

6.3 Ohaus GA200D Five Place Calibration Sheet

6.4 Mettler AJ100 Calibration Sheet

BALANCE LINEARITY CHECK

ISO NUMBER: LAB-9

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

a. DAILY CHECK

In a notebook designated for these recordings, note the date, time, and your initials.

1. Zero the balance- press the tare bar
2. Calibrate the balance- Place the silver 100 gram calibration weight on the weighing pan and press autocal button. (Use tweezers- never touch the cal. weights with fingers). During weighting be sure both doors of balance are closed.
3. In balance log book, record the weight of the 100 gram cal. weight after the balance stabilizes (a small "g" will appear in the left corner of the display).
4. Place the 10 gram cal. weight on the weighing pan and record weight.
5. Place the 1 gram cal. weight on the weighing pan and record weight.
6. Place all weights on the weighing pan and record total weight.

NOTE: All weights should be +/- 0.0005 grams of that given weight

7. If total weight is +/- 0.0005 grams of the sum of the individual weights, the linearity is acceptable.

EX. E.N. 10/22/97 8:45

100- 100.0000

10- 10.0001

1- 0.9999

111.0000 / 111.0001

NOTE: If you are doing more weightings, 4 or more hours after your daily linearity check, place the 100 gram cal. weight on the weighing pan and press autocal before doing any more weightings.

b. RESETTING LINEARITY

1. Press set-up and display lock (lin1 should appear).
2. After lin 1 display leaves, press display lock (lin 2 should appear).
3. Place silver 100 gram cal. weight on the weighing pan and press display lock after it stabilizes (lin 3 should appear).
4. Remove cal. weight and place brass 100 gram weight on weighing pan. Press display lock after it stabilizes (lin 4 should appear).
5. Place silver cal. weight on weighing pan without removing the brass weight. Press display lock after it stabilizes (lin **** should appear).
6. Remove weights and conduct daily linearity check.

DESICCATING AND TARING

ISO NUMBER: LAB-10

EFFECTIVE DATE: Pending

AUTHOR: Dept. 68

APPROVED BY: Pending

a. BEAKERS AND THIMBLES

1. Wash beakers and thimbles in hot soapy water.
2. Rinse 3 times with hot tap water.
3. Rinse 3 times with DI water.
4. Rinse 3 times with acetone. (Thimbles do not need to be rinsed with acetone)
5. Allow to air dry.
6. Beakers can be placed in desiccator at this time. Thimbles should be placed in the muffler furnace and baked for 4 hours, cooled, and placed in desiccator.

b. FILTERS

1. Filters should be sequentially numbered on their back. Write the ID #s on the lid of a designated petri-dish with a permanent marker. Look in the tare book for the next number series.
2. Filters can now be placed in the desiccator with the lid of the petri-dish removed but kept with the designated filter for ID purposes.

NOTE: 4.7 cm glass micro-fibre filters are to be tared with two corresponding stainless steel gelman rings.

c. DESICCATOR INFORMATION

Before placing items in desiccator, check the condition of desiccant. Indicator is blue when fresh. Desiccant should be replaced when indicator has turned pinkish in color. Check the hydrometer if unsure.

NOTE: Desiccant should be baked for reuse. Place used desiccant in baking dish along with spent (pink) indicator. Bake for several hours until indicator is original blue color.

Items should remain in desiccator for 24 hours before first tare weight is taken.

d. WEIGHTING (BEAKERS, THIMBLES AND FILTERS)

After 24 hours, items are ready for first tare weighting. The following items must be included on the tare data sheet.

1. date
2. time
3. your initials
4. item ID #

After first weighting, return item to desiccator. After an additional 6 hours or more, you may obtain a second tare weight. Second tare weights must be ± 0.0005 grams of the first tare weight. If the difference is greater than ± 0.0005 grams, a third tare weight must be taken, with an additional 6 hours in the desiccator.

NOTE: There must be two (2) consecutive weightings with a difference no greater than ± 0.0005 grams.

After two tare weights are achieved, tared beakers are placed in the "clean box". Thimbles are to be placed into nalgene bottles, open side up, with ID#s marked on the bottles, and kim-wipes placed around and on top of thimble to prevent breakage. Filters are to be placed into designated petri-dishes, stacked and taped together in sets.

NOTE:

1. No item being weighted is to be handled with bare hands. Use applicable handling tool [tweezers(metal or teflon), tongs, disposable gloves]
2. Do not leave items being weighted outside desiccator for more than two (2) minutes at a time.

CAE STANDARD OPERATING PROCEDURE

PREPARATION OF TARED BEAKERS

ISO NUMBER: PFIL-3

EFFECTIVE DATE: Pending

AUTHOR: Dept. 64

APPROVED BY: Pending

1.0 Purpose- This procedure provides the steps for proper preparation and handling of tared beakers and metals prepped tared beakers for use in the Gravimetric analysis of particulate.

2.0 Scope- This procedure applies to any employee taring beakers for use in the CAE Gravimetrics Lab.

3.0 Materials-

- Powder free lab gloves
- Rubber Coated Beaker Tongs
- Beaker Tare Sheet
- Black Pen
- Desiccator
- Drierite (Calcium Sulfate)
- 8.26 cm Cardboard Circle
- Four Place Analytical Balance
- Particulate Prepped or Metals Prepped Beakers

4.0 Definitions-

Particulate Prepped Beakers- These are beakers which are cleaned and then rinsed with acetone in CAE's field support laboratory.

Metals Prepped Beakers- These are beakers that are placed in the metals bath for at least four hours, cleaned and then rinsed with acetone in CAE's Field support laboratory.

g- Abbreviation for gram

5.0 Procedure 1 - Tared Particulate Prepped Beakers-

- 5.0.1. Obtain beakers from the Field Support Lab.
 - 5.0.2. Check the Drierite in the dessicator.
 - a) If Drierite is blue/purple proceed to Step 5.0.3.
 - b) If Drierite is pink, complete the procedure for changing and regenerating drierite before proceeding.
 - 5.0.3. Place the beakers in the dessicator. Close the dessicator door and dessicate for no less than 24 hours before proceeding.
- Note:* Do not expose desiccated beakers to the lab environment for more than 2 minutes. Keep in a dessicator until needed.
- 5.0.4. Calibrate the analytical balance. Complete the procedure for the calibration of the Ohaus GA200D analytical balance before continuing.
 - 5.0.5. Using beaker tongs, remove the beaker from the desiccator and place it on the center of the weight pan. Close the balance door and wait for the balance to stabilize.
 - 5.0.6. Record the weight on the first line of the beaker tare sheet and fill in all the required information.
- Note¹:* Time can be recorded in 5 minute increments. Usually a number of beakers can be weighed in this time period, so more than one beaker can have the same time.
- Note²:* Beakers are usually identified by a letter and number combination. (examples: XX23, B130, Z28 etc.) If no identifying code is present write one in the white space or do not use that beaker.
- 5.0.7. Remove the beaker from the balance and return it to the dessicator.
 - 5.0.8. Re-zero balance, if necessary. Repeat steps 5.0.5-5.0.7 until all beakers have a weight.
 - 5.0.9. Transfer all beakers back into the dessicator. Close the dessicator door and dessicate for no less than six hours.
 - 5.0.10. After dessicating for at least six hours, repeat Steps 5.0.5-5.0.9 until two consecutive weights are within ± 0.5 mg of each other.
 - 5.0.11. Once the conditions in Step 5.0.10 are met, place the beakers into the plexiglass cases labeled "TARED BEAKERS" located to the left of the Gravimetric Lab entrance.
 - 5.0.12. Repeat Step 5.0.11 until all the beakers have been placed in the cases.

5.1 Procedure 2 - Tared Metals Prepped Beakers-

5.1.1. Follow the same procedure for the preparation of particulate prepped beakers, except substitute metals prepped beakers.

6.0 References-

6.1 40 CFR Part 60 Appendix A Method 5

7.0 Attachments-

7.1 Beaker Tare Sheet

CAE STANDARD OPERATING PROCEDURE

PREPARATION OF
4.7 CM GLASS FIBER (GELMAN) FILTERS

ISO NUMBER: PFIL-4

EFFECTIVE DATE: Pending

AUTHOR: Dept. 64

APPROVED BY: Pending

1.0 Purpose- This procedure provides the steps for proper preparation and handling of tared 4.7 cm glass fiber (Gelman) filters for use in EPA Methods 17 and 201A.

2.0 Scope- This procedure applies to 4.7 cm glass fiber (Gelman) filters tared in the CAE Gravimetrics Lab.

3.0 Materials-

- Powder free lab gloves
- Plastic or metal tweezers
- 4.7 cm 934-AH glass fiber filters
- 4.7 cm O.D. Stainless Steel Rings
- Bates Numbering Machine
- Teflon Coated Stamping Board
- Desiccator
- Drierite (Calcium Sulfate)
- Anti-static Brush
- Four Place Analytical Balance
- Filter Tare Sheet
- Black Marker
- Black Pen
- Kimwipes
- Plastic Petri Dishes

4.0 Definitions-

g - Abbreviation for gram

GFF - Abbreviation for glass fibre filter

5.0 Procedure-

- 5.1. Obtain untared 4.7 cm glass fiber filters from glass cabinet in the Gravimetric Lab and, using tweezers or gloves, transfer the needed amount to a 15 cm petri dish.
- 5.2. Obtain the Bates numbering machine from the drawer in the center aisle of the lab.
- 5.3. Adjust the Bates numbering machine to reflect the next filters in the Gelman series.
- 5.4. Place stamping board teflon side up and clean the surface with a Kimwipe.
- 5.5. Using tweezers, remove the filter from the petri dish and place it rough side down on the stamping board.
- 5.6. Carefully stamp the number in the center of the filter.

Note¹: Too much pressure can rupture the filter!!

Note²: For a complete Gelman filter setup, two filters with the same number are required.

- 5.7. Pick up the filters with tweezers and check them against the light to make sure that no break through has occurred and that the printing is legible.
 - a) If a break through has occurred or the printing is illegible, note the filter number and a brief reason for voiding, then dispose of the filter in the trash. This information will be used later to account for all filter numbers in this series.
 - b) If no break through has occurred and the printing is legible, continue with this procedure.
- 5.8. Continue to repeat Steps 5.5-5.7 until all filters have been stamped.
- 5.9. Make each filter set into a Gelman setup by placing a stainless steel ring in a petri dish, placing the two filters with the same number on top of the ring and then place another stainless steel ring on top of this. (In short: Ring, Filter, Filter, Ring). Filters are now ready for the dessicator.
- 5.10. Check the Drierite in the dessicator.
 - a) If Drierite is blue/purple proceed to Step 5.10.
 - b) If Drierite is pink, complete the procedure for changing and regenerating drierite before proceeding.

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PREPARATION OF 4.7 cm GLASS FIBER (GELMAN) FILTERS

SOP:PFIL-4
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- 5.11. Place the uncovered petri dishes of filters in the dessicator. Close the dessicator door and dessicate for no less than 24 hours before proceeding.
- 5.12. After dessicating for at least 24 hours, remove the filters from the desiccator and transfer to a small desiccator.
- Note:* Do not expose desiccated filters to the lab environment for more than 2 minutes. Keep in a dessicator until needed.
- 5.13. Calibrate the analytical balance. Complete the procedure for the calibration of the Ohaus GA100 analytical balance before continuing.
- 5.14. Using tweezers, remove the first filter from the small desiccator and place it on the center of the weight pan. Close the balance door and wait for the balance to stabilize.
- 5.15. Record the weight on the first line of the filter tare sheet and fill in all the required information
- Note:* Time can be recorded in 5 minute increments. Usually a number of filters can be weighed in this time period, so more than one filter can have the same time.
- 5.16. Remove the filter from the balance and return it to the dessicator.
- 5.17. Re-zero balance, if necessary. Repeat steps 5.14-5.16 until all filters have a weight.
- 5.18. Transfer all filters back into the dessicator. Close the dessicator door and dessicate for no less than six hours.
- 5.19. After dessicating for at least six hours, repeat Steps 5.13-5.18 until two consecutive weights are within ± 0.5 mg of each other.
- 5.20. Once the conditions in Step 5.19 are met, place each filter in its own petri dish and write the corresponding filter number on the lid of the dish with a black marker.
- 5.21. Repeat Step 5.20 until all the filters have been placed in petri dishes and labeled.
- 5.22. Tape the lids of the petri dishes with scotch tape, a small piece on each side is sufficient.
- 5.23. Bring the tared filters to the Field Support Lab and place them in the tared media cabinet located in the chemical room.
- 5.24. Fill out the date and filter numbers on the media tracking form on the cabinet.

6.0 References-

- 6.1 40 CFR Part 60 Appendix A Method 5
- 6.2 40 CFR Part 60 Appendix A Method 17
- 6.3 EPA Method 201A

7.0 Attachments-

- 7.1 Filter Tare Sheet
- 7.2 Tared Media Tracking Form

CAE STANDARD OPERATING PROCEDURE

PREPARATION OF GLASS FIBER FILTERS

ISO NUMBER: PFIL-5

EFFECTIVE DATE: Pending

AUTHOR: Dept. 64

APPROVED BY: Pending

1.0 Purpose- This procedure provides the steps for proper preparation and handling of tared glass fiber filters 8.26 cm-11 cm for use in EPA Method 5 and state equivalents.

2.0 Scope- This procedure applies to glass fiber filters tared in the CAE Gravimetrics Lab.

3.0 Materials-

- Powder free lab gloves
- Plastic or metal tweezers
- 934-AH glass fiber filters
- Plastic Petri Dishes
- Bates Numbering Machine
- Teflon Coated Stamping Board
- Desiccator
- Drierite (Calcium Sulfate)
- Anti-static Brush
- Four Place Analytical Balance
- Filter Tare Sheet
- Black Marker
- Black Pen
- Kimwipes

4.0 Definitions-

g - Abbreviation for gram

GFF - Abbreviation for glass fibre filter

cm - Abbreviation for centimeter

5.0 Procedure-

- 5.1. Obtain desired size of glass fiber filters from glass cabinet in the Gravimetric Lab and, using tweezers or gloves, transfer the needed amount to a 15 cm petri dish.
- 5.2. Obtain the Bates numbering machine from the drawer in the center aisle of the lab.
- 5.3. Adjust the Bates numbering machine to reflect appropriate number series for that filter.
- 5.4. Place stamping board Teflon side up and clean the surface with a Kimwipe.
- 5.5. Using tweezers, remove the filter from the petri dish and place it rough side down on the stamping board.
- 5.6. Carefully stamp the number in the center of the filter.

Note: Too much pressure can rupture the filter!!

- 5.7. Pick up the filter with tweezers and check it against the light to make sure that no break through has occurred and that the printing is legible.
 - a) If a break through has occurred or the printing is illegible, note the filter number and a brief reason for voiding, then dispose of the filter in the trash. This information will be used later to account for all filter numbers in this series.
 - b) If no break through has occurred and the printing is legible, place the filter in the unused half of the petri dish.
- 5.8. Continue to repeat Steps 5.5 -5.7 until all filters have been stamped. Filters are now ready for the desiccator.
- 5.9. Check the Drierite in the dessicator.
 - a) If Drierite is blue/purple proceed to Step 5.10.
 - b) If Drierite is pink, complete the procedure for changing and regenerating drierite before proceeding.
- 5.10. Place the uncovered petri dish of stamped filters in the dessicator. Close the dessicator door and dessicate for no less than 24 hours before proceeding.

- 5.11. After dessicating for at least 24 hours, remove the filters from the desiccator and transfer to a small desiccator. Place the unused half of the petri dish on the bottom shelf and place the half with the filters on the top shelf. Make sure that the filters are in sequential order beginning with the first number in the series.
- Note:* Do not expose desiccated filters to the lab environment for more than 2 minutes. Keep in a dessicator until needed.
- 5.12. Calibrate the analytical balance. Complete the procedure for the calibration of the Ohaus GA110 balance before continuing.
- 5.13. Using tweezers, remove the first filter from the small desiccator and place it on the center of the weight pan. Close the balance door and wait for the balance to stabilize.
- 5.14. Record the weight on the first line of the filter tare sheet and fill in all the required information
- Note:* Time can be recorded in 5 minute increments. Usually a number of filters can be weighed in this time period, so more than one filter can have the same time.
- 5.15. Remove the filter from the balance and place it in the petri dish half on the bottom shelf.
- 5.16. Re-zero balance, if necessary. Repeat steps 5.13-5.15 until all filters have a weight.
- 5.17. Transfer all filters back into the dessicator. Close the dessicator door and dessicate for no less than six hours.
- 5.18. After dessicating for at least six hours, repeat Steps 5.12-5.17 until two consecutive weights are within ± 0.5 mg of each other.
- 5.19. Once the conditions in Step 5.18 are met, place each filter in its own petri dish and write the corresponding filter number on the lid of the dish with a black marker.
- 5.20. Repeat Step 5.19 until all the filters have been placed in petri dishes and labeled.
- 5.21. Tape the lids of the petri dishes with scotch tape, a small piece on each side is sufficient.
- 5.22. Bring the tared filters to the Field Support Lab and place them in the tared media cabinet located in the chemical room.
- 5.23. Fill out the date and filter numbers on the media tracking form on the cabinet.

6.0 References-

- 6.1 40 CFR Part 60 Appendix A Method 5

7.0 Attachments-

- 7.1 Filter Tare Sheet
- 7.2 Tared Media Tracking Form

CAE STANDARD OPERATING PROCEDURE

PREPARATION OF QUARTZ FILTERS

ISO NUMBER: PFIL-6

EFFECTIVE DATE: Pending

AUTHOR: Dept. 64

APPROVED BY: Pending

1.0 Purpose- This procedure provides the steps for proper preparation and handling of tared 8.26 cm quartz filters for use in combined EPA Methods 5 and 29.

2.0 Scope- This procedure applies to quartz filters tared in the CAE Gravimetrics Lab.

3.0 Materials-

- Powder free lab gloves
- Plastic or Teflon Tweezers
- 8.26 cm Quartz filters
- Plastic Petri Dishes
- Metals Prepped Glass Petri Dishes
- Filter Tare Sheet
- Desiccator
- Drierite (Calcium Sulfate)
- 8.26 cm Cardboard Circle
- Four Place Analytical Balance
- Black Pen
- Black Marker
- Lab Oven
- Invisible Tape

4.0 Definitions-

cm - Abbreviation for centimeter

g - Abbreviation for gram

5.0 Procedure-

- 5.1. Obtain quartz filters from glass cabinet in the Gravimetric Lab and, using tweezers or gloves, transfer the needed amount to a 15 cm metals prepped glass petri dish.
- 5.2. Place the petri dish in the lab oven and bake for four hours at 110° C.
- 5.3. Check the Drierite in the dessicator.
 - a) If Drierite is blue/purple proceed to Step 5.4.
 - b) If Drierite is pink, complete the procedure for "Changing and Regenerating Drierite" before proceeding.
- 5.4. Remove the uncovered petri dish of filters from the oven and place them in the dessicator. Close the dessicator door and dessicate for no less than 24 hours before proceeding.
- 5.5. After dessicating for at least 24 hours, remove the filters from the desiccator and transfer to a small desiccator. Place the unused half of the petri dish on the bottom shelf and place the half with the filters on the top shelf.
- Note:** Do not expose desiccated filters to the lab environment for more than 2 minutes. Keep in a dessicator until needed.
- 5.6. Calibrate the analytical balance. Complete the procedure for the calibration of the Ohaus GA110 analytical balance before continuing.
- 5.7. Tape the 8.26 cm cardboard circle to the weighing pan in the balance.
- 5.8. Re-zero the balance.
- 5.9. Using tweezers, remove the first filter from the small desiccator and place it on the center of the weight pan. Close the balance door and wait for the balance to stabilize.
- 5.10. Record the weight on the first line of the filter tare sheet and fill in all the required information
- Note:** Time can be recorded in 5 minute increments. Usually a number of filters can be weighed in this time period, so more than one filter can have the same time.
- 5.11. Remove the filter from the balance and place it in the petri dish half on the bottom shelf.

- 5.12. Re-zero balance, if necessary. Repeat steps 5.9 through 5.11 until all filters have a weight.

Note: Since quartz filters have no identification number on them, it is extremely important to remember what order the filters are in. Keep notes.

- 5.13. Transfer all filters back into the dessicator. Close the dessicator door and dessicate for no less than six hours.
- 5.14. After dessicating for at least six hours, repeat Steps 5.9-5.13 until two consecutive weights are within ± 0.5 mg of each other.
- 5.15. Once the conditions in Step 5.14 are met, place each filter in its own petri dish and write the corresponding filter number on the lid of the dish with a black marker.
- 5.16. Repeat Step 5.15 until all the filters have been placed in petri dishes and labeled.
- 5.17. Tape the lids of the petri dishes with invisible tape, a small piece on each side is sufficient.
- 5.18. Bring the tared filters to the Field Support Lab and place them in the tared media cabinet located in the chemical room.
- 5.19. Fill out the date and filter numbers on the media tracking form on the cabinet.

6.0 References-

- 6.1 40 CFR Part 60 Appendix A Method 5

7.0 Attachments-

- 7.1 Filter Tare Sheet
- 7.2 Tared Media Tracking Form

CAE STANDARD OPERATING PROCEDURE

PREPARATION OF TEFLON FILTERS

ISO NUMBER: PFIL-7

EFFECTIVE DATE: Pending

AUTHOR: Dept. 64

APPROVED BY: Pending

1.0 Purpose- This procedure provides the steps for proper preparation and handling of tared Teflon filters for use in particulate emission testing.

2.0 Scope- This procedure applies to Teflon filters tared in the CAE Gravimetrics Lab.

3.0 Materials-

- Powder free lab gloves
- Plastic or Teflon Tweezers
- Teflon Filters
- Plastic Petri Dishes
- Filter Tare Sheet
- Invisible Tape
- Desiccator
- Drierite (Calcium Sulfate)
- Four Place Analytical Balance
- Black Marker
- Black Pen

4.0 Definitions-

g - Abbreviation for grams

5.0 Procedure-

- 5.1. Obtain Teflon filters from glass cabinet in the Gravimetric Lab and, using tweezers or gloves, transfer the needed amount to a 15 cm petri dish.
- 5.2. Check the Drierite in the dessicator.
 - a) If Drierite is blue/purple proceed to Step 5.3.
 - b) If Drierite is pink, complete the procedure for "Changing and Regenerating Drierite" before proceeding.
- 5.3. Place the uncovered petri dish of filters in the dessicator. Close the dessicator door and dessicate for no less than 24 hours before proceeding.
- 5.4. After dessicating for at least 24 hours, remove the filters from the desiccator and transfer to a small desiccator. Place the unused half of the petri dish on the bottom shelf and place the half with the filters on the top shelf.
- Note:* Do not expose desiccated filters to the lab environment for more than 2 minutes. Keep in a dessicator until needed.
- 5.5. Calibrate the analytical balance (Ohaus GA110). Complete the procedure for the "Calibration of Balances" before continuing.
- 5.6. Using tweezers, remove the first filter from the small desiccator and place it on the center of the weight pan. Close the balance door and wait for the balance to stabilize.
- 5.7. Record the weight on the first line of the filter tare sheet and fill in all the required information
- Note:* Time can be recorded in 5 minute increments. Usually a number of filters can be weighed in this time period, so more than one filter can have the same time.
- 5.8. Remove the filter from the balance and place it in the petri dish half on the bottom shelf.
- 5.9. Re-zero balance, if necessary. Repeat steps 5.6-5.8 until all the filters have a weight.
- Note:* Since Teflon filters have no identification number on them, it is extremely important to remember what order the filters are in. Keep notes.
- 5.10. Transfer all filters back into the dessicator. Close the dessicator door and dessicate for no less than six hours.
- 5.11. After dessicating for at least six hours, repeat Steps 5-10 until two consecutive weights are within ± 0.5 mg of each other.
- 5.12. Once the conditions in Step 5.11 are met, place each filter in its own petri dish and write the corresponding filter number on the lid of the dish with a black marker.

- 5.13. Repeat Step 5.12 until all the filters have been placed in petri dishes and labeled.
- 5.14. Tape the lids of the petri dishes with invisible tape, a small piece on each side is sufficient.
- 5.15. Bring the tared filters to the Field Support Lab and place them in the tared media cabinet located in the chemical room.
- 5.16. Fill out the date and filter numbers on the media tracking form on the cabinet.

6.0 References-

- 6.1 EPA Method 5 - 40 CFR Part 60 Appendix A Method 5

7.0 Attachments-

- 7.1 Filter Tare Sheet
- 7.2 Tared Media Tracking Form

CAE STANDARD OPERATING PROCEDURE

PREPARATION OF TARED THIMBLES

ISO NUMBER: PFIL-8

EFFECTIVE DATE: Pending

AUTHOR: Dept. 64

APPROVED BY: Pending

1.0 Purpose- This procedure provides the steps for proper preparation and handling of tared thimbles for use in the Gravimetric analysis of particulate.

2.0 Scope- This procedure applies to thimbles tared in the CAE Gravimetrics Lab.

3.0 Materials-

- Powder free lab gloves
- Rubber Coated Beaker Tongs
- Thimble Tare Sheet
- Black Pen
- Desiccator
- Drierite (Calcium Sulfate)
- Four Place Analytical Balance
- Muffle Furnace
- Nalgene or Glass Sample Jars
- Kimwipes

5.0 Procedure -

5.1. Obtain clean thimbles from CAE's Field Support Lab.

5.2. Place the thimbles in the muffle furnace at about 750° C.

Note: The thimbles should be placed in the muffle furnace as early in the morning as possible. It takes a couple of hours to heat to the temperature desired. The thimbles should be heated at the desired temperature for at least two hours and then the muffle furnace should be turned off. Allow the thimbles to cool off in the furnace overnight.

5.3. Check the Drierite in the dessicator.

- a) If Drierite is blue/purple proceed to Step 5.4.
- b) If Drierite is pink, complete the procedure for "Changing and Regenerating Drierite" before proceeding.

5.4. Remove the thimbles from the muffle furnace and place them in the dessicator. Close the dessicator door and dessicate for no less than 24 hours before proceeding.

Note: Do not expose desiccated thimbles to the lab environment for more than 2 minutes. Keep in a dessicator until needed.

5.5. Calibrate the analytical balance (Ohaus GA200D). Complete the procedure for the "Calibration of Balances" before continuing.

5.6. Using beaker tongs, remove the thimble from the desiccator and place it on the center of the weight pan. Close the balance door and wait for the balance to stabilize.

5.7. Record the weight on the first line of the thimble tare sheet and fill in all the required information.

Note¹: Time can be recorded in 5 minute increments. Usually a number of thimbles can be weighed in this time period, so more than one thimble can have the same time.

Note²: Thimbles are usually identified by a letter and number combination. (examples: TH17, ST19, N364 etc.) If no identifying code is present write one on the thimble in marking ink or do not use that thimble.

5.8. Remove the thimble from the balance and return it to the dessicator.

5.9. Re-zero balance, if necessary. Repeat steps 6-8 until all thimbles have a weight.

5.10. Transfer all thimbles back into the dessicator. Close the dessicator door and dessicate for no less than six hours.

5.11. After dessicating for at least six hours, repeat Steps 5.5-5.10 until two consecutive weights are within ± 0.5 mg of each other.

5.12. Once the conditions in Step 5.11 are met, place each thimble into its own Nalgene or glass sample jar (lined with Kimwipes to protect it) and label the sample jar with the same number code that is on the thimble itself.

5.13. Repeat Step 5.12 until all the thimbles have been placed in sample jars..

5.14. Bring the tared thimbles to the Field Support Lab and place them in the tared media cabinet located in the chemical room.

5.15. Fill out the date and thimble numbers on the media tracking form on the cabinet.

6.0 References-

- 6.1 EPA Method 5 - 40 CFR Part 60 Appendix A Method 5
- 6.2 EPA Method 17 - 40 CFR Part 60 Appendix A Method 17

7.0 Attachments-

- 7.1 Thimble Tare Sheets
- 7.2 Tared Media Tracking Form

CAE STANDARD OPERATING PROCEDURE

GRAVIMETRIC ANALYSIS OF BENZENE SOLUBLE PARTICULATE
(8 X 10 FILTERS)

ISO NUMBER: PGRV-1

AUTHOR: Dept. 64

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1.0 Purpose- The purpose of this procedure is to ensure that anyone performing gravimetric analysis of benzene soluble particulate on 8 x 10 Filters, does so in an accurate, consistent manner.

2.0 Scope- This procedure applies to any employee performing the gravimetric analysis of benzene soluble particulate samples.

3.0 Laboratory Equipment and Glassware-

- Tared Weight Tins (tared to 0.01 mg)
- Teflon Benzene Squeeze Bottle
- Tube Heater
- Lab Gloves
- Tweezers
- Five Place Analytical Balance (Ohaus GA200D)
- Four Place Analytical Balance with 8 x 10 Filter Chamber (Mettler AJ100)
- Dessicator
- Vacuum Oven
- Vacuum Pump (Capable of Pulling 25 mm Hg)
- Fume Hood
- Ultrasonic Bath
- 60 mL Clear Glass Sample Jars (1 per Sample)
- Glass Manifold
- Syringes
- Syringe Filters (1 per Sample)
- Concentrator Tubes

- Disposable Pasteur Pipettes
- Polonium Anti-Static Strip
- Pipette Bulb
- Particulate Testing Weight Sheet

3.1 Chemicals-

- Benzene
- Nitrogen
- Drierite (Calcium Sulfate)

4.0 Definitions-

g - Abbreviation for gram

mL - Abbreviation for milliliter

5.0 Procedure-

- 5.1. Samples must be checked in. Complete the procedure for sample receiving before continuing.

5.2 Procedure 1 - Total Particulate

- 5.2.1. Place the filters into a dessicator.
- 5.2.2. Dessicate for 12 to 24 hours.
- 5.2.3. Calibrate the analytical balance. Complete the procedure for the calibration of the Mettler AJ100 analytical balance before continuing.
- 5.2.4. Using tweezers, remove the filter from the dessicator and hold it over the polonium anti-static strip for few seconds and then place the filter on the balance. Close the balance lid and wait for the balance to stabilize.
- 5.2.5. Record the weight on the particulate testing weight sheet.

5.3 Procedure 2 - Benzene Soluble Particulate

- 5.3.1. Using tweezers, place the filter in a labeled 60 mL sample container.
- 5.3.2. Using a syringe, cover the filter with 40 mL of benzene and cap the container.
- 5.3.3. Place the sample jar containing the filter in the ultrasonic bath. Sonicate for one hour.
- 5.3.4. Precondition a filtration unit by using nitrogen pressure to filter 1 mL of benzene through a syringe fitted with a syringe filter.
- 5.3.5. Using a disposable pipette, transfer all the benzene extract from the vial to the prerinsed syringe filtration unit.
- 5.3.6. Using nitrogen pressure, force the benzene extract through the syringe filtration unit into a concentrator tube.
- 5.3.7. Rinse the vial with 0.5 mL of benzene and pour the rinse into the syringe filtration unit.
- 5.3.8. Using nitrogen pressure, force the rinse through the syringe filtration unit into the concentrator tube.
- 5.3.9. Repeat steps 5.3.7 and 5.3.8.
- 5.3.10. Using a clean disposable pipette, rinse the syringe with 0.5 mL of benzene. Using nitrogen pressure, filter the rinse into the concentrator tube.
- 5.3.11. Place the concentrator tube in the tube heater at 85° C and reduce the total volume in the tube to 0.5 mL while sparging with nitrogen.
- 5.3.12. Transfer the remaining 0.5 mL of extract to a tared, labeled, aluminum weight tin. Rinse the concentrator tube with rinses of 0.2 mL, 0.2 mL and finally 0.1 mL of benzene and add them to the weight tin.
- 5.3.13. Place the weight tins in the vacuum oven (under the fume hood) at ambient temperature and 20-25 mm Hg vacuum pressure for 24 hours.
- 5.3.14. Calibrate the analytical balance. Complete the procedure for the calibration of the Ohaus GA110 analytical balance (five place mode) before continuing.
- 5.3.15. Using tweezers, remove the weight tin from the vacuum oven and place it on the analytical balance. Close the balance door and wait for the balance to stabilize.
- 5.3.16. Record the weight on the particulate testing weight sheet.

6.0 References

- 6.1 Benzene Soluble Compounds in Air - Physical and Chemical Analysis Branch - Analytical Method P&CAM 217
- 6.2 Standard Operating Procedure Benzene Solubles Method for Asphalt Institute Round Robin Study
- 6.3 National Institute of Occupational Safety and Health (NIOSH 5023, 3rd edition)
- 6.4 Occupational Safety and Health Administration (OSHA 58)

7.0 Attachments-

- 7.1 Particulate Testing Weight Sheet

CAE STANDARD OPERATING PROCEDURE

GRAVIMETRIC ANALYSIS OF BENZENE SOLUBLE PARTICULATE

ISO NUMBER: PGRV-2

AUTHOR: Dept. 64

EFFECTIVE DATE: Pending

APPROVED BY: Pending

GRAVIMETRIC ANALYSIS OF BENZENE SOLUBLE PARTICULATE

1.0 Purpose- The purpose of this procedure is to ensure that anyone performing gravimetric analysis of benzene soluble particulate does so in an accurate, consistent manner.

2.0 Scope- This procedure applies to any employee performing the gravimetric analysis of benzene soluble particulate samples.

3.0 Laboratory Equipment and Glassware-

- Tared Weight Tins (tared to 0.01 mg)
- Teflon Benzene Squeeze Bottle
- Tube Heater
- Lab Gloves
- Tweezers
- Five Place Analytical Balance
- Dessicator
- Vacuum Oven
- Vacuum Pump (Capable of Pulling 25 mm Hg)
- Fume Hood
- Ultrasonic Bath
- 7 mL Vials (1 per Sample)
- Glass Manifold
- Syringes

- Syringe Filters (1 per Sample)
- Concentrator Tubes
- Disposable Pasteur Pipettes
- Polonium Anti-Static Strip
- Pipette Bulb
- Particulate Testing Weight Sheet

3.1 Chemicals-

- Benzene
- Nitrogen
- Drierite (Calcium Sulfate)

4.0 Definitions-

g - Abbreviation for gram

mL - Abbreviation for milliliter

5.0 Procedure-

- 5.1. Samples must be checked in. Complete the procedure for sample receiving before continuing.

5.2 Procedure 1 - Total Particulate

- 5.2.1. Place the filter cassettes into a dessicator.
- 5.2.2. Dessicate for 12 to 24 hours.
- 5.2.3. Calibrate the analytical balance. Complete the procedure for the calibration of the Ohaus GA200D analytical balance (five place mode) before continuing.
- 5.2.4. Using tweezers, remove the filter from the filter cassette, hold it over the polonium anti-static strip for few seconds and then place the filter on the balance. Close the balance door and wait for the balance to stabilize.
- 5.2.5. Record the weight on the particulate testing weight sheet.

5.3 Procedure 2 - Benzene Soluble Particulate

- 5.3.1. Using tweezers, place the filter in a labeled 7 mL vial.
- 5.3.2. Using a syringe, cover the filter with 5 mL of benzene and cap the vial.
- 5.3.3. Place the vial containing the filter in the ultrasonic bath. Sonicate for one hour.
- 5.3.4. Precondition a filtration unit by using nitrogen pressure to filter 1 mL of benzene through a syringe fitted with a syringe filter.
- 5.3.5. Using a disposable pipette, transfer all the benzene extract from the vial to the prerinsed syringe filtration unit.
- 5.3.6. Using nitrogen pressure, force the benzene extract through the syringe filtration unit into a concentrator tube.
- 5.3.7. Rinse the vial with 0.5 mL of benzene and pour the rinse into the syringe filtration unit.
- 5.3.8. Using nitrogen pressure, force the rinse through the syringe filtration unit into the concentrator tube.
- 5.3.9. Repeat steps 5.3.7 and 5.3.8.
- 5.3.10. Using a clean disposable pipette, rinse the syringe with 0.5 mL of benzene. Using nitrogen pressure, filter the rinse into the concentrator tube.
- 5.3.11. Place the concentrator tube in the tube heater at 85° C and reduce the total volume in the tube to 0.5 mL while sparging with nitrogen.
- 5.3.12. Transfer the remaining 0.5 mL of extract to a tared, labeled, aluminum weight tin. Rinse the concentrator tube with rinses of 0.2 mL, 0.2 mL and finally 0.1 mL of benzene and add them to the weight tin.
- 5.3.13. Place the weight tins in the vacuum oven (under the fume hood) at ambient temperature and 20-25 mm Hg vacuum pressure for 24 hours.
- 5.3.14. Calibrate the analytical balance. Complete the procedure for the calibration of the Ohaus GA110 analytical balance (five place mode) before continuing.
- 5.3.15. Using tweezers, remove the weight tin from the vacuum oven and place it on the analytical balance. Close the balance door and wait for the balance to stabilize.
- 5.3.16. Record the weight on the particulate testing weight sheet.

6.0 References

- 6.1 Benzene Soluble Compounds in Air - Physical and Chemical Analysis Branch - Analytical Method P&CAM 217
- 6.2 Standard Operating Procedure Benzene Solubles Method for Asphalt Institute Round Robin Study
- 6.3 National Institute of Occupational Safety and Health (NIOSH 5023, 3rd edition)
- 6.4 Occupational Safety and Health Administration (OSHA 58)

7.0 Attachments-

- 7.1 Particulate Testing Weight Sheet

CAE STANDARD OPERATING PROCEDURE

**ANALYSIS OF CONDENSABLE PARTICULATE BY CHLOROFORM / ETHER
EXTRACTION**

ISO NUMBER: PGRV-3

EFFECTIVE DATE: Pending

AUTHOR: Dept. 64

APPROVED BY: Pending

1.0 Purpose- The purpose of this procedure is to ensure that any employee performing chloroform/ether extractions for the analysis of condensable particulate does so in an accurate, consistent manner.

2.0 Scope - This procedure applies to any employee performing the analysis of condensable particulate using chloroform / ether extractions. (This includes, but is not limited to Back half analysis of particulate in the following states: New Jersey, Iowa, Minnesota)

3.0 Materials - Laboratory Equipment-

- 1000 mL Separatory Funnel with Stopcock
- Two 100 mL Graduated Cylinders
- 250 mL Beakers (Two per Extraction)
- Ring Stand (Rod and Base)
- Disposable Pasteur Pipettes
- Pipette Bulb
- Ring Clamp
- Dessicator
- HDPE Squeeze Bottle (for De-ionized Water)
- 1000 mL Graduated Cylinder
- Gooch Crucible*
- Vacuum Flask*
- Pump*
- Hot Plate

- Lab Oven*
- Glass Petri Dishes*
- Tweezers*

3.1 Materials - Chemicals

- Chloroform
- Diethyl Ether
- De-ionized Water
- Tared 4.7 cm Glass Fiber Filters*
- Drierite (Calcium Sulfate)

* New Jersey Analysis Only

4.0 Definitions-

mL - Abbreviation for milliliters

5.0 Procedure-

- 5.0.1 If analyzing for particulate by New Jersey Method 5 omit procedure 5.3.
- 5.0.2 If analyzing for particulate by Minnesota Method 5 omit procedures 5.1 and 5.3.
- 5.0.3 If analyzing for particulate by Iowa Method 5 omit procedure 5.2.

5.1 Procedure 1 - Back Half Filtration (New Jersey Back Half Analysis)

- 5.1.1. Place a tared 4.7 cm glass fiber filter into the Gooch crucible. Place the Gooch Crucible on the vacuum flask and attach to the pump. Turn the pump on.
- 5.1.2. Carefully pour the sample through the filter. Rinse the sample container and pour the rinses through the filter.
- 5.1.3. After filtering, pour the sample back into the original container.
- 5.1.4. With tweezers, remove the filter from the Gooch crucible and place it on a glass petri dish.
- 5.1.5. Place the petri dish and filter in the lab oven and bake until dry.
- 5.1.6. After drying, remove petri dish and filter from oven and place in dessicator.
- 5.1.7 Follow the procedure for the gravimetric analysis of particulate.

5.2 Procedure 2 - Chloroform/Ether Extraction (New Jersey and Minnesota Back Half Analysis)

- 5.2.1. Measure the volume of the sample in a 1000 mL graduated cylinder. Record the volume on the data sheet.
- 5.2.2. Carefully pour the sample into a separatory funnel. (Make sure stopcock is closed!!!)
- 5.2.3. Add 25 mL of chloroform to the separatory funnel.
- 5.2.4. Place stopper in the top of the separatory funnel and shake for one minute. (Vent frequently by inverting the separatory funnel, with the stopper held in place, and rotating the stopcock.)
- 5.2.5. Remove stopper and wait for the liquids to separate; the chloroform will be the bottom layer.
- 5.2.6. Drain the bottom portion (chloroform) into a tared 250 mL beaker. Leave a very small amount of chloroform in the funnel to prevent water from getting in the beaker.
- 5.2.7. Repeat Steps 5.2.3 through 5.2.6 two more times.
- 5.2.8. Add 25 mL of diethyl ether to the separatory funnel.
- 5.2.9. Place stopper in the top of the separatory funnel and shake for one minute. (Vent frequently by inverting the funnel, with the stopper held in place, and rotating the stopcock.)
- 5.2.10. Remove the stopper and wait for the liquids to separate; the ether will be the top layer.
- 5.2.11. Drain the bottom portion (water) into the original sample container.
- 5.2.12. Drain the top layer (ether) into the same tared 250 mL beaker as the chloroform.
- 5.2.13. Pour the sample from the original container back into the separatory funnel.
- 5.2.14. Repeat Steps 5.2.8 through 5.2.13 two more times.
- 5.2.15. After extracting a total of six times (three chloroform, three ether), record the volume of the combined ether and chloroform on the data sheet. This is the organic fraction.
- 5.2.16. Place the organic fraction under a fume hood and evaporate to dryness at room temperature. (If water is present in the sample, remove it using a disposable Pasteur pipette. Water will appear as clear beads between the layers.)
- 5.2.17. The remaining liquid in the separatory funnel is the inorganic fraction. Shake the funnel and drain 100 mL of the fraction into a clean tared 250 mL beaker.
- 5.2.18. Place the beaker containing the inorganic fraction on a hot plate and evaporate to dryness. (Be careful not to allow the sample to boil.)
- 5.2.19. After samples are evaporated to dryness, place them in a dessicator.
- 5.2.20. Follow the procedure for the gravimetric analysis of particulate.

5.3 Procedure 3 - Ether/Chloroform Extraction (Iowa Back Half Analysis)

- 5.3.1. Measure the volume of the sample in a 1000 mL graduated cylinder. Record the volume on the data sheet.
- 5.3.2. Carefully pour the sample into a separatory funnel. (Make sure stopcock is closed!!!)
- 5.3.3. Add 25 mL of ether to the separatory funnel.
- 5.3.4. Place stopper in the top of the separatory funnel and shake for one minute. (Vent frequently by inverting the separatory funnel, with the stopper held in place, and rotating the stopcock.)
- 5.3.5. Remove stopper and wait for the liquids to separate; the ether will be the top layer.
- 5.3.6. Drain the bottom portion (water) into the original sample container.
- 5.3.7. Drain the top layer (ether) into a tared 250 mL beaker.
- 5.3.8. Pour the sample from the original sample container back into the separatory funnel
- 5.3.9. Repeat Steps 5.3.3 through 5.3.8 two more times.
- 5.3.10. Add 25 mL of chloroform to the separatory funnel.
- 5.3.11. Place stopper in the top of the separatory funnel and shake for one minute. (Vent frequently by inverting the funnel, with the stopper held in place, and rotating the stopcock.)
- 5.3.12. Remove the stopper and wait for the liquids to separate; the chloroform will be the bottom layer.
- 5.3.13. Drain the bottom layer (chloroform) into the same 250 mL beaker as the ether.
- 5.3.14. Repeat Steps 5.3.10 through 5.3.13 two more times.
- 5.3.15. After extracting a total of six times (three ether, three chloroform), record the volume of the combined ether and chloroform on the data sheet. This is the organic fraction.
- 5.3.16. Place the organic fraction under a fume hood and evaporate to dryness at room temperature. (If water is present in the sample, remove it using a disposable Pasteur pipette. Water will appear as clear beads between the layers.)
- 5.3.17. The remaining liquid in the separatory funnel is the inorganic fraction. Shake the funnel and drain 100 mL of the fraction into a clean, tared 250 mL beaker.
- 5.3.18. Place the beaker containing the inorganic fraction on a hot plate and evaporate to dryness. (Be careful not to allow the sample to boil.)
- 5.3.19. After samples are evaporated to dryness, place them in a dessicator.
- 5.3.20. Follow the procedure for the gravimetric analysis of particulate samples.

6.0 References-

- 6.1 Iowa Sampling Procedure for Particulates
- 6.2 Minnesota Performance Test Procedures, Exhibit C
- 6.3 New Jersey State Department of Environmental Protection, New Jersey Administrative Code, Title 7. Chapter 27B-1, Air Test Method 5

CAE STANDARD OPERATING PROCEDURE

ANALYSIS OF CONDENSABLE PARTICULATE BY DICHLOROMETHANE EXTRACTION
(GRAVIMETRIC)
METHOD 202 & WISCONSIN BACK HALF

ISO NUMBER: PGRV-4

AUTHOR: Dept. 64

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1.0 Purpose- The purpose of this procedure is to ensure that any employee performing dichloromethane extractions for the gravimetric analysis of back-half condensable particulate, does so in an accurate, consistent manner.

2.0 Scope- This procedure applies to any employee performing dichloromethane extractions for the analysis of back-half condensable particulate. This procedure includes, but is not limited to, the analysis sections of EPA Method 202 and Wisconsin DNR Method 5.

3.0 Materials - Laboratory Equipment and Glassware-

- 1000 mL Separatory Funnel
- 100 mL Graduated Cylinder
- 250 mL Beakers (Two per Extraction)
- Disposable Pasteur Pipettes
- Pipette bulb
- 1000 mL Graduated Cylinder
- Ring stand (Rod and Base)
- Ring Clamp
- Teflon Squeeze Bottle for MeCl_2
- pH paper (0-7 Range)
- HDPE Squeeze Bottle for D.I. Water
- Dessicator

3.1 Materials - Chemicals-

- Dichloromethane (MeCl_2)
- De-ionized water
- Phenolphthalein (1% into 60% isopropanol)
- Ammonium Hydroxide
- Drierite (Calcium Sulfate)

4.0 Definitions-

MeCl_2 - Abbreviation for dichloromethane (methylene chloride)

mL - Abbreviation for milliliters

5.0 Procedure-

Note:: If analyzing back half samples according to Wisconsin Method 5, use volumes indicated in parenthesis following the volumes used for EPA Method 202. Also omit steps with an asterisk (*) (5.1, 5.13, 5.14 and 5.15).

- *5.1. Measure the pH of the sample.
 - a) If the pH is greater than 4.5 proceed to step 5.2.
 - b) If the pH is less than 4.5, remove a 50 mL aliquot and give to the Ion Chromatography Lab for Sulfate analysis before proceeding to step 5.2..
- 5.2. Using the 1000 mL graduated cylinder, measure the volume of the sample. Record it on the data sheet.
- 5.3. Carefully pour the sample into the separatory funnel. (Make sure stopcock is closed!!!!!!)
- 5.4. Add 75 mL (50 mL) of MeCl_2 to the separatory funnel.
- 5.5. Place stopper in the top of the funnel and shake for about one minute. (Vent frequently by inverting the separatory funnel, with the stopper held in place, and rotating the stopcock).
- 5.6. Remove stopper and wait for liquids to separate; the MeCl_2 will be the bottom layer.
- 5.7. Drain bottom portion of liquid into a tared 250 mL beaker. Leave a very small amount of MeCl_2 in the funnel to prevent water from getting into the beaker. This is the organic fraction.
- 5.8. Repeat steps 5.4 through 5.7 two more times.
- 5.9. After extracting with MeCl_2 three times, record the volume of the organic fraction on the data sheet and place beaker under hood to evaporate at room temperature. (If water is present in the sample, remove it using a disposable Pasteur Pipette. Water will appear as clear beads floating on top of the dichloromethane.)

- 5.10. After samples are evaporated to dryness, place them in a dessicator.
- 5.11. Follow the procedure for the gravimetric analysis of particulate samples.
- 5.12. The remaining liquid in the separatory funnel is the inorganic fraction. Shake the funnel and drain 100 mL of the inorganic fraction into a separate tared beaker.
- *5.13. If the pH of the sample in step 5.1 was less than 4.5, proceed to step 5.12. If it was greater than 4.5, proceed to step 5.14.
- *5.14. Add 3 to 4 drops of phenolphthalein solution to the inorganic fraction.
- *5.15. Add ammonium hydroxide drop wise to the solution until it turns pink.
- 5.16. Place beaker on hot plate and evaporate to dryness (be careful not to allow sample to boil).
- 5.17. After the samples are evaporated to dryness, place them in a dessicator.
- 5.18. Follow the procedure for the gravimetric analysis of particulate samples.

6.0 References-

- 6.1 EPA Method 202
- 6.2 Wisconsin Air Management Operations Handbook, Modified Method 5 Test Method for Condensible Particulate

Leave a small amount of MeCl_2 in the sep. funnel to ensure that no H_2O is collected in the organic phase. Place the organic extract at room temperature and pressure under a hood.

Following total evaporation, desiccate for 24 hours in a desiccator containing Anhydrous Calcium Sulfate. Weigh to a constant, with a minimum of 6 hours between weightings, and report the results to the nearest 0.1 mg.

NOTE: If NH_4Cl is to be counted as CPM the inorganic fraction should be taken to near dryness (less than 1 ml liquid) in the oven and then allowed to air dry at ambient temperature. If multiple acid emissions are suspected, the ammonia titration procedure, described later, may be preferred.

Redissolve the residue in 100 ml of DI water.

Add five drops of phenolphthalein; then add concentrated (14.8M) NH_4OH to a pink end point. Any excess NH_4OH will evaporate in the final drying.

Evaporate the sample to dryness in a vented 105 °C oven, desiccate for 24 hours, weigh to a constant, and record results to the nearest 0.1 mg

REMINDER: The addition of NH_4OH is recommended, but is optional when little or no SO_2 is present in the gas stream. When the impinger solution has a pH greater than 4.5, the addition of NH_4OH is not necessary. N_2 Purge = NH_4OH addition.

Analysis of Sulfate of IC to Determine Ammonium Ion (NH_4^+) Retained in the Sample.

If NH_4OH is not added to the sample, omit this step

1. Determine the amount of sulfate in the 5 m; aliquot previously taken.
2. Based on the result of the IC analysis, calculate the correction factor to subtract the NH_4^+ remaining in the sample, and add the water removed by the acid-base reaction.

Analysis of MeCl_2 and DI water Blanks

Analyze these blanks the same as you would the samples. Use the blank MeCl_2 to extract the blank DI water.

Analysis of the Acetone blank

Place 100 ml of the blank acetone in a tared beaker, evaporate, desiccate and weigh to a constant.

CAE STANDARD OPERATING PROCEDURE

GRAVIMETRIC ANALYSIS OF PARTICULATE
EPA METHODS 5, 17, 201A

ISO NUMBER: PGRV-5

AUTHOR: Dept. 64

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1.0 Purpose- The purpose of this procedure is to ensure that anyone performing gravimetric analysis of particulate does so in an accurate, consistent manner.

2.0 Scope- This procedure applies to any employee performing the gravimetric analysis of particulate samples collected using EPA Methods 5, 17, 201A.

3.0 Laboratory Equipment and Glassware-

- Tared 250 mL Beakers
- Teflon Acetone Squeeze Bottle
- Beaker Tongs
- Lab Gloves
- Tweezers
- Four Place Analytical Balance
- Particle Brush Dessicator
- Hot Plate
- Fume Hood
- Particulate Testing Weight Sheet

3.1 Chemicals-

- Acetone
- Drierite (Calcium Sulfate)

4.0 Definitions-

g - Abbreviation for gram

5.0 Procedure-

Note: At no time should gravimetric samples be handled with anything but tweezers or beaker tongs. Oils and even heat from your hands can interfere with weights.

- 5.1. Samples must be checked in. Complete the procedure for sample receiving before continuing.

5.2 Procedure 1 - Thimbles

- 5.2.1. Remove thimbles from sample container and carefully place them into a dessicator.
- 5.2.2. Dessicate thimbles for no less than 24 hours.
- 5.2.3. Calibrate the analytical balance. Complete the procedure for the calibration of the Ohaus GA200D analytical balance before continuing.
- 5.2.4. Using beaker tongs, place the thimble on the balance. Close the balance door and wait for the balance to stabilize.
- 5.2.5. Record the weight on the particulate testing weight sheet.
- 5.2.6. Carefully place the thimble back in the dessicator.
- 5.2.7. Dessicate for no less than six hours.
- 5.2.8. Repeat Steps 5.2.4 through 5.2.8 until two consecutive weights are within ± 0.5 mg (or 1% of the total weight) of each other.

5.3 Procedure 2 - Filters

- 5.3.1. Filters should be received in sealed petri dishes. Remove the Teflon tape and the cover of the petri dish so that the filter is exposed, but is still in the bottom of the petri dish..
- 5.3.2. Place the filter in a dessicator.
- 5.3.3. Dessicate for no less than 24 hours.
- 5.3.4. Calibrate the analytical balance. Complete the procedure for the calibration of the Ohaus GA110 analytical balance before continuing.
- 5.3.5. Using tweezers, remove the filter from the petri dish and place the filter on the analytical balance. Brush any loose particulate from the petri dish onto the filter. Close the balance door and wait for the balance to stabilize.
- 5.3.6. Record the weight on the particulate testing weight sheet.
- 5.3.7. Carefully place the filter back into the petri dish and then back into the dessicator.
- 5.3.8. Dessicate for no less than six hours.

5.3.9. Repeat steps 5.3.5 through 5.3.8 until two consecutive weights are within ± 0.5 mg (or 1% of the total weight) of each other.

5.4 Procedure 3 - Front Half Rinses

Note: Usually front half rinses will be acetone, however, water may be used instead. If this is the case, follow the procedure substituting water for acetone.

5.4.1. Carefully pour the sample into a tared 250 mL beaker.

5.4.2. Rinse the sample container three times with acetone. Add the rinses to the sample in the beaker. Record the volume.

5.4.3. Using beaker tongs or lab gloves, place the beaker on a hot plate. Evaporate to dryness. **Do not let the sample boil. If the sample starts to boil, lower the heat.**

5.4.4. Once the sample is evaporated, place it in a dessicator.

5.4.5. Dessicate for no less than 24 hours.

5.4.6. Calibrate the analytical balance. Complete the procedure for the calibration of the Ohaus GA200D analytical balance before continuing.

5.4.7. Using beaker tongs, place the beaker on the balance. Close the balance door and wait for the balance to stabilize.

5.4.8. Record the weight on the particulate testing weight sheet.

5.4.9. Return the beaker to the dessicator.

5.4.10. Dessicate for no less than six hours.

5.4.11. Repeat Steps 5.4.7 through 5.4.10 until two consecutive weights are within ± 0.5 mg (or 1% of the total weight) of each other.

6.0 References

6.1 40 CFR Part 60 Appendix A Method 5

6.2 40 CFR Part 60 Appendix A Method 17

6.3 EPA Method 201A

7.0 Attachments-

7.1 Particulate Testing Weight Sheet

CAE STANDARD OPERATING PROCEDURE

**GRAVIMETRIC ANALYSIS OF PARTICULATE
EPA METHOD 5A**

ISO NUMBER: PGRV-6

AUTHOR: Dept. 64

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1.0 Purpose- The purpose of this procedure is to ensure that anyone performing gravimetric analysis of particulate by EPA Method 5A does so in an accurate, consistent manner.

2.0 Scope- This procedure applies to any employee performing the gravimetric analysis of particulate samples collected using EPA Method 5A.

3.0 Laboratory Equipment and Glassware-

- Tared 250 mL Beakers
- 100 mL Graduated Cylinder
- 1000 mL Separatory Funnel
- Teflon TCE Squeeze Bottle
- Beaker Tongs
- Tweezers
- Four Place Analytical Balance
- Particle Brush
- Dessicator
- Hot Plate
- Fume Hood
- Lab Gloves
- Particulate Testing Weight Sheet

3.1 Chemicals-

- 1,1,1 trichloroethane
- Drierite (Calcium Sulfate)

4.0 Definitions-

g - Abbreviation for gram

TCE - Abbreviation for 1,1,1 trichloroethane

5.0 Procedure-

Note: At no time should gravimetric samples be handled with anything but tweezers or beaker tongs. Oils and even heat from your hands can interfere with weights.

- 5.1. Samples must be checked in. Complete the procedure for sample receiving before continuing.

5.2 Procedure 1 - Filters

- 5.2.1. Filters should be received in sealed glass petri dishes. Remove the filter from the glass dish and place it in a clean, labeled petri dish.
- 5.2.2. Place the filter in a dessicator.
- 5.2.3. Dessicate for no less than 24 hours.
- 5.2.4. Calibrate the analytical balance. Complete the procedure for the calibration of the Ohaus GA110 analytical balance before continuing.
- 5.2.5. Using tweezers, remove the filter from the petri dish and place the filter on the analytical balance. Brush any loose particulate from the petri dish onto the filter. Close the balance door and wait for the balance to stabilize.
- 5.2.6. Record the weight on the particulate testing weight sheet.
- 5.2.7. Carefully place the filter back into the petri dish and then back into the dessicator.
- 5.2.8. Dessicate for no less than 24 hours.
- 5.2.9. Repeat steps 5.2.4 through 5.2.8 until two consecutive weights are within ± 2.0 mg (or 10% of the total weight) of each other.

5.3 Procedure 2 - Front Half Rinses

- 5.3.1. Carefully pour the front half rinse into a tared 250 mL beaker.
- 5.3.2. Rinse the sample container three times with TCE. Add the rinses to the sample in the beaker.
- 5.3.3. Rinse the original glass petri dish from the filter (in step 5.2.1) three times with TCE and combine the filter container rinse with the corresponding front half rinse in the beaker. Record the volume.
- 5.3.4. Check to see if any water is present in the rinse. (Look for a boundary layer which indicates the separation of two liquids.)
 - a) If the amount of water is 5.0 milliliters or less, proceed to step 5.3.10 .
 - b) If the amount of water is greater than 5.0 milliliters proceed to the next step (5.3.5).
- 5.3.5. Pour the TCE/water Rinse into a separatory funnel.
- 5.3.6. Add 25 milliliters of fresh TCE to the funnel and shake for one minute.
- 5.3.7. Allow the liquids to settle. After the boundary layer has appeared, drain off the TCE (bottom) layer into a tared 250 mL beaker.
- 5.3.8. Repeat steps 5.3.6 through 5.3.7 until no visible organic matter remains in the water.
- 5.3.9. Drain the remaining water into a separate tared beaker.
- 5.3.10. Using beaker tongs or lab gloves, place the beaker containing the water fraction on a hot plate. Evaporate to dryness at no more than 200° F. **Do not let the sample boil. If the sample starts to boil, lower the heat.**
- 5.3.11. Using beaker tongs or lab gloves, place the beaker containing the TCE fraction on the hot plate. Evaporate to dryness at no more than 100° F. **Do not let the sample boil. If the sample starts to boil, lower the heat.**
- 5.3.12. Once the samples are evaporated, place them in a dessicator.
- 5.3.13. Dessicate for no less than 24 hours.
- 5.3.14. Calibrate the analytical balance. Complete the procedure for the calibration of the Ohaus GA200D analytical balance before continuing.
- 5.3.15. Using beaker tongs, place the beaker on the balance. Close the balance door and wait for the balance to stabilize.
- 5.3.16. Record the weight on the particulate testing weight sheet.
- 5.3.17. Place the beaker back into the dessicator.

5.3.18. Dessicate for no less than 24 hours.

5.3.19. Repeat Steps 5.3.14 through 5.3.18 until two consecutive weights are within ± 2.0 mg (or 10% of the total weight) of each other.

6.0 References

6.1 40 CFR Part 60 Appendix A Method 5A

7.0 Attachments-

7.1 Particulate Testing Weight Sheet

CAE STANDARD OPERATING PROCEDURE

GRAVIMETRIC ANALYSIS OF NON-SULFURIC ACID PARTICULATE
EPA METHOD 5B

ISO NUMBER: PGRV-7

AUTHOR: Dept. 64

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1.0 Purpose- The purpose of this procedure is to ensure that anyone performing gravimetric analysis of non-sulfuric acid particulate does so in an accurate, consistent manner.

2.0 Scope- This procedure applies to any employee performing the gravimetric analysis of particulate samples collected using EPA Method 5B.

3.0 Laboratory Equipment and Glassware-

- Tared 250 mL Beakers
- Glass Petri Dishes
- Teflon Acetone Squeeze Bottle
- Beaker Tongs
- Lab Gloves
- Tweezers
- Four Place Analytical Balance
- Particle Brush Dessicator
- Lab Oven
- Fume Hood
- Particulate Testing Weight Sheet

3.1 Chemicals-

- Acetone
- Drierite (Calcium Sulfate)

4.0 Definitions-

g - Abbreviation for gram

5.0 Procedure-

Note: At no time should gravimetric samples be handled with anything but tweezers or beaker tongs. Oils and even heat from your hands can interfere with weights.

- 5.1. Samples must be checked in. Complete the procedure for sample receiving before continuing.

5.2 Procedure 1 - Filters

- 5.2.1. Filters should be received in sealed petri dishes. Remove the teflon tape and the cover of the petri dish, and, using tweezers, transfer the filter into a labeled glass petri dish. Brush any loose particulate from the original dish onto the filter.
- 5.2.2. Place the filter in the lab oven.
- 5.2.3. Dry the filter in the oven at 320° F for six hours.
- 5.2.4. Place the filter in a dessicator.
- 5.2.5. Dessicate the filter for two hours.
- 5.2.6. Calibrate the analytical balance. Complete the procedure for the calibration of the Ohaus GA110 analytical balance before continuing.
- 5.2.7. Using tweezers, remove the filter from the petri dish and place the filter on the analytical balance. Brush any loose particulate from the petri dish onto the filter. Close the balance door and wait for the balance to stabilize.
- 5.2.8. Record the weight on the particulate testing weight sheet.
- 5.2.9. Carefully place the filter back into the petri dish and then back into the dessicator.
- 5.2.10. Dessicate for no less than six hours.
- 5.2.11. Repeat steps 5.2.7 through 5.2.10 until two consecutive weights are within ± 0.5 mg (or 1% of the total weight) of each other.

5.3 Procedure 2 - Front Half Rinses

Note: Usually front half rinses will be acetone, however, water may be used instead. If this is the case, follow the procedure substituting water for acetone.

- 5.3.1. Carefully pour the sample into a tared 250 mL beaker.
- 5.3.2. Rinse the sample container three times with acetone. Add the rinses to the sample in the beaker. Record the volume.
- 5.3.3. Using beaker tongs or lab gloves, place the beaker under the fume hood. Evaporate to dryness at ambient temperature.
- 5.3.4. Once the sample is evaporated, place it in the lab oven at 320° F for six hours.
- 5.3.5. Place the beaker in a dessicator.
- 5.3.6. Dessicate for two hours.
- 5.3.7. Calibrate the analytical balance. Complete the procedure for the calibration of the Ohaus GA200D analytical balance before continuing.
- 5.3.8. Using beaker tongs, place the beaker on the balance. Close the balance door and wait for the balance to stabilize.
- 5.3.9. Record the weight on the particulate testing weight sheet.
- 5.3.10. Return the beaker to the dessicator.
- 5.3.11. Dessicate for no less than six hours.
- 5.3.12. Repeat Steps 5.3.8 through 5.3.11 until two consecutive weights are within ± 0.5 mg (or 1% of the total weight) of each other.

6.0 References

- 6.1 40 CFR Part 60 Appendix A Method 5B

7.0 Attachments-

- 7.1 Particulate Testing Weight Sheet

CAE STANDARD OPERATING PROCEDURE

GRAVIMETRIC ANALYSIS OF NON-SULFATE PARTICULATE
EPA METHOD 5F

ISO NUMBER: PGRV-8

AUTHOR: Dept. 64

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1.0 Purpose- The purpose of this procedure is to ensure that anyone performing gravimetric analysis of non-sulfate particulate does so in an accurate, consistent manner.

2.0 Scope- This procedure applies to any employee performing the gravimetric analysis of non-sulfate particulate samples collected using EPA Method 5F.

3.0 Laboratory Equipment and Glassware-

- Tared 250 mL Beakers (1 Per Sample)
- HDPE De-ionized Water Squeeze Bottle
- Beaker Tongs
- Lab Gloves
- Tweezers
- Four Place Analytical Balance
- Particle Brush
- Dessicator
- Hot Plate
- Fume Hood
- 5 mL Pipettes
- Pipette Bulb
- 50 mL Volumetric Flask (1 Per Sample)
- 500 mL Volumetric Flask (1 Per Sample)
- 125 mL Erlenmeyer Flask (1 Per Sample)

- Glass Cold Finger Condenser (1 Per Sample)
- Air Manifold
- Scissors (Razor Blade)
- Method 5F Particulate Testing Weight Sheet

3.1 Chemicals-

- De-ionized Water
- Phenolphthalien Indicator Solution
- Ammonium Hydroxide
- Drierite (Calcium Sulfate)

4.0 Definitions-

g - Abbreviation for gram

5.0 Procedure-

Note: At no time should gravimetric samples be handled with anything but tweezers or beaker tongs. Oils and even heat from your hands can interfere with weights.

- 5.1. Samples must be checked in. Complete the procedure for sample receiving before continuing.
- 5.2. Cut the filter into small pieces.
- 5.3. Place the pieces of the filter into a 125 mL Erlenmeyer flask.
- 5.4. Rinse the filter container three times with de-ionized water. Add the rinses to the pieces of filter in the Erlenmeyer flask.
- 5.5. Add de-ionized water to the Erlenmeyer flask until the total volume in the flask is 75 milliliters.
- 5.6. Place the cold finger condenser into the flask. (Note: It may be necessary to seal the seam between the flask and the condenser with teflon tape.)
- 5.7. Reflux the flask for six hours at 105° C.
- 5.8. After the filter has refluxed for six hours, remove it from the hot plate and transfer all the filter pieces, particulate and water to a 500 mL volumetric flask.

- 5.9. Add the front half water rinse to the flask. Rinse the sample container three times with de-ionized water and add the rinses to the flask.
- 5.10. Add de-ionized water to the combined front half rinse and filter in the flask until the volume is exactly 500 mL. Allow the particulate and pieces of filter to settle to the bottom of the flask.
- 5.11. Pipette a 5 mL aliquot from the flask and transfer it to a 50 mL volumetric flask. Dilute to the 50 mL mark with de-ionized water.
- 5.12. Give the 50 mL volumetric flask to the Ion Chromatography laboratory for sulfate analysis.
- 5.13. Pour the remainder of the sample into a tared 250 mL beaker. In order to get the entire sample into the same beaker, allow portions of the sample to evaporate and then add more of the sample to the beaker until it is all in the beaker.
- 5.14. Evaporate the sample down to 100 milliliters on the hot plate, be sure that the sample does not boil.
- 5.15. Once the sample is down to 100 mL, remove it from the hot plate and allow it to cool.
- 5.16. Add six drops of phenolphthalien indicator to the sample.
- 5.17. Add ammonium hydroxide drop wise until the sample turns pink.
- 5.18. Return the sample to the hot plate and evaporate to dryness.
- 5.19. Once the sample is evaporated, place it in a dessicator.
- 5.20. Dessicate for no less than 24 hours.
- 5.21. Calibrate the analytical balance. Complete the procedure for the calibration of the Ohaus GA200D analytical balance before continuing.
- 5.22. Using beaker tongs, place the beaker on the balance. Close the balance door and wait for the balance to stabilize.
- 5.23. Record the weight on the particulate testing weight sheet.
- 5.24. Place the beaker back into the dessicator.
- 5.25. Dessicate for no less than six hours.
- 5.26. Repeat Steps 5.22 through 5.25 until two consecutive weights are within ± 0.5 mg (or 1% of the total weight) of each other.

6.0 References

- 6.1 40 CFR Part 60 Appendix A Method 5F

7.0 Attachments-

7.1 Method 5F Particulate Testing Weight Sheet

EPA M5 GRAVIMETRIC ANALYSIS

ISO NUMBER: LAB-5

EFFECTIVE DATE: Pending

AUTHOR: Dept. 68

APPROVED BY: Pending

1. PURPOSE

The purpose of this procedure is to provide detailed information for performing "in-house" analysis of EPA Method 5 samples.

2. SCOPE

This procedure should be followed by all individuals bringing field samples into the laboratory area and all laboratory technicians performing gravimetric analysis.

3. MATERIALS

The following materials are needed to properly execute this procedure

- Client Weight Sheets
- Chain of Custody
- Tared particulate prepped beakers
- Desiccator
- Tweezers
- Tongs
- Clip board
- Acetone
- Hood
- Hot Plate
- Rubber policeman/ spatula
- Squeeze bottle

4. DEFINITIONS

- 4.1 "In House" analysis: Any analytical procedure that takes place in the Pittsburgh laboratory
- 4.2 Gravimetric: Quantitative analysis based on weights
- 4.3 Chain of Custody: Paperwork that identifies samples and accompanies samples whenever they are relinquished to another party
- 4.4 Client Weight Sheet: Data sheet which contains all sample identification information including sample type, run number, location, ID numbers, sample volumes, gross weights, tare weights, and net weights
- 4.5 Tared: Sample containers, (beakers and thimbles), or filters that have been weighed twice; with consecutive weighings differing by no more than 0.0005g

- 4.6 Reconstitute: Returning an evaporated sample back into a solution in which it originally existed.
- 4.7 Gross weight: The total weight of a sample, including the weight of any container in which the sample is contained
- 4.8 Net weight: The gross weight minus the tare weight
- 4.9 Archive shelves: The shelves against the back wall of the lab which are labeled with the months of the year

5. PROCEDURE

- 5.1 Arrival of Samples (The following procedures in this section are the responsibility of the person bringing the samples into the laboratory).
 - 5.1.1 Place samples on center island in laboratory.
 - 5.1.2 Identify samples with Job number, Client name, and corresponding chain of custody if available.

Job # _____
Client name: _____
Analysis requested: _____
Job leader: _____
Project Manager: _____
Hold samples for further analysis: yes <input type="checkbox"/> no <input type="checkbox"/>
(If no, then reconstitute.)

Figure 1 - Initial sample identification label example

- 5.1.3 If samples are to be held for further analysis after gravimetric analysis, note these instructions now.
- 5.2 Organization of Samples
 - 5.2.1 Organize samples by location and run number.
 - 5.2.2 Place coordinating samples together, i.e., Unit X run x filter, front half acetone wash, and thimble (if applicable) should be placed together.
- 5.3 Chain of Custody
 - 5.3.1 Match all samples with accompanying chain of custody forms.
 - 5.3.2 If no chain of custody exist, the lab technician should create a custody for each sample.

5.4 Client Weight Sheets

- 5.4.1 Create a client weight sheet for each job. These weight sheets should be kept in the Gravimetric lab folder on the server. All client weight sheets should be named using a consistent naming convention. This naming convention should reference the job number and method, followed by the word "weights". All American Ref-Fuel weights should be placed in a separate folder for easy reference.

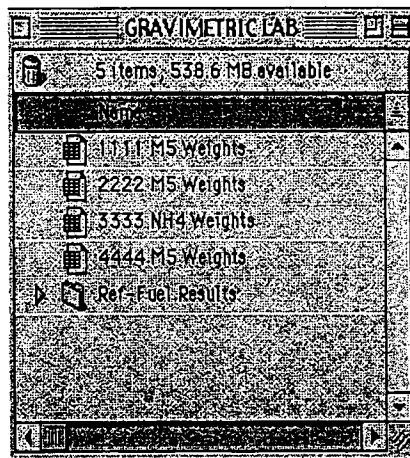


Figure 2 - Weight sheet naming convention example

- 5.4.2 Within each spreadsheet, create separate worksheets to coincide with each location tested.
- 5.4.3 Create weight sheets to coincide with samples, i.e., list each fraction, location, and run number in specified area.

Weight Sheet							
Client		abc	Project Number		XXXX	Analyst	
Plant		a	Unit		Sack	Balance	
Test Date			Method		EPA 5	Fisher 16191	
Description	Lab. and Sample Description	Sample Volume (ml)	Date/Time	Grav. Weight (g)	Net Weight (g)	Date/Time	Net Weight (g)
Type Filter	Yellow residue Q0012	Filter	2/20 11:08	0.4353	0.3653	7/2/97	
Run 1			2/23 08:32	0.4340			
Location Sack			2/23 15:02	0.4339			0.0486
Type F1/2 Acetone	20-150	120 Beaker	2/20 11:20	65.8281	65.8143	1/30/96	
Run 1			2/23 08:36	65.8300			
Location Sack			2/23 15:09	65.8299			0.0156
Type Thimble	TH3	Beaker	2/20 11:12	0.3861	0.3850	7/2/97	
Run 1			2/23 08:33	0.3853			
Location Sack			2/23 15:03	0.3853			0.0003
Type F1/2 Acetone	18-150	100 Beaker	2/20 11:22	66.3365	66.3336	1/30/96	
Run 2			2/23 08:34	66.3375			
Location Sack			2/23 15:08	66.3375			0.0039
Type Filter	Yellow residue Q0015	Filter	2/20 11:10	0.4039	0.4031	7/2/97	
Run 2			2/23 08:33	0.4028			
Location Sack			2/23 15:03	0.4030			-0.0001

Figure 3 - Worksheet set-up example

5.5 Filters

- 5.5.1 Record each filter's ID number on client weight sheet in appropriate column along side it's location and run number.
- 5.5.2 Place filters in desiccator.
- 5.5.3 Attach note to desiccator door to identify samples.

Job # _____	
Client Name: _____	
Sample fraction: _____	
(date/time)	Done
1st Weight available @ _____	_____
2nd Weight available @ _____	_____

Figure 4 - Note for desiccator example

Note: This (date/time) indicates when weighing may take place.
Check **Done** box when weighing has actually occurred.

- 5.5.4 Perform 1st gross weighing of filter.
- 5.5.5 Perform 2nd gross weighing of filter.
- 5.5.6 Remove filters from desiccator and place on appropriate archive shelf.
- 5.5.7 Archive filters for 6 months.

5.6 Thimbles

- 5.6.1 Record each thimble ID number on Client weight sheet in appropriate column along side it's location and run number.
- 5.6.2 Place thimbles into desiccator
- 5.6.3 Attach note to desiccator door identifying samples.
 - 5.6.3.1 Refer to Figure 4 - Note for desiccator example.
- 5.6.4 Perform 1st gross weighing of thimble.
- 5.6.5 Perform 2nd gross weighing of thimble.
- 5.6.6 Remove thimbles from desiccator.
- 5.6.7 Comfirm with job leader or Project Manager if particulate in thimbles should be saved.
 - 5.6.7.1 If yes, brush loose particulate from sides of thimble and place in sample bottle.
- 5.6.8 If no, thimbles may be cleaned, baked and retared.

5.7 Front half Acetone Samples

- 5.7.1 Obtain tared beakers. (Choose beaker size compatible with sample volume).

- 5.7.1.1 Tared beakers are kept in the plexiglass box on the shelf in the weighing room
- 5.7.1.2 Extra tared beakers are stored wrapped in syran wrap or foil in a labeled box in the weighing room.
- 5.7.2 Match one beaker to each front half acetone sample.
- 5.7.3 Write the beaker ID number on the sample jar that you are matching it with.
- 5.7.4 Record this beaker ID number on the client weight sheet beside the appropriate sample location and run.
- 5.7.5 Arrange the beakers on the hot plate under the hood in logical order i.e., place samples from the same location together or arrange sequential runs together.
- 5.7.6 Turn on hood fan.
- 5.7.7 Carefully and quantitatively, transfer the acetone samples from the jars to the coordinating beakers.
- 5.7.8 Record the volume of the sample in the beaker on the client weight sheet.
- 5.7.9 After emptying all samples into beakers and recording ID numbers and volumes, turn on the hot plate. Set initial temperture dial to 80-100° C. Watch the beakers and adjust temperature as necessary to ensure that the samples do not boil. **SAMPLES SHOULD NEVER BE ALLOWED TO BOIL.** Adjust temperature accordingly.
- 5.7.10 Attach note to hood to identify samples.

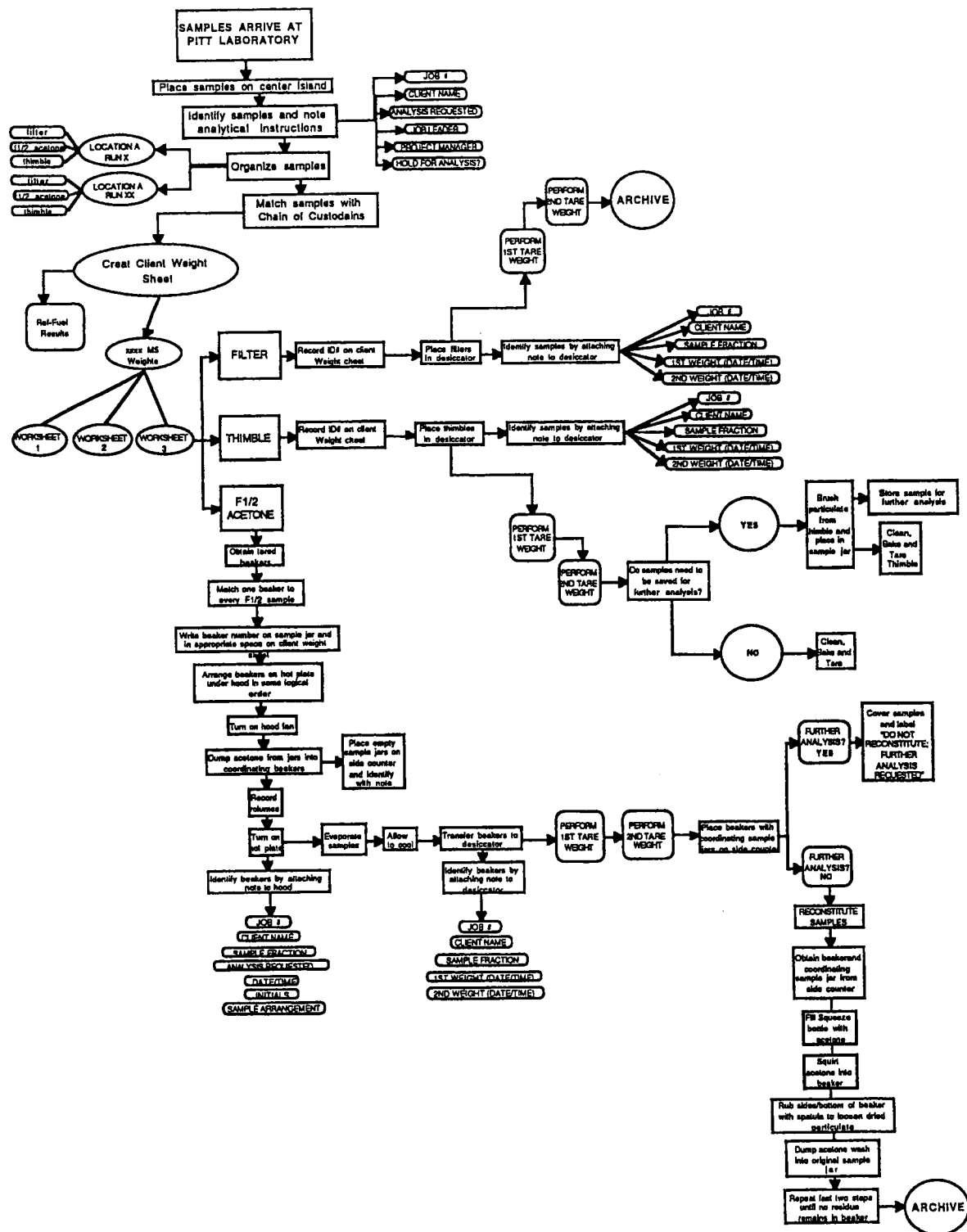
Job # _____	
Client name: _____	
Sample fraction: _____	
Analysis requested: _____	
Date/Time: _____	
Initial: _____	
Sample arrangement:	

Figure 5 - Note for hood example

- 5.7.11 Place empty sample jars on side counter with a note identifying them. (See Figure 1 - Initial sample identificaiton label example). (Be sure to note whether samples should be reconstituted or held for further analysis. Check with job leader or project manager to confirm these instructions.
- 5.7.12 When front half acetone samples are completely evaporated, turn hot plate off and allow beakers to cool.
- 5.7.13 Transfer beakers to the desiccator.

- 5.7.13.1 Remember not to touch beakers with bare hands.
 - 5.7.13.2 Wearing gloves or using tongs, place beakers onto clipboard and carefully carry them into weighing room.
 - 5.7.14 Arrange beakers in desiccator in a logical order, i.e., place unit A samples together, unit B samples together, to simplify weighing process.
 - 5.7.15 Place note on desiccator door to identify samples. (See Figure 4).
 - 5.7.16 Perform 1st gross weighing
 - 5.7.17 Perform 2nd gross weighing.
 - 5.7.18 Remove beakers from desiccator and place on side counter with coordinating sample jar.
 - 5.7.19 Confirm with job leader or Project Manager if further analysis is requested or if samples can be reconstituted if this has not already been established.
 - 5.7.20 If further analysis is requested, cover beakers with watch glasses or syran wrap so that no foreign particulate contaminates samples.
 - 5.7.21 Leave note with samples with instructions, "DO NOT RECONSTITUTE; FURTHER ANALYSIS REQUESTED".
 - 5.7.22 If only gravimetric analysis is required, samples may be reconstituted.
- 5.8 Reconstituting Samples
- 5.8.1 Obtain beaker and coordinating sample jar from the side counter in the lab.
 - 5.8.2 Fill squeeze bottle with acetone.
 - 5.8.3 Squirt approximately 10 ml of acetone into beaker.
 - 5.8.4 With rubber policeman or Teflon spatula, rub sides and bottom of beaker to loosen dried particulate.
 - 5.8.5 Carefully and quantitatively, transfer acetone wash into original sample jar.
 - 5.8.6 Repeat this process until beaker is thoroughly cleaned and no visible particulate remains in beaker.
 - 5.8.7 Place jar with reconstituted sample on appropriate archive shelf in the lab.
 - 5.8.8 Archive for six months.

6. PROCESS FLOW DIAGRAM



7. ATTACHMENTS

EPA M5/202 GRAVIMETRIC ANALYSIS

ISO NUMBER: LAB-6.

EFFECTIVE DATE: Pending

AUTHOR: Dept. 68

APPROVED BY: Pending

1. PURPOSE

The purpose of this procedure is to provide detailed information on the procedure for "in-house" M5/202 analysis.

2. SCOPE

This procedure applies to those individuals bringing field samples into the laboratory area and the laboratory technicians performing the requested analysis.

3. MATERIALS

The following materials are needed to properly execute this procedure.

- Client Weight Sheets
- Chain of Custody
- Tared beakers
- Desiccator
- Indicator
- Tweezers
- Tongs
- Acetone
- Hood
- Hot Plate
- Rubber policeman/ spatula
- Squeeze bottle
- Gloves
- Sample bottles
- Graduated cylinders
- Separatory funnel
- Analytical balance
- Methylene Chloride
- 5ml pipette

4. DEFINITIONS

- 4.1 Condensable Particulate Matter (CPM): material that passes through a filter and is measured by M202. the organic and aqueous fractions of a sample are taken to dryness and the residue is weighed. The combined weight represents CPM.

5. INTERFERENCES

- 5.1 Ammonia: In sources that use ammonia injection as a control technique for hydrogen chloride (HCl), the ammonia interferes by reacting with HCl in the gas stream to form ammonium chloride (NH_4Cl) which would be measured as CPM. The sample may be analyzed for chloride and the equivalent amount of NH_4Cl can be subtracted from the CPM weight. However, if NH_4Cl is to be counted as CPM, the inorganic fraction should be taken to near dryness (less than 1 ml liquid) in the oven and then allowed to air dry at ambient temperature to prevent any NH_4Cl from vaporizing.

6. PROCEDURE

6.1 Arrival of Samples at Lab

- 6.1.1 Refer to "In house" M5 analysis SOP.

6.2 Organization of Samples

- 6.2.1 Refer to "In house" M5 analysis SOP.

6.3 Chain of Custody

- 6.3.1 Refer to "In house" M5 analysis SOP.

6.4 Client Weight Sheets

- 6.4.1 Refer to "In house" M5 analysis SOP.

6.5 Tared Quartz Filters

- 6.5.1 Record each filter's ID number on client weight sheet in appropriate column along side it's location and run number.

6.5.2 Place filters in desiccator.

- 6.5.2.1 Attach note to desiccator door to identify samples. (Refer to "In house" M5 analysis SOP Figure 4).

6.5.3 Perform 1st gross weighing.

6.5.4 Perform 2nd gross weighing.

6.5.5 Remove filters from desiccator.

- 6.5.5.1 Place filters on side counter and label accordingly. (Refer to "In house" M5 analysis SOP Figure 1).

6.6 Front Half Acetone Samples

- 6.6.1 Obtain tared beakers. (Choose beaker size compatible with sample volume).

- 6.6.1.1 Tared beakers are kept in the plexiglass box on the shelf in the weighing room

- 6.6.1.2 Extra tared beakers are stored wrapped in syran wrap or foil in a labeled box in the weighing room.
- 6.6.2 Match one beakers to each front half acetone sample.
- 6.6.3 Write the beaker ID number on the sample jar that you are matching it with.
- 6.6.4 Record this beaker ID number on the client weight sheet beside the appropriate sample location and run.
- 6.6.5 Arrange the beakers on the hot plate under the hood in some logical order, i.e., place samples from the same location together or arrange sequential runs together.
- 6.6.6 Turn on hood fan.
- 6.6.7 Carefully and quantitatively transfer the acetone samples from the jars to the coordinating beakers.
- 6.6.8 Record the volume of the sample in the beaker on the client weight sheet.
- 6.6.9 After emptying all samples into beakers and recording ID numbers and volumes, turn on the hot plate. Set initial temperture dial to 80-100° C. Watch the beakers and adjust temperature as necessary to ensure that the samples do not boil. SAMPLES SHOULD NEVER BE ALLOWED TO BOIL. Adjust temperature accordingly.
- 6.6.10 Attach note to hood to identify samples. (Refer to "In house" M5 analysis SOP Figure 5).
- 6.6.11 Place empty sample jars on side counter with a note identifying them (Refer to "In house" M5 analysis SOP Figure 1).
- 6.6.12 When front half acetone samples are completely evaporated, turn hot plate off and allow beakers to cool.
- 6.6.13 Transfer beakers to the desiccator.
 - 6.6.13.1 Remember not to touch beakers with bare hands.
 - 6.6.13.1.1 Wearing gloves or using tongs, place beakers onto clipboard and carefully carry them into weighing room.
- 6.6.14 Arrange beakers in desiccator in a logical order, i.e., place unit A samples together, unit B samples together, to simplify weighing process.
 - 6.6.14.1 Place note on desiccator door to identify samples. (Refer to "In house" M5 analysis SOP Figure 4).
- 6.6.15 Perform 1st gross weighing
- 6.6.16 Perform 2nd gross weighing
- 6.6.17 After 2nd gross weight is acquired, remove beakers from desiccator.
- 6.6.18 Place beakers with coordinating sample jar on side counter.
- 6.6.19 Reconstitute front half acetone samples.

6.6.19.1 Refer to "In house" M5 analysis section 5.7.

6.7 Back Half Impinger Catch

6.7.1 Before extraction, remove a 5 ml aliquot of the B1/2 sample for later ion chromatography of sulfates.

6.8 Extraction

6.8.1 Combine the organic (MeCl_2) and the aqueous (B1/2 impinger catch) samples in a 1000ml Separatory funnel.

6.8.2 Place stopper in funnel.

6.8.3 Gently and carefully shake funnel to mix contents.

6.8.3.1 This process should be performed under the hood while wearing proper PPE.

6.8.4 Burp the funnel.

6.8.4.1 Tilt the funnel so that the valve is pointed upward and the liquid settles in the top portion of the funnel. Slowly open the valve to release built-up pressure.

6.8.4.2 Make sure you have the stopper securely in place and hold your hand over it so it doesn't fall out.

6.8.5 Close the funnel valve.

6.8.6 Set funnel in holder.

6.8.7 Allow contents to settle and separate.

6.8.7.1 It may be necessary to tap the sides of the funnel to release any bubbles clinging to the sides of the glass.

6.8.8 Slowly open valve and drain off most of the organic (MeCl_2) phase.

6.8.8.1 Drain into a tared beaker.

6.8.8.2 Record the beaker ID number on the Client Weight Sheet.

6.8.8.3 Allow a small amount of MeCl_2 to remain in the Sep. funnel to ensure that no inorganic phase is collected in the organic sample.

6.8.9 Add 75ml of MeCl_2 to the Sep. funnel.

6.8.10 Repeat steps 6.8.3 through 6.8.8.3.

6.8.11 Repeat steps 6.8.9 and 6.8.10.

6.8.12 Empty remaining aqueous fraction into a tared beaker.

6.8.12.1 Record the beaker ID number on the Client Weight Sheet.

6.9 Evaporation

6.9.1 Place the organic samples (MeCl_2 beakers) under the hood but not on the hot plate.

- 6.9.1.1 These samples should be allowed to evaporate at ambient conditions
- 6.9.2 Place the aqueous samples (Back half impinger catch beakers) under the hood on the hot plate.
- 6.9.3 Turn on hot plate. Set initial temperature dial to 80-100°C. Watch the beakers and adjust temperature as necessary to ensure that the samples do not boil. **SAMPLES SHOULD NEVER BE ALLOWED TO BOIL.** Adjust temperature accordingly.
- 6.9.4 Attach note to hood to identify samples. (Refer to "In House" M5 analysis SOP Figure 5).
- 6.9.5 When back half samples are completely evaporated, turn hot plate off and allow beakers to cool.
- 6.9.6 Continue evaporating organic samples until completely dry.
- 6.9.7 Transfer beakers to the desiccator.
 - 6.9.7.1 Remember not to touch beakers with bare hands.
- 6.9.8 Place note on desiccator door to identify samples. (Refer to "In house" M5 analysis SOP Figure 4).
- 6.9.9 Perform 1st gross weighing.
- 6.9.10 Perform 2nd gross weighing.
- 6.9.11 Remove beakers from desiccator.
 - 6.9.11.1 If weights are verified, beakers may be cleaned and retared.

```

graph TD
    Start([M5/202 SAMPLES  
ARRIVE AT PITT  
LABORATORY]) --> Place[Place samples on center island]
    Place --> Identify[Identify samples and note  
analytical instructions]
    Identify --> Organize[Organize samples]
    Identify --> JobInfo[JOB #  
CLIENT NAME  
ANALYSIS REQUESTED  
CERTIFICATION  
PROJECT MANAGER  
DETERMINATION]
    Organize --> Match[Match samples with  
Chain of Custodians]
    Match --> CreateSheet([Create Client Weight  
Sheet])
    CreateSheet --> RefFuel[Ref-Fuel  
Results]
    CreateSheet --> JobM5([JOB M5  
Weights])
    JobM5 --> Worksheet1([WORKSHEET  
1])
    JobM5 --> Worksheet2([WORKSHEET  
2])
    JobM5 --> Worksheet3([WORKSHEET  
3])
    CreateSheet --> Filter[FILTER]
    Filter --> RecordID[Record ID# on client  
Weight sheet]
    RecordID --> PlaceFilters[Place filters in  
desiccator]
    PlaceFilters --> IdentifyHood[Identify samples by attaching  
note in desiccator]
    IdentifyHood --> JobInfo2[JOB #  
CLIENT NAME  
SAMPLE FRACTION  
1ST WEIGHT (DATE/TIME)  
2ND WEIGHT (DATE/TIME)]
    IdentifyHood --> Perform1[PERFORM  
1ST TARE  
WEIGHT]
    Perform1 --> Perform2[PERFORM  
2ND TARE  
WEIGHT]
    Perform2 --> Remove[Remove samples  
from  
desiccator  
and place  
on side  
counter]
    Remove --> Archive1((ARCHIVE))
    CreateSheet --> Rinse[MeCl2  
RINSE]
    Rinse --> Catch[B1/2 IMP  
CATCH]
    Catch --> RemoveAliq[Remove final aliquot  
from sample]
    RemoveAliq --> PerformEx[PERFORM  
EXTRACTION]
    PerformEx --> Combine[Combine MeCl2 and  
B1/2 Imp catch in  
1000ml Sep. funnel]
    Combine --> Shake[Shake]
    Shake --> TurnHood[Turn on hood]
    TurnHood --> AllowSep[Allow contents to separate]
    AllowSep --> OpenValve[Open valve and  
drain MeCl2 into  
a tared beaker]
    OpenValve --> Add75ml[Add 75ml MeCl2 to Sep. funnel]
    Add75ml --> RepeatProcess[Repeat process: Shake, Burp,  
Separate, Drain]
    RepeatProcess --> Add75ml2[Add 75ml MeCl2 to Sep. funnel]
    Add75ml2 --> RepeatProcess2[Repeat process: Shake, Burp,  
Separate, Drain]
    RepeatProcess2 --> EmptyWater[Empty remaining B1/2 water  
into a tared beaker]
    EmptyWater --> Evaporation[EVAPORATION]
    Evaporation --> PlaceOrganic[Place organic (MeCl2) beakers  
under hood (not on hot plate)]
    PlaceOrganic --> TurnHood2[Turn on hood and  
hot plate (adjust  
temp., accordingly)]
    TurnHood2 --> AttachNote[Attach note to  
Identify samples]
    AttachNote --> Evaporate[Evaporate until  
completely dry]
    Evaporate --> Desiccate[Desiccate]
    Desiccate --> Perform1st[Perform 1st  
gross weight]
    Perform1st --> Perform2nd[Perform 2nd  
gross weight]
    Perform2nd --> RemoveFrom[Remove from  
desiccator]
    RemoveFrom --> NoteClean[If weights are  
verified, beakers  
may be cleaned and  
reused]
    NoteClean --> Cover[Cover samples and label DO  
NOT  
RECONSTITUTE,  
FURTHER  
ANALYSIS  
NECESSARY]
    Cover --> FurtherYes[FURTHER  
ANALYSIS? YES]
    FurtherYes --> FurtherNo[FURTHER  
ANALYSIS? NO]
    FurtherNo --> Reconstitute[RECONSTITUTE  
SAMPLES]
    Reconstitute --> ObtainAcetone[Obtain acetone  
backlund coordinating  
sample jar from side  
counter]
    ObtainAcetone --> FillBottle[Fill squeeze  
bottle with  
acetone]
    FillBottle --> SquirtAcetone[Squirt acetone  
into beaker]
    SquirtAcetone --> RubBeaker[Rub sides/bottom of beaker  
with acetone to loosen dried  
particulate]
    RubBeaker --> DumpWash[Dump acetone wash  
into original sample  
jar]
    DumpWash --> RepeatSteps[Repeat last two steps until no  
residue remains in beaker.]
    RepeatSteps --> Archive2((ARCHIVE))
    CreateSheet --> Catch2[F1/2  
ACETONE]
    Catch2 --> ObtainTared[Obtain tared  
beakers]
    ObtainTared --> MatchBeaker[Match one beaker to  
every F1/2 sample]
    MatchBeaker --> WriteBeaker[Write beaker number on sample jar and  
in appropriate space on client weight  
sheet]
    WriteBeaker --> ArrangeHood[Arrange beakers on hot plate  
under hood in some logical  
order]
    ArrangeHood --> TurnHood3[Turn on hood fan]
    TurnHood3 --> DumpAcetone[Dump acetone from jars into  
coordinating beakers]
    DumpAcetone --> PlaceEmpty[Place empty  
sample jars on  
side counter  
and identify  
with note]
    PlaceEmpty --> RecordVolume[Record volume]
    RecordVolume --> TurnPlate[Turn on oil plate]
    TurnPlate --> Evaporate2[Evaporate samples]
    Evaporate2 --> AllowCool[Allow to cool]
    AllowCool --> TransferDesiccator[Transfer beakers to  
desiccator]
    TransferDesiccator --> IdentifyHood3[Identify beakers by  
attaching note to  
desiccator]
    IdentifyHood3 --> JobInfo3[JOB #  
CLIENT NAME  
SAMPLE FRACTION  
1ST WEIGHT (DATE/TIME)  
2ND WEIGHT (DATE/TIME)]
    IdentifyHood3 --> Perform1Tare[PERFORM  
1ST TARE  
WEIGHT]
    Perform1Tare --> Perform2Tare[PERFORM  
2ND TARE  
WEIGHT]
    Perform2Tare --> PlaceCoordinating[Place beakers with  
coordinating sample  
jars on side counter]
    PlaceCoordinating --> FurtherYes2[FURTHER  
ANALYSIS? YES]
    FurtherYes2 --> FurtherNo2[FURTHER  
ANALYSIS? NO]
    FurtherNo2 --> Reconstitute2[RECONSTITUTE  
SAMPLES]
    Reconstitute2 --> ObtainAcetone2[Obtain acetone  
backlund coordinating  
sample jar from side  
counter]
    ObtainAcetone2 --> FillBottle2[Fill squeeze  
bottle with  
acetone]
    FillBottle2 --> SquirtAcetone2[Squirt acetone  
into beaker]
    SquirtAcetone2 --> RubBeaker2[Rub sides/bottom of beaker  
with acetone to loosen dried  
particulate]
    RubBeaker2 --> DumpWash2[Dump acetone wash  
into original sample  
jar]
    DumpWash2 --> RepeatSteps2[Repeat last two steps until no  
residue remains in beaker.]
    RepeatSteps2 --> Archive2
  
```

8. ATTACHMENTS

CAE STANDARD OPERATING PROCEDURE

EPA M5/29 GRAVIMETRIC ANALYSIS

ISO NUMBER: LAB-7.

AUTHOR: DEPT. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1. PURPOSE

The purpose of this procedure is to provide detailed information for performing "in-house" analysis of EPA M5/29 samples.

2. SCOPE

This procedure should be followed by all individuals bringing field samples into the laboratory area and all laboratory technicians performing the gravimetric analysis.

3. MATERIALS

The following materials are needed to properly execute this procedure

- Client Weight Sheet
- Chain of Custody
- Tared metals prepped beakers
- Desiccator
- Tweezers
- Tongs
- Clip board
- Acetone
- Hood
- Hot Plate
- Rubber policeman/ spatula
- Squeeze bottle
- Teflon tape
- Plastic shipping bags
- Zip-ties
- Shipping box
- Fed-Ex form (non-haz.)
- Vermiculite
- Tape

4. DEFINITIONS

- 4.1 "In House" analysis: Any analytical procedure that takes place in the Pittsburgh laboratory
- 4.2 Gravimetric: Quantitative analysis based on weights
- 4.3 Chain of Custody: Paperwork that identifies samples and accompanies samples whenever they are relinquished to another party
- 4.4 Client Weight Sheet: Data sheet which contains all sample identification information including sample type, run number, location, ID numbers, sample volumes, gross weights, tare weights, and net weights
- 4.5 Tared: Sample containers, (beakers and thimbles), or filters that have been weighed twice; with consecutive weighings differing by no more than 0.0005g
- 4.6 Reconstitute: Rehydrating a sample which has been evaporated
- 4.7 Gross weight: The total weight of a sample, including the weight of any container in which the sample is contained
- 4.8 Net weight: The gross weight minus the tare weight
- 4.9 Archive shelves: The shelves against the back wall of the Pitt lab which are labeled with the months of the year

5. PROCEDURE

5.1 Arrival of Samples

- 5.1.1 Refer to "In house" M5 analysis SOP.

5.2 Organization of Samples

- 5.2.1 Refer to "In house" M5 analysis SOP.

5.3 Chain of Custody

- 5.3.1 Refer to "In house" M5 analysis SOP.

5.4 Client Weight Sheets

- 5.4.1 Refer to "In house" M5 analysis SOP.

5.5 Filters

- 5.5.1 Record each filter's ID number on client weight sheet in appropriate column along side it's location and run number.
- 5.5.2 Place filters in desiccator.
 - 5.5.2.1 Attach note to desiccator door to identify samples. (Refer to "In house" M5 analysis SOP Figure 4).
- 5.5.3 Perform 1st gross weighing.

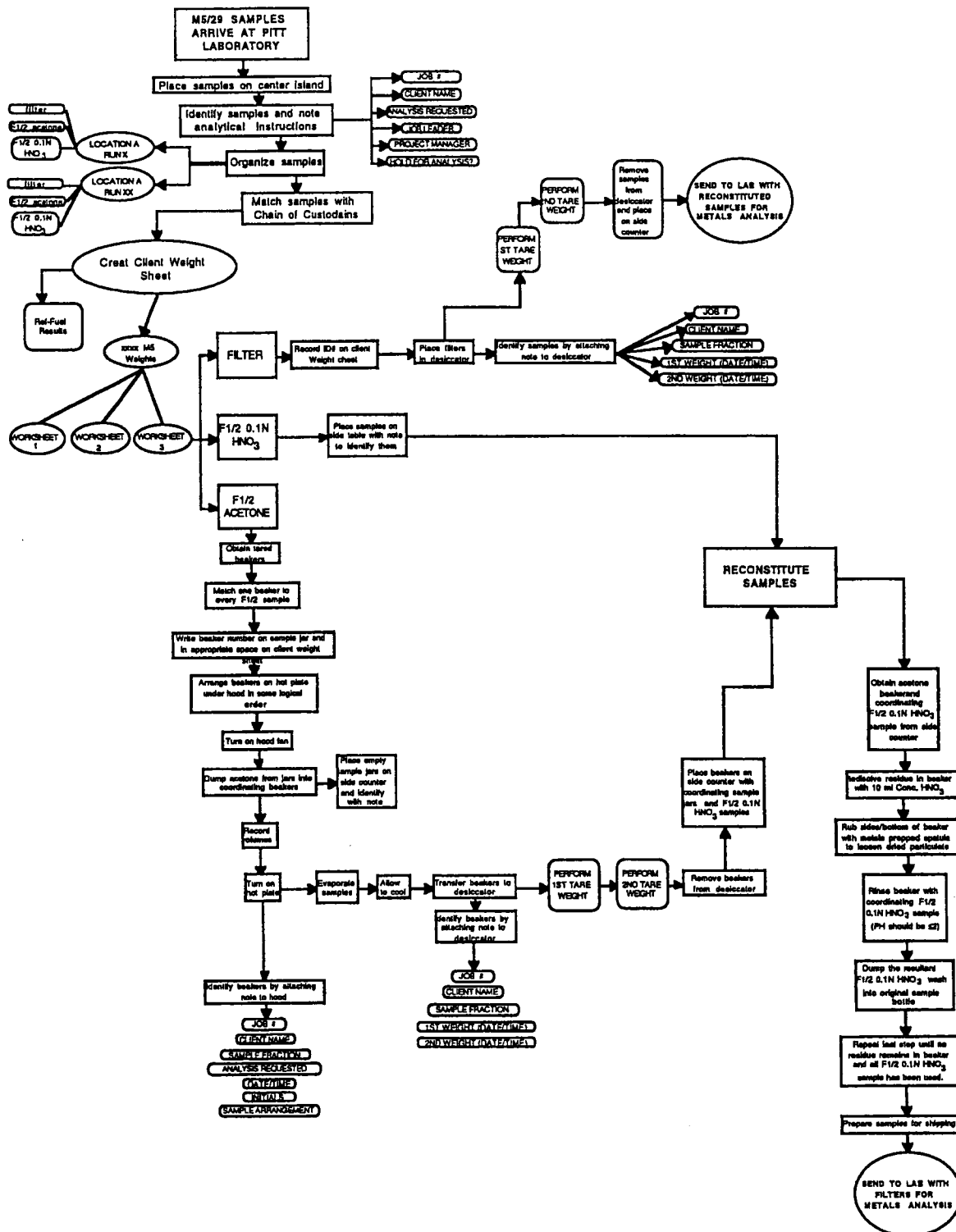
- 5.5.4 Perform 2nd gross weighing.
- 5.5.5 Remove filters from desiccator.
 - 5.5.5.1 Place filters on side counter and label accordingly. (Refer to "In house" M5 analysis SOP Figure 1).

5.6 Front Half Acetone Samples

- 5.6.1 Obtain tared beakers. (Choose beaker size compatible with sample volume).
 - 5.6.1.1 Tared beakers are kept in the plexiglass box on the shelf in the weighing room
 - 5.6.1.2 Extra tared beakers are stored wrapped in syran wrap or foil in a labeled box in the weighing room.
- 5.6.2 Match one beaker to each front half acetone sample.
- 5.6.3 Write the beaker ID number on the sample jar that you are matching it with.
- 5.6.4 Record this beaker ID number on the client weight sheet beside the appropriate sample location and run.
- 5.6.5 Arrange the beakers on the hot plate under the hood in some logical order, i.e., place samples from the same location together or arrange sequential runs together.
- 5.6.6 Turn on hood fan.
- 5.6.7 Carefully and quantitatively transfer the acetone samples from the jars to the coordinating beakers.
- 5.6.8 Record the volume of the sample in the beaker on the client weight sheet.
- 5.6.9 After emptying all samples into beakers and recording ID numbers and volumes, turn on the hot plate. Set initial temperture dial to 80-100° C. Watch the beakers and adjust temperature as necessary to ensure that the samples do not boil. **SAMPLES SHOULD NEVER BE ALLOWED TO BOIL.** Adjust temperature accordingly.
- 5.6.10 Attach note to hood to identify samples. (Refer to "In house" M5 analysis SOP Figure 5).
- 5.6.11 Place empty sample jars on side counter with a note identifying them (Refer to "In house" M5 analysis SOP Figure 1).
- 5.6.12 When front half acetone samples are completely evaporated, turn hot plate off and allow beakers to cool.
- 5.6.13 Transfer beakers to the desiccator.
 - 5.6.13.1 Remember not to touch beakers with bare hands.
 - 5.6.13.1.1 Wearing gloves or using tongs, place beakers onto clipboard and carefully carry them into weighing room.

- 5.6.14 Arrange beakers in desiccator in a logical order, i.e., place unit A samples together, unit B samples together, to simplify weighing process.
 - 5.6.14.1 Place note on desiccator door to identify samples. (Refer to "In house" M5 analysis SOP Figure 4).
- 5.6.15 Perform 1st gross weighing
- 5.6.16 Perform 2nd gross weighing
- 5.6.17 After 2nd gross weight is acquired, remove beakers from desiccator.
- 5.6.18 Place beakers with coordinating sample jar on side counter.
- 5.7 Reconstituting Samples
 - 5.7.1 Obtain Front half 0.1 N HNO₃ samples from coordinating test locations and runs.
 - 5.7.2 Carefully and quantitatively, transfer Front half 0.1 N HNO₃ samples into coordinating acetone beakers.
 - 5.7.3 With rubber policeman or Teflon spatula (that has been metals prepped) rub sides and bottom of beaker to loosen dried particulate.
 - 5.7.4 Carefully and quantitatively, transfer liquid into original acetone sample bottles or Front half 0.1 N HNO₃ sample bottles.
 - 5.7.5 Repeat process until beaker is thoroughly cleaned and no visible particulate remains in beaker.
- 5.8 Send Samples to Independent Lab
 - 5.8.1 Obtain filters and reconstituted samples.
 - 5.8.2 Wrap lids with teflon tape.
 - 5.8.3 Place samples in plastic bags and zip tie.
 - 5.8.4 Prepare for shipping (Place samples in box with vermiculite).
 - 5.8.5 Place Chain of Custodians in a plastic bag and place in box of samples.
 - 5.8.6 Prepare Fed-Ex form (non-haz.).
 - 5.8.7 Ship to independent lab.

6. PROCESS FLOW DIAGRAM



CAE STANDARD OPERATING PROCEDURE

NJATM5 GRAVIMETRIC

ISO NUMBER: LAB-8.

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1. PURPOSE

The purpose of this procedure is to provide detailed information for performing "in-house" analysis of NJATM5 samples.

2. SCOPE

This procedure should be followed by all individuals bringing field samples into the laboratory area and all laboratory technicians performing gravimetric analysis.

3. MATERIALS

- Client Weight Sheets
- Chain of Custody
- Tared particulate prepped beakers
- Desiccator
- Tweezers
- Tongs
- Clip board
- Acetone
- Hood
- Hot Plate
- Rubber policeman/ spatula
- Teflon squeeze bottles
- Tared Gooch crucibles fitted with glass fiber filter discs
- Chloroform
- Ethyl Ether
- Graduated Cylinders
- Personal Protective Equipment
- Separatory Funnel
- Filtering Flask
- Thomas Pump
- Rubber stopper

4. DEFINITIONS

- 4.1 "In House" analysis: Any analytical procedure that takes place in the Pittsburgh laboratory
- 4.2 Gravimetric: Quantitative analysis based on weights
- 4.3 Chain of Custody: Paperwork that identifies samples and accompanies samples whenever they are relinquished to another party
- 4.4 Client Weight Sheet: Data sheet which contains all sample identification information including sample type, run number, location, ID numbers, sample volumes, gross weights, tare weights, and net weights
- 4.5 Tared: Sample containers, (beakers and thimbles), or filters that have been weighed twice; with consecutive weighings differing by no more than 0.0005g
- 4.6 Reconstitute: Rehydrating a sample which has been evaporated
- 4.7 Gross weight: The total weight of a sample, including the weight of any container in which the sample is contained
- 4.8 Net weight: The gross weight minus the tare weight
- 4.9 Archive shelves: The shelves against the back wall of the lab which are labeled with the months of the year
- 4.10 Gooch crucible: Porcelain capsules with tiny holes in the bottom to allow filtration
- 4.11 Glass fiber filter discs: 2.4 cm filters used with Gooch crucibles
- 4.12 Thomas pump: Small lightweight pump used to create vacuum to filter liquid
- 4.13 Filtering Flask: Erlenmeyer flask which has a stem to connect tubing from pump
- 4.14 Separatory Funnel (Sep. funnel): Pear-shaped funnel equipped with teflon stopcock plug which allows you to drain off separated fractions
- 4.15 Personal Protective Equipment (PPE): Any equipment, glasses, goggles, apron, gloves, etc., that ensures worker's health and safety
- 4.16 Rubber crucible holder: Holds Gooch or similar crucibles in the neck of standard suction flasks
- 4.17 Material Safety Data Sheets (MSDS): Provides various amounts of information regarding specific chemicals.

5. PROCEDURE

5.1 Arrival of Samples

5.1.1 Refer to "In house" M5 analysis SOP.

5.2 Organization of Samples

5.2.1 Refer to "In house" M5 analysis SOP.

5.3 Chain of Custody

5.3.1 Refer to "In house" M5 analysis SOP.

5.4 Client Weight Sheets

5.4.1 Refer to "In house" M5 analysis SOP.

5.5 Tared Quartz Filters

5.5.1 Record each filter's ID number on client weight sheet in appropriate column along side it's location and run number.

5.5.2 Place filters in desiccator.

5.5.2.1 Attach note to desiccator door to identify samples. (Refer to "In house" M5 analysis SOP Figure 4).

5.5.3 Perform 1st gross weighing.

5.5.4 Perform 2nd gross weighing.

5.5.5 Remove filters from desiccator.

5.5.5.1 Place filters on side counter and label accordingly. (Refer to "In house" M5 analysis SOP Figure 1).

5.6 Front Half Acetone Samples

5.6.1 Obtain tared beakers. (Choose beaker size compatible with sample volume).

5.6.1.1 Tared beakers are kept in the plexiglass box on the shelf in the weighing room

5.6.1.2 Extra tared beakers are stored wrapped in syran wrap or foil in a labeled box in the weighing room.

5.6.2 Match one beaker to each front half acetone sample.

5.6.3 Write the beaker ID number on the sample jar that you are matching it with.

5.6.4 Record this beaker ID number on the client weight sheet beside the appropriate sample location and run.

5.6.5 Arrange the beakers on the hot plate under the hood in some logical order, i.e., place samples from the same location together or arrange sequential runs together.

- 5.6.6 Turn on hood fan.
- 5.6.7 Carefully and quantitatively transfer the acetone samples from the jars to the coordinating beakers.
- 5.6.8 Record the volume of the sample in the beaker on the client weight sheet.
- 5.6.9 After emptying all samples into beakers and recording ID numbers and volumes, turn on the hot plate. Set initial temperature dial to 80-100° C. Watch the beakers and adjust temperature as necessary to ensure that the samples do not boil. **SAMPLES SHOULD NEVER BE ALLOWED TO BOIL.** Adjust temperature accordingly.
- 5.6.10 Attach note to hood to identify samples. (Refer to "In house" M5 analysis SOP Figure 5).
- 5.6.11 Place empty sample jars on side counter with a note identifying them (Refer to "In house" M5 analysis SOP Figure 1).
- 5.6.12 When front half acetone samples are completely evaporated, turn hot plate off and allow beakers to cool.
- 5.6.13 Transfer beakers to the desiccator.
 - 5.6.13.1 Remember not to touch beakers with bare hands.
 - 5.6.13.1.1 Wearing gloves or using tongs, place beakers onto clipboard and carefully carry them into weighing room.
- 5.6.14 Arrange beakers in desiccator in a logical order, i.e., place unit A samples together, unit B samples together, to simplify weighing process.
 - 5.6.14.1 Place note on desiccator door to identify samples. (Refer to "In house" M5 analysis SOP Figure 4).
- 5.6.15 Perform 1st gross weighing
- 5.6.16 Perform 2nd gross weighing
- 5.6.17 After 2nd gross weight is acquired, remove beakers from desiccator.
- 5.6.18 Place beakers with coordinating sample jar on side counter.
- 5.6.19 Reconstitute front half acetone samples.
 - 5.6.19.1 Refer to "in house" M5 analysis section 5.7.

5.7 Back Half Impinger Catch Samples

- 5.7.1 Obtain tared Gooch crucibles fitted with glass fiber filter discs, rubber stopper, Thomas pump, filtering flask, back half impinger catch samples, and tared particulate prepped beakers.
- 5.7.2 Match one tared crucible and filter and two tared beakers to each back half impinger catch sample.
- 5.7.3 Record ID numbers on the Client Weight Sheet.
- 5.7.4 Place Gooch crucible and filter into rubber stopper.

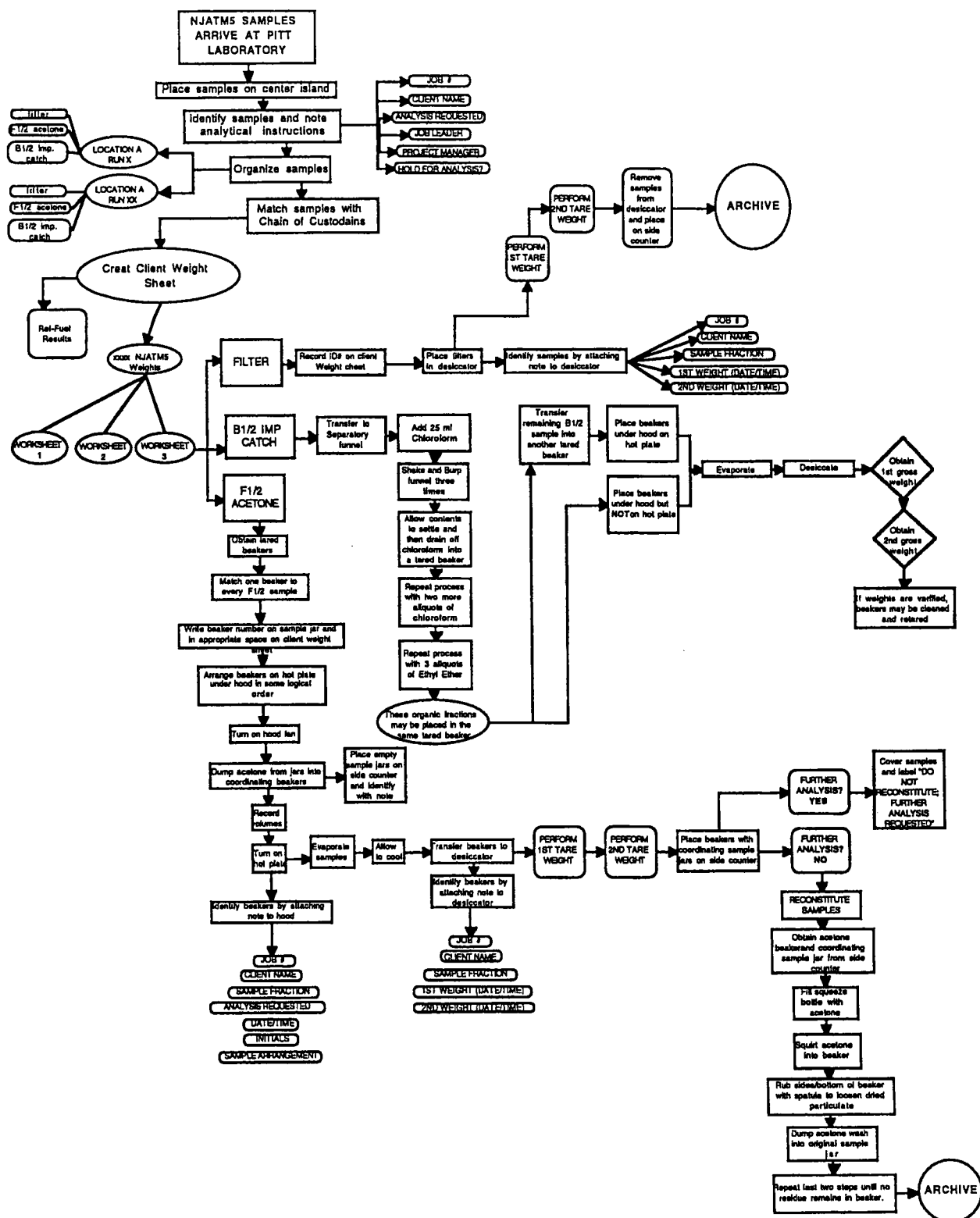
- 5.7.5 Attach tubing from Thomas pump to the stem on the filtering flask.
 - 5.7.5.1 Note: A knockout jar filled with silica gel should be attached to the Thomas pump to ensure no liquid enters the pump.
- 5.7.6 Place rubber stopper and crucible snugly into the top of the filtering flask.
- 5.7.7 Turn on the pump.
- 5.7.8 Quantitatively transfer the back half impinger catch sample into the crucible.
 - 5.7.8.1 Do not over flow the crucible.
 - 5.7.8.2 Continue to transfer sample until all liquid has been filtered.
- 5.7.9 Remove pump tubing from filtering flask.
- 5.7.10 Remove crucible from rubber stopper.
 - 5.7.10.1 Remember, do not touch crucible with bare hands.
- 5.7.11 Place crucible on the hot plate under the hood.
- 5.7.12 Transfer filtered sample from flask back to its original sample container.
- 5.7.13 Continue this process with all remaining samples.
- 5.7.14 Turn on hot plate. Temperature should not exceed 75°C.
- 5.7.15 Turn on hood fan.
 - 5.7.15.1 Attach note to hood to identify samples (Refer to "In House" M5 Analysis SOP Figure 5).
- 5.7.16 When crucibles and filters are dry, remove from hot plate and transfer to desiccator.
 - 5.7.16.1 Reminder: Do not touch crucibles with bare hands.
 - 5.7.16.2 Attach note to desiccator to identify samples (Refer to "In House" M5 Analysis SOP Figure 4).
- 5.7.17 Obtain 1st gross weighing.
- 5.7.18 Obtain 2nd gross weighing.
- 5.7.19 After 2nd gross weighing is acquired, remove crucibles from desiccator.
- 5.7.20 Remove filters from crucibles and discard.
- 5.7.21 Crucibles may be washed, refitted with a glass fiber filter disc, and retared.

5.8 Extraction

- 5.8.1 Obtain separatory funnel, filtered back half samples, tared beakers, graduated cylinders, PPE, Chloroform, Ethyl Ether, and teflon squeeze bottles.
- 5.8.2 Extract organic particulate from the impinger solution.

- 5.8.2.1 Transfer filtered back half sample from sample jar to Sep. funnel.
 - 5.8.2.1.1 Be sure Sep. funnel valve is closed.
 - 5.8.2.1.2 Place empty sample jars on side counter in lab.
- 5.8.2.2 Add 25 ml chloroform to Sep funnel.
- 5.8.2.3 Place stopper on Sep. funnel.
- 5.8.2.4 Holding stopper securely in place, carefully shake Sep. funnel two or three times.
- 5.8.2.5 Release built up pressure from funnel.
 - 5.8.2.5.1 Tilt funnel so liquid is in the upper portion of the funnel and away from valve, slowly open the valve.
- 5.8.2.6 Shake and release pressure three or four times.
 - 5.8.2.6.1 This process should be performed under the hood and while wearing proper PPE.
 - 5.8.2.6.2 For information on Chloroform, consult the MSDS.
- 5.8.2.7 Place Sep. funnel in holder.
- 5.8.2.8 Place tared beaker under Sep. valve.
- 5.8.2.9 When fractions completely separate, open valve and drain off chloroform.
 - 5.8.2.9.1 It may be necessary to tap the sides of the Sep. funnel to settle out any bubbles.
- 5.8.2.10 Repeat Sections 5.8.2.2 through 5.8.2.9.1.
- 5.8.2.11 Add 35ml Ethyl Ether to Sep. funnel.
 - 5.8.2.11.1 Combine organic fractions in the same beaker.
 - 5.8.2.11.2 For information on Ethyl Ether, consult the MSDS.
- 5.8.2.12 Repeat Sections 5.8.2.3 through 5.8.2.9.1.
- 5.8.2.13 Repeat Sections 5.8.2.11 and 5.8.2.12.
 - 5.8.2.13.1 Repeat Sections 5.8.2.11 and 5.8.2.12.
- 5.8.2.14 Transfer remaining back half sample into a tared beaker.
 - 5.8.2.14.1 You should now have two beakers for each sample; one organic (chloroform and ethyl ether) fraction, and one aqueous (back half water) fraction.
- 5.8.2.15 Record ID numbers on client weight sheet.
- 5.8.3 Place beakers with the aqueous fractions on hot plate under the hood.
- 5.8.4 Place beakers with the organic fractions under the hood, but not on the hot plate. These should be allowed to evaporate at ambient conditions.
 - 5.8.4.1 Attach note to hood to identify samples (Refer to "In House" M5 Analysis SOP Figure 5).
- 5.8.5 Adjust temperature of hot plate.
 - 5.8.5.1 Initial temperature should be between 50 and 100°C. Watch

6. PROCESS FLOW DIAGRAM



CAE STANDARD OPERATING PROCEDURE

ANALYSIS OF PARTICULATE COLLECTED ON 8" X 10" GLASS FIBER FILTERS

ISO NUMBER: PGRV-9

AUTHOR: Dept. 64

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1.0 Purpose- This procedure provides the steps for the analysis of particulate collected on 8" x 10" glass fibers used in high volume testing.

2.0 Scope- This procedure applies to glass fiber filters analyzed in the CAE Gravimetrics Lab.

3.0 Materials-

- Powder free lab gloves
- Plastic or Teflon Tweezers
- Desiccator
- Drierite (Calcium Sulfate)
- Anti-static Brush
- Four Place Analytical Balance (Mettler AJ100)
- Particulate Testing Weight Sheet
- Black Pen

4.0 Definitions-

g - Abbreviation for gram(s)

8 x 10 - Refers to the size of the filter; eight inches by ten inches

GFF - Abbreviation for glass fibre filter

5.0 Procedure-

- 5.1. Check in samples. Complete the procedure for sample receiving before continuing.
- 5.2. Place filters in a dessicator.
- 5.3. Dessicate for no less than 24 hours.

- 5.4. Calibrate the analytical balance. Complete the procedure for the calibration of the Mettler AJ100 balance before continuing.
- 5.5. Using tweezers, remove the filter from the dessicator and place it on the center of the weight pan. Close the balance lid and wait for the balance to stabilize.
- 5.6. Record the weight on the particulate weight sheet.
- 5.7. Remove the filter from the balance and return it to the dessicator.
- 5.8. Dessicate for no less than six hours.
- 5.9. Repeat Steps 5.5 through 5.8 until two consecutive weights are within ± 0.5 mg (or 1% of the total weight) of each other.

6.0 References-

- 6.1. Standard Test Method for High Volume Sampling for Solid Particulate Matter and Determination of Particulate Emissions
- 6.2. 40 CFR Part 60 Appendix A Method 5

7.0 Attachments-

- 7.1 Filter Tare Sheet

CAE STANDARD OPERATING PROCEDURE
GRAVIMETRIC ANALYSIS OF PARTICULATE/METALS SAMPLES
EPA METHOD 5/29

ISO NUMBER: PGRV-10
EFFECTIVE DATE: Pending

AUTHOR: Dept. 64
APPROVED BY: Pending

1.0 Purpose- The purpose of this procedure is to ensure that anyone performing gravimetric analysis of particulate does so in an accurate, consistent manner.

2.0 Scope- This procedure applies to any employee performing the gravimetric analysis of particulate samples collected using combined EPA Methods 5 and 29, which will be forwarded to an outside lab for metals analysis.

3.0 Laboratory Equipment and Glassware-

- Tared Metals Prepped 250 mL Beakers
- Glass Metals Prepped Petri Dishes
- Teflon Acetone Squeeze Bottle
- HDPE 0.1 N Nitric Acid Solution Squeeze Bottle
- Beaker Tongs
- Lab Gloves
- Plastic or Teflon Tweezers
- Four Place Analytical Balance
- Particle Brush
- Dessicator
- Lab Oven
- Hot Plate
- Fume Hood
- 8.5 cm Cardboard Circle
- Invisible Tape
- Particulate Testing Weight Sheet

3.1 Chemicals-

- Acetone
- 0.1 N Nitric Acid Solution
- Drierite (Calcium Sulfate)

4.0 Definitions-

g - Abbreviation for gram

N - Abbreviation for normality

cm - Abbreviation for centimeter

mL - Abbreviation for milliliter

5.0 Procedure-

Note: At no time should gravimetric samples be handled with anything but tweezers or beaker tongs. Oils and heat from your hands can interfere with weights.

- 5.1. Samples must be checked in. Complete the procedure for sample receiving before continuing.

5.2 Procedure 1 - Filters

- 5.2.1. Filters should be received in sealed petri dishes. Remove the teflon tape and the cover of the petri dish, and, using tweezers, transfer the filter into a labeled glass, metals prepped petri dish. Brush any loose particulate from the original dish onto the filter.
- 5.2.2. Place the filter in the lab oven.
- 5.2.3. Dry the filter in the oven at 250° F for six hours.
- 5.2.4. Place the filter in a dessicator.
- 5.2.5. Dessicate the filter for no less than 24 hours.
- 5.2.6. Calibrate the analytical balance. Complete the procedure for the calibration of the Ohaus GA110 analytical balance before continuing.
- 5.2.7. Place (tape) a 8.5 cm cardboard circle on the balance pan. Zero the balance.
- 5.2.8. Using plastic or teflon tweezers, remove the filter from the petri dish and place the filter on the analytical balance. Brush any loose particulate from the petri dish onto the filter. Close the balance door and wait for the balance to stabilize.
- 5.2.9. Record the weight on the particulate testing weight sheet.

5.2.10. Carefully place the filter back into the petri dish and then back into the dessicator.

5.2.11. Dessicate for no less than six hours.

5.2.12. Repeat steps 5.2.8 through 5.2.11 until two consecutive weights are within ± 0.5 mg (or 1% of the total weight) of each other.

5.3 Procedure 2 - Front Half Rinses

5.3.1. Carefully pour the sample into a tared metals prepped 250 mL beaker.

5.3.2. Rinse the sample container three times with acetone. Add the rinses to the sample in the beaker. Record the volume.

5.3.3. Using beaker tongs or lab gloves, place the beaker on the hot plate. Evaporate to dryness. **Do not let the sample boil. If the sample starts to boil lower the heat.**

5.3.4. Once the sample is evaporated, place it in a dessicator.

5.3.5. Dessicate the sample for no less than 24 hours.

5.3.6. Calibrate the analytical balance. Complete the procedure for the calibration of the Ohaus GA200D analytical balance before continuing.

5.3.7. Using beaker tongs, place the beaker on the balance. Close the balance door and wait for the balance to stabilize.

5.3.8. Record the weight on the particulate testing weight sheet.

5.3.9. Return the beaker to the dessicator.

5.3.10. Dessicate for no less than six hours.

5.3.11. Repeat Steps 5.3.7 through 5.3.10 until two consecutive weights are within ± 0.5 mg (or 1% of the total weight) of each other.

5.3.12. Reconstitute the rinse samples with a measured amount of 0.1 N nitric acid solution. Combine this with the Method 29 front half 0.1 N nitric acid rinse collected in the field.

6.0 References

6.1 40 CFR Part 60 Appendix A Method 5

7.0 Attachments-

7.1 Particulate Testing Weight Sheet

CAE STANDARD OPERATING PROCEDURE

**DETERMINATION OF PARTICULATE CONCENTRATION- MODIFIED
EPA METHOD 4**

ISO NUMBER: PGRV-12

AUTHOR: Dept. 64

EFFECTIVE DATE: Pending

APPROVED BY: Pending

Purpose: EPA Method 4 was modified for the collection of particulate matter in the impingers to determine total particulate emissions.

Materials:

Whatman 934 AH filter paper, 11cm

Glass petri dishes

Filtering flask

Buchner funnel

Vacuum pump

Tweezers - 2 pairs

Acetone

DI H₂O

Graduated cylinder

Anti-static brush

Definition:

Constant weight: Two consecutive weights take ≥ 6 hours with a difference $\leq .0005g$ between two weights.

Method:

1. Put numbered filters into clean numbered glass petri dish halves and desiccate for ≥ 24 hours, then weigh filters to a constant weight.
2. Set up buchner funnel on top of filtration flask and attach vacuum hose.
3. Using tweezers, place the tared filter into the Buchner funnel. Make sure to cover all the holes.

4. Moisten filter with DI H₂O and turn vacuum pump on. Again, make sure all holes are covered. If all holes are not covered discontinue vacuum, release the pressure, then carefully adjust filter using a pair of tweezers.
5. Shake sample vigorously (make sure lid is tightly closed).
6. Begin vacuum, then slowly pour sample into the funnel maintaining a thin layer of water over the filter. Do not fill funnel! Continue this until sample bottle is empty, then rinse any remaining particulate matter out of the bottle and add to funnel.
7. Once all moisture appears removed, carefully rinse the inside of the funnel walls with DI H₂O, try to rinse so the particulate matter ends up on the filter. Continue rinsing until all particulate is on the filter; maintain vacuum until all moisture is removed.
8. Once vacuum is turned off, release the pressure. Then, using two pairs of tweezers, carefully remove the filter and return it to its petri dish. Scrape any remaining particulate matter out of the funnel onto the filter. Rinse spatula and tweezers into petri dish using acetone. Rinse any particulate matter left in funnel into petri dish using acetone. Keep acetone rinses low.
9. Return filtered water to its original sample bottle and rinse the flask and funnel before beginning the next sample.
10. Once samples are filtered, place them in the lab oven and heat at 110° F for 4 hours.
11. Remove samples from oven and place in a tightly sealed dessicator for ≥ 24 hours.
12. After 24 hours, weigh samples (one weight) and record results. Once all weights are complete, place the samples in the lab oven and heat at 350° F for 6 hours. Remove samples and place in a tightly sealed dessicator for ≥ 24 hours.
13. After 24 hours, weigh samples to a constant weight and record results. Once samples are completed place filters in a marked plastic petri dish and archive.

CAE STANDARD OPERATING PROCEDURE

OBTAINING PRELIMINARY GRAVIMETRIC RESULTS FOR PM/METALS
SAMPLES COLLECTED BY
EPA METHODS 5 AND 29

ISO NUMBER: PGRV-13

AUTHOR: Dept. 64

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1.0 Purpose- The purpose of this procedure is to ensure that anyone performing the preliminary gravimetric analysis of particulate/metals samples does so in an accurate, consistent manner.

2.0 Scope- This procedure applies to any employee performing the preliminary gravimetric analysis of particulate/metals samples collected by combined Methods 5/29.

3.0 Laboratory Equipment and Glassware-

- Tared 250 mL (or 150 mL) Metals Prepped Beakers
- Teflon Acetone Squeeze Bottle
- Beaker Tongs
- Lab Gloves
- Teflon or Plastic Tweezers
- Four Place Analytical Balances
- Particle Brush
- Dessicator
- Hot Plate
- Fume Hood
- Particulate Testing Weight Sheet
- Air Manifold

3.1 Chemicals-

- Acetone
- Drierite (Calcium Sulfate)

4.0 Definitions-

g - Abbreviation for gram

5.0 Procedure-

Note: At no time should gravimetric samples be handled with anything but tweezers or beaker tongs. Oils and even body heat from hands can interfere with weights.

5.0.1. Samples must be checked in. Complete the procedure for sample receiving before continuing.

5.1 Procedure 1 - Filters

- 5.1.1. Filters should be received in sealed petri dishes. Remove the Teflon tape and the cover of the petri dish so that the filter is exposed, but is still in the bottom of the petri dish..
- 5.1.2. Place the filter in the lab oven for at least two hours at 150.
- 5.1.3. Remove the filters from the lab oven and allow them to cool in a dessicator. (Approximately 30 minutes)
- 5.1.4. Calibrate the analytical balance. Complete the procedure for the calibration of the Ohaus GA110 analytical balance before continuing.
- 5.1.5. Using tweezers, remove the filter from the petri dish and place the filter on the analytical balance. Brush any loose particulate from the petri dish onto the filter. Close the balance door and wait for the balance to stabilize.
- 5.1.6. Record the weight on the preliminary particulate testing weight sheet.
- 5.1.7. Carefully place the filter back into the petri dish and then back into the dessicator.
- 5.1.8. Perform a final analysis on the sample by following the procedure for the gravimetric analysis of particulate.

5.2 Procedure 2 - Front Half Rinses

- 5.2.1. Carefully pour the sample into a tared 250 mL (150 mL) metals prepped beaker.
- 5.2.2. Rinse the sample container three times with acetone. Add the rinses to the sample in the beaker. Record the volume.
- 5.2.3. Using beaker tongs or lab gloves, place the beaker on a hot plate. Position the air manifold so that it blows air across the top of the beakers, not directly into the beakers. Evaporate to dryness. **Do not let the sample boil. If the sample starts to boil, lower the heat.**
- 5.2.4. Once the sample is evaporated, allow the beaker to cool under the fume hood (Approximately 30 minutes).

- 5.2.5. Calibrate the analytical balance. Complete the procedure for the calibration of the Ohaus GA200D analytical balance before continuing.
- 5.2.6. Using beaker tongs, place the beaker on the balance. Close the balance door and wait for the balance to stabilize.
- 5.2.7. Record the weight on the preliminary particulate testing weight sheet.
- 5.2.8. Place the beaker into a dessicator.
- 5.2.9. Perform a final analysis on the sample by following the procedure for the gravimetric analysis of particulate.

6.0 References

- 6.1 40 CFR Part 60 Appendix A Method 5

7.0 Attachments-

- 7.1 Preliminary Particulate Testing Weight Sheet

CAE STANDARD OPERATING PROCEDURE
**ANALYSIS OF ROAD SILT
(PERCENT MOISTURE AND PERCENT SILT)**

ISO NUMBER: PGRV-14
EFFECTIVE DATE: Pending

AUTHOR: Dept. 64
APPROVED BY: Pending

1.0 Purpose- The purpose of this procedure is to ensure that any employee performing the analysis on road silt samples for percent moisture and percent silt, does so in an accurate, consistent manner.

2.0 Scope- This procedure applies to any employee performing percent moisture and percent silt analysis on vacuum bag samples.

3.0 Materials-

- Sieves (1 each 1/4, 4, 20, 60, 100, 140, 200 and pan)
- Sieve Shaker
- Electronic Balance (10th of a gram)
- Lab Oven
- Particle Brush
- Scissors
- Road Silt Data Sheet

4.0 Definitions-

g - Abbreviation for gram

C - Abbreviation for Celsius

5.0 Procedure for Percent Moisture

- 5.0.1. Weigh the vacuum bag on the balance. Record the weight on the road silt data sheet.
- 5.0.2. Place vacuum bag in lab oven and heat at 110° C for 24 hours.
- 5.0.3. Re-weigh the vacuum bag on the balance after drying in oven.
- 5.0.4. Using the following equation determine the percent moisture

$$\text{Percent Moisture} = \frac{(\text{Weight Before Drying} - \text{Weight After Drying})}{(\text{Weight Before Drying})} \times 100$$

5.1 Procedure for Percent Silt

- 5.1.1. After the percent moisture has been determined, tare each sieve (including the pan) on the balance and record the weight on the road silt data sheet. Place them in the following order: 1/4, 4, 20, 60, 100, 140, 200, pan, top to bottom.
- 5.1.2. Carefully cut the vacuum bag and pour the contents into the sieves. Brush any remaining particulate from the bag into the sieves.
- 5.1.3. Cover the sieves and clamp them onto the sieve shaker.
- 5.1.4. Shake sieves for 20 minutes.
- 5.1.5. After the sieves have been shaken for 20 minutes, remove and weigh the pan only. Record the weight on the road silt data sheet.
- 5.1.6. Replace the pan, cover the sieves and shake for an additional 10 minutes.
- 5.1.7. After the sieves have been shaken for 10 minutes, remove and weigh the pan only. Record the weight on the road silt data sheet.
- 5.1.8. Compare the weight to the previous weight.
 - a) If the weights are within 1% of the total weight of the pan, to each other, proceed to step 5.1.9.
 - b) If the weights are not within 1% of the total weight of the pan, to each other, repeat steps 5.1.6-5.1.8.
- 5.1.9. Weigh each sieve and record the weight.
- 5.1.10. Determine the net weight of each pan by subtracting the tare weight from the gross weight. Record the weights on the road silt data sheet.
- 5.1.11. Determine the total weight of the sample by adding all the net weights together. Record the net weight on the road silt data sheet.
- 5.1.12. Determine the percent silt using the following equation:

$$\text{Percent Silt} = \frac{(\text{Net Weight of the Pan})}{(\text{Total Weight of the Sample})} \times 100$$

6.0 References-

- 6.1 EPA Method 450/3-88-008, Silt Analysis Procedure and Moisture Analysis Procedure

7.0 Attachments-

- 7.1 Road Silt Data Sheet

CAE STANDARD OPERATING PROCEDURE
CHANGING AND REGENERATING DRIERITE

ISO NUMBER: PMSC-1
EFFECTIVE DATE: Pending

AUTHOR: Dept. 64
APPROVED BY: Pending

1.0 Purpose- The purpose of this procedure is to provide steps for regenerating drierite (Calcium Sulfate) for use in CAE's Gravimetric Laboratory.

2.0 Scope- This procedure applies to any employee in CAE's Gravimetric Laboratory that is regenerating drierite for the purpose of using it in the dessicators.

3.0 Materials-

- Drierite (Calcium Sulfate)
- Baking Pans
- Lab Oven
- Dessicators
- Metal Stirring Rod
- Oven Gloves

4.0 Definitions-

F - Abbreviation for Farenheit

5.0 Procedure-

Note¹: Drierite indicates when it needs to be regenerated by changing from blue to pink. Only pink drierite should be regenerated.

Note²: More than one tray of drierite may be baked at a time.

Note³: This procedure will probably need to be repeated more when the relative humidity is higher (summer) than when the air is dry (winter).

- 5.1. Pull out the tray from the dessicator containing pink drierite.
- 5.2. Pour the pink drierite from the tray into a glass baking pan.
- 5.3. Place the baking pan in the lab oven at about 350° F.
- 5.4. Once an hour, stir the drierite with a metal rod. (Wear oven gloves.)
- 5.5. Bake the drierite for about six hours or until it turns blue and the pink color is gone.

- 5.6. Pour the regenerated drierite into the dessicator tray.
- 5.7. Place the tray back into the dessicator and make sure the door is closed tightly.

CAE STANDARD OPERATING PROCEDURE
**PREPARATION OF COMMON CHEMICAL SOLUTIONS
(GENERAL GUIDELINES)**

ISO NUMBER: PMSC-2

AUTHOR: Dept. 64

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1.0 Purpose- The purpose of this procedure is to ensure that any employee preparing chemical solutions for the testing and/or analysis of air pollutants, does so in an accurate, consistent and safe manner.

2.0 Scope - This procedure applies to any employee preparing chemical solutions for air pollution testing and/or analysis performed by Clean Air Engineering.

3.0 Materials-

- Balance for weighing dry chemicals
- Weighing Dishes
- Fume Hood
- Lab Gloves
- Safety Glasses (Goggles)
- Lab Coat (Apron)
- Stir Plate
- Teflon Coated Stir Bars
- Magnetic Stir Bar Remover
- Pipette Bulb
- Graduated Cylinders (Various Sizes - See Specific Chemical Recipes)
- Volumetric Flasks (Various Sizes - See Specific Chemical Recipes)
- Pipettes (Various Sizes - See Specific Chemical Recipes)

4.0 Definitions-

g - Abbreviation for grams, used in measuring dry chemicals

mL - Abbreviation for milliliters, used in measuring liquid chemicals

5.0 Procedure - Guidelines- The following steps are general guidelines which outline safe, accurate, consistent ways to prepare chemical solutions.

- 5.0.1. Preparation of all solutions should be done with adequate ventilation. A fume hood is better.
- 5.0.2. Dry chemicals should be weighed on a plastic weighing dish. The balance should be "zeroed" with the dish on it to prevent the weight of the dish from interfering with the weight of the chemical.
- 5.0.3. Safety glasses should always be worn. If working with strong acids or oxidizers the use of goggles is highly recommended.
- 5.0.4. Lab gloves should always be worn.
- 5.0.5. Whenever possible, a volumetric flask and/or pipette should be used for measuring liquids.
- 5.0.6. Always use new, clean glassware when preparing a solution.

5.1 Procedures for Preparing Chemical Solutions- Solutions are listed alphabetically. The information in parenthesis gives the concentration of the solution and indicates which method(s) the solution is generally used for.

Barium Perchlorate Titrant (0.01 Normal) (M6/8)- To make 1 liter of solution: Dissolve 1.95 grams of solid barium perchlorate trihydrate into 200 milliliters of de-ionized water. Dilute to 1 liter with 100% isopropanol.

Citrate Buffer Solution (M15)- To make 1 liter of solution: Dissolve 41 grams of solid citric acid and 300 grams of solid potassium citrate into 500 milliliters of de-ionized water. Dilute to 1 liter with de-ionized water. Using potassium citrate (basic) or citric acid (acidic), adjust the pH of the solution to between 5.4 and 5.6.

EPA Audit Samples for Method 6- To make 100 milliliters of solution: Pipette exactly 5 milliliters of audit sample solution into a 100 milliliter volumetric flask. Add 30 milliliters of 3% hydrogen peroxide solution. Dilute to exactly 100 milliliters with de-ionized water. Keep from evaporation.

Hydrochloric Acid Solution (0.1 Normal) (General)- To make 1 liter of solution: Add 8.6 milliliters of concentrated hydrochloric acid to 500 milliliters of de-ionized water. Dilute to 1 liter with de-ionized water.

Hydrochloric Acid Solution (8 Normal) (M101A)- To make 100 milliliters of solution: Add 69 milliliters of concentrated hydrochloric acid to 25 milliliters of de-ionized water. Dilute to 100 milliliters with de-ionized water.

Hydrogen Peroxide Solution (3%) (M6/8/General)- To make 1 liter of solution: Add 100 milliliters of 30% hydrogen peroxide to 900 milliliters of de-ionized water. Prepare fresh daily.

Hydrogen Peroxide Solution (5%) (M7D)- To make 1 liter of solution: Add 167 milliliters of 30% hydrogen peroxide to 833 milliliters of de-ionized water. Prepare fresh daily.

Isopropanol (80%) (M8)- To make 1 liter of solution: Add 200 milliliters of de-ionized water to 800 milliliters of 100% isopropanol.

Mercuric Nitrate Titrant (0.141 Normal) (HCl Titration)- To make 1 liter of solution: Add 5.0 milliliters of concentrated nitric acid to 900 milliliters of de-ionized water. Dissolve 25 grams of solid mercuric nitrate monohydrate into this solution. Dilute to 1 liter with de-ionized water. Keep away from light.

Mercuric Nitrate Titrant (0.0141 Normal) (HCl Titration)- To make 1 liter of solution: Add 100 milliliters of 0.141 normal mercuric nitrate titrant to 900 milliliters of de-ionized water. Keep away from light.

Mercury Absorbing Solution (4% Potassium Permanganate, 10% Sulfuric Acid) (M101A)- To make 1 liter of solution: Dissolve 40 grams of solid potassium permanganate into 900 milliliters of de-ionized water. Right before use, add 100 milliliters of concentrated sulfuric acid. Prepare fresh daily.

Mixed Indicator for HCl Titration- To make 100 milliliters of solution: Dissolve 0.5 grams of solid diphenylcarbazone and 0.05 grams of solid bromophenol blue powder into 75 milliliters of 95% ethanol. Dilute to 100 milliliters with 95% ethanol.

Multi-Metals Absorbing Solution (10% Hydrogen Peroxide, 5% Nitric Acid) (M29)- To make 1 liter of solution: Add 333 milliliters of 30% hydrogen peroxide to 500 milliliters of de-ionized water. Slowly add 50 milliliters of concentrated nitric acid. Dilute to 1 liter with de-ionized water.

Nitric Acid Solution (10%) (Preparation of Metals Glassware)- To make 1 liter of solution: Add 143 milliliters of concentrated nitric acid to 500 milliliters of de-ionized water. Dilute to 1 liter with de-ionized water.

Nitric Acid Solution (0.1 Normal) (M12/29)- To make 1 liter of solution: Add 6.4 milliliters of concentrated nitric acid to 500 milliliters of de-ionized water. Dilute to 1 liter with de-ionized water.

Nitric Acid Solution (0.05 Normal) (HCl Titration)- To make 1 liter of solution: Add 3.0 milliliters of concentrated nitric acid to 500 milliliters of de-ionized water. Dilute to 1 liter with de-ionized water.

NOx Absorbing Solution (M7)- To make 1 liter of solution: Add 2.8 milliliters of concentrated sulfuric acid to 500 milliliters of de-ionized water. Add 6.0 milliliters of 30% hydrogen peroxide to this solution. Dilute to 1 liter with de-ionized water. Prepare fresh daily.

NOx Absorbing Solution (M7C/D)- To make 1 liter of solution: Dissolve 40 grams of solid potassium permanganate and 20 grams of solid sodium hydroxide into 500 milliliters of de-ionized water. Dilute to 1 liter with de-ionized water. Prepare fresh daily.

Phenolphthalien Solution (1%) (M202)- To make 100 milliliters of solution: Dissolve 1.0 gram of phenolphthalien powder into 60 milliliters of 100% isopropanol. Dilute to 100 milliliters with de-ionized water.

Potassium Hydroxide Solution (0.1 Normal) (BIF 0013)- To make 1 liter of solution: Dissolve 5.6 grams of solid potassium hydroxide into 900 milliliters of de-ionized water. Dilute to 1 liter with de-ionized water.

Potassium Hydroxide Solution (0.2 Normal) (BIF 0013)- To make 1 liter of solution: Dissolve 11.2 grams of solid potassium hydroxide into 900 milliliters of de-ionized water. Dilute to 1 liter with de-ionized water.

Potassium Iodide Solution (10%) (M6)- To make 100 milliliters of solution: Dissolve 10 grams of solid potassium iodide into 50 milliliters of de-ionized water. Dilute to 100 milliliters with de-ionized water.

Sodium Bisulfite Solution (1%) - To make 100 milliliters of solution: Dissolve 1.0 gram of solid sodium bisulfite solution into 50 milliliters of de-ionized water. Dilute to 100 milliliters with de-ionized water.

Sodium Hydroxide Solution (0.1 Normal) (M26/0050/General)- To make 1 liter of solution: Dissolve 4.0 grams of solid sodium hydroxide into 900 milliliters of de-ionized water. Dilute to 1 liter with de-ionized water.

Sodium Hydroxide Solution (1 Normal) (M7/General)- To make 1 liter of solution: Dissolve 40 grams of solid sodium hydroxide into 500 milliliters of de-ionized water. Dilute to 1 liter with de-ionized water.

Sodium Hydroxide Solution (0.25 Normal) (HCl Titration)- To make 1 liter of solution: Dissolve 10.0 grams of solid sodium hydroxide into 500 milliliters of de-ionized water. Dilute to 1 liter with de-ionized water.

Sulfuric Acid Solution (0.1 Normal) (M26/General)- To make 1 liter of solution: Add 2.8 milliliters of concentrated sulfuric acid to 900 milliliters of de-ionized water. Dilute to 1 liter with de-ionized water.

Sulfuric Acid Solution (0.01 Normal) (General)- To make 1 liter of solution: Add 100 milliliters of 0.1 normal sulfuric acid solution to 900 milliliters of de-ionized water.

Thorin Indicator Solution (M6/8)- To make 100 milliliters of solution: Dissolve 0.2 grams of solid thorin to 50 milliliters of de-ionized water. Dilute to 1 liter with de-ionized water.

Xylene Cyanole Solution (HCl Titration)- To make 100 milliliters of solution: Dissolve 0.05 grams of solid xylene cyanole dye into 50 milliliters of 95% ethanol (or 100% isopropanol). Dilute to 100 milliliters with 95% ethanol (or 100% isopropanol).

6.0 References-

40 CFR Part 60 Appendix A Methods 6, 7, 8, 12, 13, 15, 26

40 CFR Part 61 Appendix B Method 101

EPA Proposed Method 29

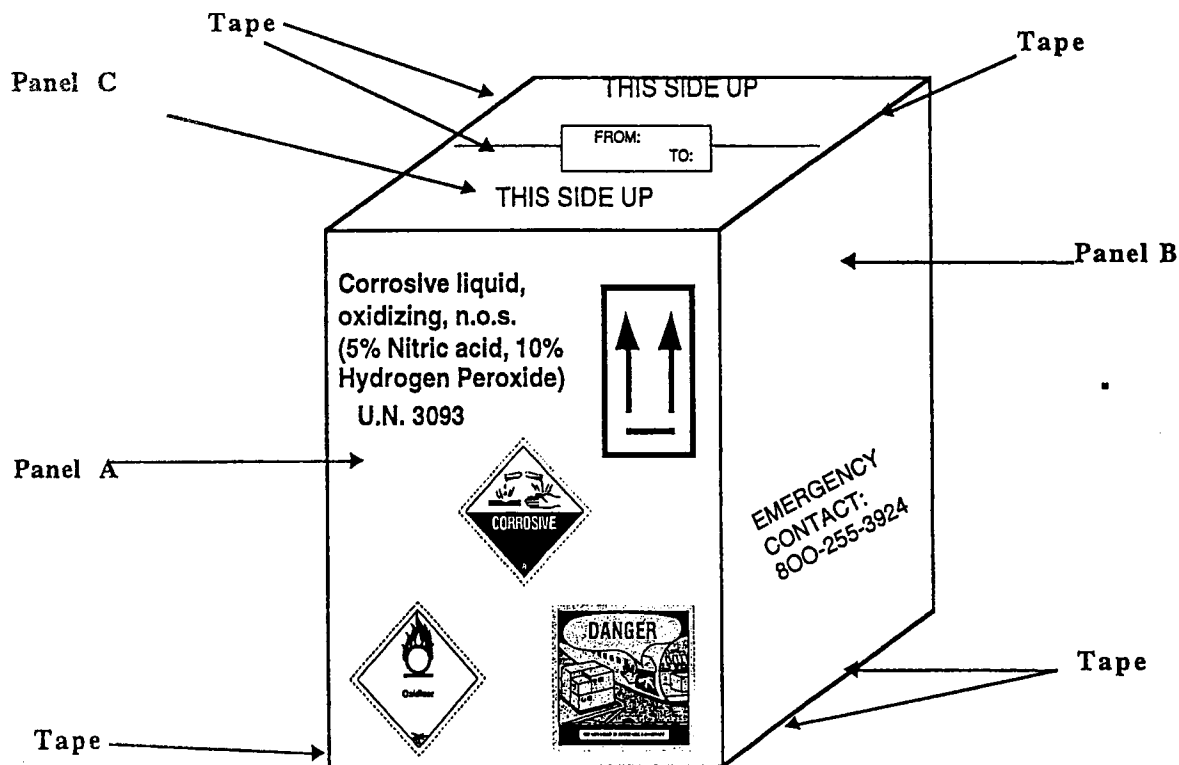
EPA Method 202

Method 325.3 (Chloride- Titrimetric, Mercuric Nitrate)

EPA Methods Manual for Compliance with the BIF Regulations

- 5.16 On one face of the box with the orientation label, panel "A," print the proper shipping name and the U.N. number (this information is found in the C.A.E. D.G.G. or IATA D.G.R.) in the space to the left of the orientation label. See Figure 5B.
Note: All markings must be printed and legible.
- 5.17 In the space below the printed information, place the appropriate hazard and handling labels (this information is found in the C.A.E. D.G.G. or IATA D.G.R.).
Note: The labels must not overlap, cover any printing, or wrap around corners
- 5.18 On Panel "B" print the words "Emergency Contact" and underneath print 800-255-3924. See Figure 5B.
- 5.19 Place a shipping label, with a forwarding and return address, on the top panel "C." See Figure 5B

Figure 5B



- 5.20 Check package over to verify that it is sealed, marked, and labeled properly.

- 5.21 Once the package is complete, fill out the proper air bill and Dangerous Goods Shippers Declaration , then contact the carrier.

CAE STANDARD OPERATING PROCEDURE

THE PACKAGING OF SAMPLES AND REAGENTS IN CLASS 3 FLAMMABLE LIQUID, GROUP I, II, III AND CLASS 6.1, TOXIC, GROUP III

ISO NUMBER: PMSC-4

AUTHOR: Dept. 64

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1.0 PURPOSE

This procedure provides the steps for the packaging of samples and reagents that are Class 3, Flammable Liquid, Group I, II, III or Class 6.1, Toxic, Group III, dangerous goods according to The IATA Dangerous Goods Regulations.

2.0 SCOPE

This procedure applies to CAE. employees with current Dangerous Goods training who package and offer Dangerous Goods for transport.

3.0 MATERIALS

The following materials are needed to properly execute this procedure:

- 4G shipping boxes and inserts(2-cell dividers, 1-separation pad)
- Vermiculite
- Shipping tape
- Teflon tape
- 4ml plastic bags
- Zip ties
- Hazard and shipping labels
- C.A.E. Dangerous Goods Guide or IATA Dangerous Goods Regulations
- Black marker

4.0 DEFINITIONS

IATA D.G.R. - A Current International Air Transportation Association's Dangerous Goods Regulations

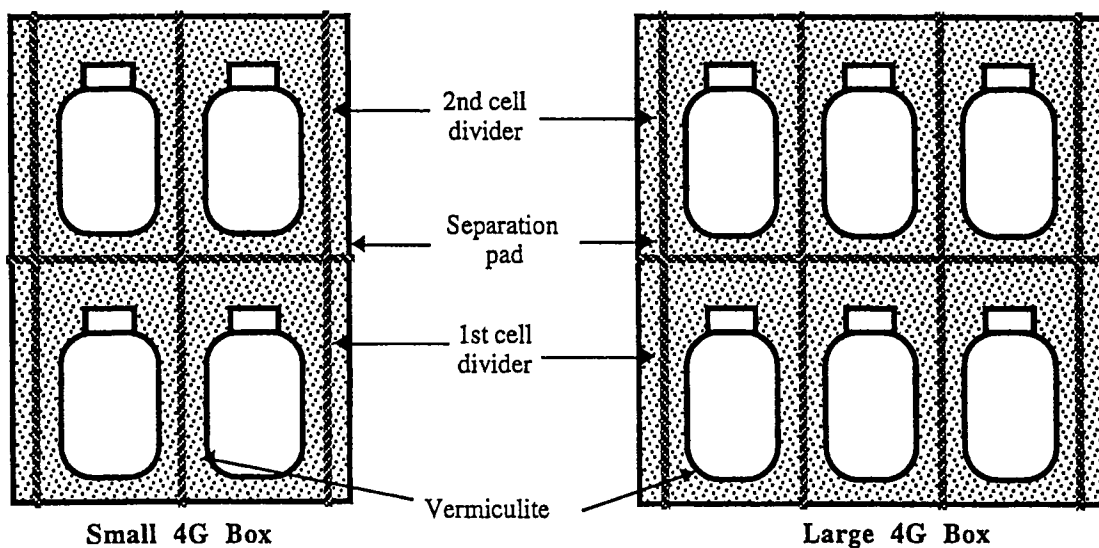
C.A.E. D.G.G. - A current Clean Air Engineering Dangerous Goods Guide

5.0 PROCEDURE

- 5.1 Verify that all containers are properly labeled and have all liquid levels marked.
- 5.2 Teflon tape the lids of the container by wrapping clockwise to keep the lids tightly closed.
- 5.3 Place the container in a 4ml plastic bag, gather extra material at the top, twist clockwise and seal with a Zip tie. One container per bag except for plastic petri dishes, where as many as 8 can be put in one bag.
- 5.4 Assemble 4G box by folding bottom flaps so that the printed manufacturer's information is visible when the bottom of the box is formed.

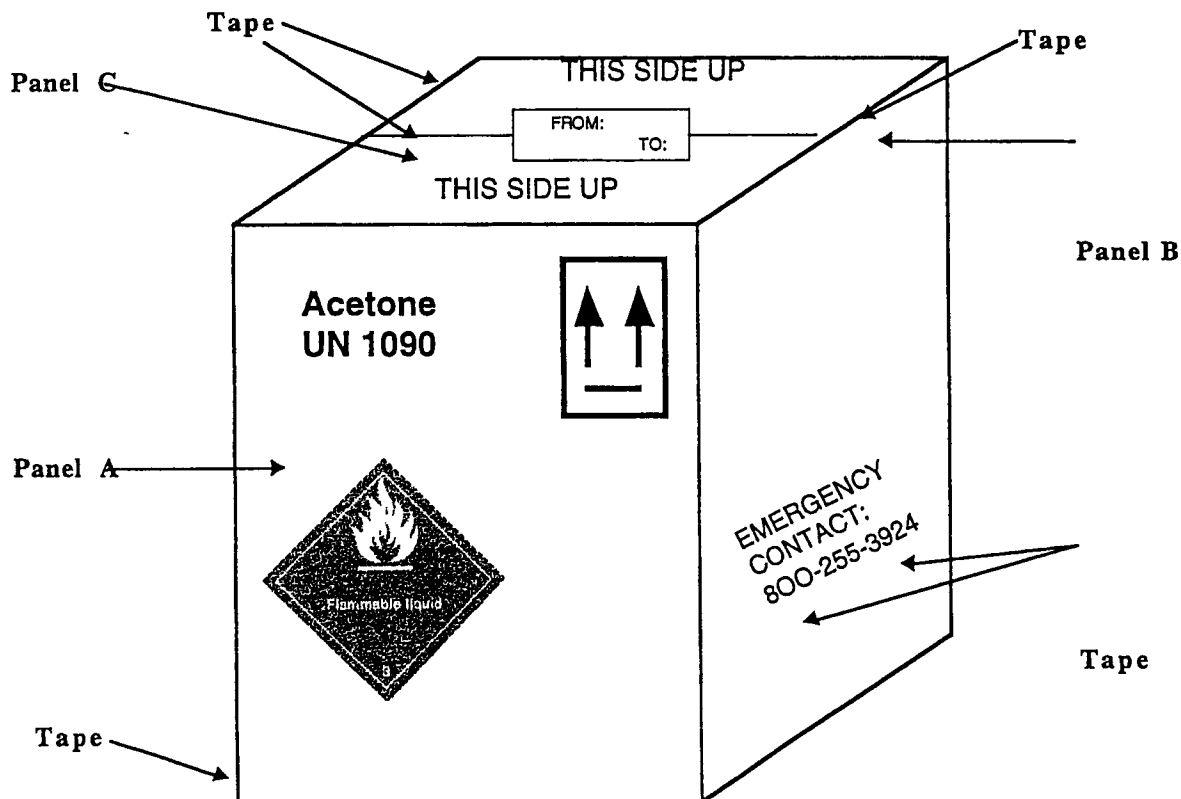
- 5.5 Tape flaps and seal open sides (to keep vermiculite from leaking out) with shipping tape. See Figure 5B
- 5.6 Unfold cell divider and place in the bottom of the box. The cell divider must be flush with the bottom or the box may not close properly.
- 5.7 Add approximately 1" of vermiculite to each of the cells.
- 5.8 Place containers in the cells, then fill around with vermiculite. Do not over fill, vermiculite should be flush with the top of cell divider or box may not close properly.
- 5.9 Place separation pad on top of divider, then add second cell divider. Top of second cell divider must be flush with top of the box or it may not close properly.
- 5.11 Repeat steps 5.6 and 5.7. If all cells are not used, fill empty cells with vermiculite. See Figure 5A
- 5.12 Put completed Chain of Custody in a zip-lock bag and place on top of divider or in an empty cell.
- 5.13 Fold top flaps so that the words "THIS SIDE UP" are visible when box is closed.
- 5.14 Tape flaps and seal open sides(to keep vermiculite from leaking out) with shipping tape.

Figure 5A



- 5.15 On one face of the box with the orientation label, panel "A," print the proper shipping name and the U.N. number (this information is found in the CAE. D.G.G. or IATA D.G.R.) in the space to the left of the orientation label. See Figure 5B.
Note: All markings must be printed and legible.
- 5.16 In the space below the printed information, place the appropriate hazard and handling labels (this information is found in the C.A.E. D.G.G. or IATA D.G.R.).
Note: The labels must not overlap, cover any printing, or wrap around corners
- 5.17 On Panel "B" print the words "Emergency Contact" and underneath print 800-255-3924. See Figure 5B.
- 5.18 Place a shipping label, with a forwarding and return address, on the top panel "C." See Figure 5B

Figure 5B



- 5.19 Check package over to verify that it is sealed, marked, and labeled properly.
- 5.20 Once the package is complete, fill out the proper air bill and Dangerous Goods Shippers Declaration, then contact the carrier.

CAE STANDARD OPERATING PROCEDURE

FIELD ANALYSIS OF HYDROCHLORIC ACID BY MERCURIC NITRATE TITRATION

ISO NUMBER: PMSC-5

EFFECTIVE DATE: Pending

AUTHOR: Dept. 64

APPROVED BY: Pending

1.0 Purpose- The purpose of this procedure is to ensure that any employee performing preliminary analysis of hydrochloric acid by a mercuric nitrate titration in the field, does so in an accurate, consistent manner.

2.0 Scope- This procedure applies to all field personnel who perform mercuric nitrate titrations for the analysis of hydrochloric acid collected by EPA method 26(A).

3.0 Materials- Lab Equipment

- Ring Stand (Rod & Base)
- Burette Clamp
- Self-zeroing Burette Kit
- Stir Plate & Stir Bars
- 250 mL Erlenmeyer Flasks (2 per Titration)
- 50 mL Burette
- Pipettes (1, 2, 5, 10, 20, 50 mL)
- Pipette Bulb
- Dropper Bottle for Indicator Solution
- 100 mL Graduated Cylinder
- Safety Glasses
- HDPE Squeeze Bottles (D.I. Water, 0.05 N Nitric Acid, 0.25 N Sodium Hydroxide)
- Hydrochloric Acid Titration Data Sheets

3.1 Materials - Chemicals-

- De-ionized Water
- 1000 ppm Sodium Chloride Standard
- 0.25 Normal Sodium Hydroxide Solution
- 0.05 Normal Nitric Acid Solution

- Mixed Indicator for Hydrochloric Acid Titrations
- 0.0141 Normal Mercuric Nitrate Titrant

4.0 Definitions-

mL - Abbreviation for milliliters

HCl - Abbreviation for hydrochloric acid

N - Abbreviation for Normality

5.0 Procedure 1 (Standardization of Mercuric Nitrate Titrant)-

- 5.0.1. Pipette exactly 5 mL of 1000ppm sodium chloride standard into an Erlenmeyer Flask.
- 5.0.2. Add de-ionized water to the flask, so that the total volume is 50 mL.
- 5.0.3. Add five to six drops of mixed indicator to the flask. The solution should turn blue.
- 5.0.4. Add 0.05 N nitric acid solution until the solution turns yellow, then add an additional 1 mL of 0.05 N nitric acid.
- 5.0.5. Add 0.0141 N mercuric nitrate titrant drop wise from the burette until the solution turns purple. (Approximately 10 mL)
- 5.0.6. Repeat the titration (Steps 5.0.1 through 5.0.5) until two consecutive trials agree within ± 0.2 mL of each other.
- 5.0.7. Record results on the titration data sheet and determine the Normality of the titrant using the following equation:

$$\text{Normality of titrant} = \frac{(\text{ppm of Sodium Chloride Standard}) \times (\text{Milliliters of Standard})}{(\text{Milliliters of Titrant}) \times (35,450)}$$

5.1 Procedure 2 (Titrating a Sample)

- 5.1.1. Pipette an exact amount of sample into an Erlenmeyer flask. The amount used should reflect an approximation of hydrochloric acid levels in the sample.

Example: A 1 or 2 mL aliquot of sample might be used to titrate an inlet sample, since the hydrochloric acid levels could be high, while a 20 or 50 mL aliquot might be used to titrate an outlet sample where the levels would be lower.

- 5.1.2. Add de-ionized water to the sample so that the volume in the flask is 50 mL.
- 5.1.3. Add five to six drops of mixed indicator to the flask. The solution should turn yellow.
- 5.1.4. Add 0.25 N sodium hydroxide solution to the flask until the solution turns a red/blue color.
- 5.1.5. Add 0.05 N nitric acid solution to the flask until the solution turns back to yellow. Add an additional 1 mL of 0.05 N nitric acid to the flask.

- 5.1.6. Add 0.0141 N mercuric nitrate drop wise from the burette until the solution turns purple.
- 5.1.7. Repeat the titration (Steps 5.1.1 through 5.1.6) until two consecutive trials agree within ± 0.2 mL of each other.
- 5.1.8. Record all data.

6.0 References-

- 6.1 Method 325.3

7.0 Attachments-

- 7.1 Hydrochloric Acid Titration Data Sheet

TITRATION FOR HYDROGEN CHLORIDE (METHOD 26)

ISO NUMBER: LAB-14

EFFECTIVE DATE: Pending

AUTHOR: Dept. 68

APPROVED BY: Pending

a. materials needed

- samples with volumes
- Erlenmeyer flasks
- 2-5-10-20-25-50 ml pipette
- pipette bulb
- 10 and 50 ml burettes
- squeeze bottles
- burette stand and holder
- stir plate and bar receiver (optional)

b. procedure

1. Dilute 0.14 N Mercuric Nitrate at a 1-9 ratio to generate 0.014 N solution
2. Rinse burette with 0.014 N Mercuric Nitrate solution
3. Prepare data sheet. (Note sample volume)
4. Pipette standard amount (5-10ml for inlets ; 20-30ml for stacks) into Erlenmeyer flask. Prepare two trails
5. Add 4-5 drops of indicator
6. If solution is blue red add 0.1 N HNO_3 dropwise until yellow and then add 1 ml in excess
If solution is yellow after adding the indicator, add NaOH solution dropwise until blue-red. Then add 0.1 N HNO_3 until yellow and add 1 ml in excess.
7. Titrate to purple end point.

NOTE: Standardize titrant daily. (5ml NaCl will require 8-10 ml titrant. Standardize in triplicate and use the average to determine the normality.

CAE STANDARD OPERATING PROCEDURE

ANALYSIS OF SULFUR DIOXIDE (SO₂) BY BARIUM PERCHLORATE TITRATION

ISO NUMBER: PMSC-6

AUTHOR: Dept. 64

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1.0 Purpose- The purpose of this procedure is to ensure that any employee performing barium perchlorate titrations for the analysis of sulfur dioxide (SO₂) does so in an accurate, consistent manner.

2.0 Scope- This procedure applies to all analysts and field personnel who perform barium perchlorate titrations for the analysis of SO₂.

3.0 Materials- Lab Equipment

- Ring Stand (Rod & Base)
- Burette Clamp
- Self-zeroing Burette Kit
- Stir Plate & Stir Bars
- 250 mL Erlenmeyer Flasks (2 per Titration)
- 50 mL Burette
- Pipettes (5, 10, 20 ,25 mL)
- Pipette Bulb
- 100 mL Volumetric Flask
- 500 mL Volumetric Flask
- 1000 mL Volumetric Flask
- Dropper Bottle for Indicator Solution
- 100 mL Graduated Cylinder
- Safety Glasses

- HDPE Squeeze Bottles (100% isopropanol, De-ionized Water)
- Method 6 Titration Data Sheets

3.1 Materials - Chemicals- *Note:* The numbers in parenthesis that follow a chemical name refer to EPA Method 6 instructions for preparation of that chemical. All numbers reference 40 CFR Part 60 Appendix A Method 6.

- De-ionized Water
- 0.01 N Sulfuric Acid Standard
- 3% Hydrogen Peroxide (3.1.3)
- EPA Audit Samples
- 100% Isopropanol
- Thorin Indicator (3.3.3)
- 0.01 N Barium Perchlorate Titrant (3.3.4)

4.0 Definitions-

mL - Abbreviation for milliliters

SO₂ - Abbreviation for sulfur dioxide

5.0 Procedure 1 (Standardization of Barium Perchlorate Titrant)-

- 5.0.1. Pipette exactly 25 mL of 0.01 Normal sulfuric acid standard into an Erlenmeyer Flask.
- 5.0.2. Add 100 mL of 100% isopropanol to the flask, so that the total volume is 125 mL.
- 5.0.3. Add three to four drops of thorin indicator to the flask. The solution should turn yellow.
- 5.0.4. Carefully add 0.01 Normal barium perchlorate titrant dropwise from the burette into the flask until the solution turns salmon pink. (approximately 25 mL)
- 5.0.5. Repeat the titration (Steps 5.0.1 through 5.0.4) until two consecutive titrations agree within ± 0.2 mL of each other.
- 5.0.6. Record results on the titration data sheet and determine the Normality of the titrant using the following equation:

$$\text{Normality of titrant} = \frac{(\text{Normality of Standard}) \times (\text{Milliliters of Standard})}{(\text{Milliliters of Titrant})}$$

5.1 Procedure 2 (EPA Audit Sample Analysis)-

- 5.1.1. Break the glass vial containing the audit solution at the line.
- 5.1.2. Pipette exactly 5.0 mL of audit solution from the vial into a 100 mL volumetric flask.
- 5.1.3. Add 30 mL of 3% hydrogen peroxide to this solution.
- 5.1.4. Dilute to the mark on the flask with de-ionized water.
- 5.1.5. Pipette exactly 20 mL of the solution into a clean Erlenmeyer flask.
- 5.1.6. Add 80 mL of 100% isopropanol.
- 5.1.7. Add three to four drops of thorin indicator. The solution should turn yellow.
- 5.1.8. Add 0.01 Normal barium perchlorate dropwise from the burette into the flask until the solution turns salmon pink.
- 5.1.9. Repeat the titration (Steps 5.1.5 through 5.1.8) until two consecutive titrations agree within ± 0.2 mL of each other.
- 5.1.10. Prepare a 100 mL blank solution following Steps 5.1.3-5.1.4. Do not add any audit solution to the blank solution. Titrate the blank solution the same way the audit solution is titrated.
- 5.1.11. Using the following equation determine the concentration of sulfur dioxide in the EPA Audit sample.

$$C = \frac{K(N \times (V_t - V_{tb}) \times (V_{soln}/V_a))}{V_{mstd}}$$

Where:

C = concentration of SO₂
K = 32.03 mg/meq
N = normality of titrant
V_t = volume of titrant used to titrate the audit sample solution
V_{tb} = volume of titrant used to titrate the blank solution
V_{soln} = volume of the audit solution (100 mL)
V_a = volume of aliquot titrated (20 mL)
V_{mstd} = 0.021 dscm

- 5.1.12. Compare the calculated SO₂ concentration to the EPA concentration value. The calculated value must be within 5% of the EPA value.
 - a) If the value is not within 5% of the EPA value, the audit titrations, steps 5.1.5 to 5.1.8 must be repeated until it is.
 - b) If the value is within 5% of the EPA value, continue to procedure 3.

5.2 Procedure 3 (Titrating a Sample)

- 5.2.1. Pipette exactly 20 mL of sample into a clean Erlenmeyer flask.
- 5.2.2. Add 80 mL of 100% isopropanol.
- 5.2.3. Add three to four drops of thorin indicator. The solution should turn yellow.

- 5.2.4. Add barium perchlorate dropwise from burette until the solution turns salmon pink.
- 5.2.5. Repeat titration (Steps 5.2.1-5.2.4) until two consecutive titrations agree within ± 0.2 mL of each other.
24. Record all data on the Method 6 titration data sheet.

6.0 References-

- 6.1 EPA Method 6 - 40 CFR Part 60 Appendix A Method 6

7.0 Attachments-

- 7.1 Method 6 Titration Data Sheet

TITRATION FOR SO₂ (METHOD 6)

ISO NUMBER: LAB-15

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

a. Materials needed:

- samples
- 100 ml volumetric flask
- 4- 250 ml Erlenmeyer flasks
- 5- 10- 20 ml pipettes
- pipette bulb
- 10 and 50 ml burettes
- 100 ml graduated cylinder
- 250 ml bottle
- 2- 500 ml chemical squeeze bottles
- Burette stand and holder
- stir plate, bar, and retriever

b. Procedure

1. Obtain test sample (3% H₂O₂ and SO₂). Note liquid level.
2. Pour sample into a 100 ml volumetric flask and bulk to 100 ml with DI water.
3. Pipette 20 ml; aliquot into a 250 ml Erlenmeyer flask.
4. Add 80 ml of 100% IPA and about 2-6 drops of Thorin indicator.
5. Add stir bar and titrate sample with .01 N BaCl₂ until pinkish endpoint. Note volume of titrate used.
6. Repeat steps 3-5 and obtain an average of two trials.

NOTE: Replicate samples must titrate to within .2 ml of each other.

CAE STANDARD OPERATING PROCEDURE

SILICA GEL HANDLING

ISO NUMBER: LAB-1

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1. PURPOSE

To provide detailed information on the procedure for mixing, weighing, and baking silica gel.

2. SCOPE

This procedure applies to those individuals involved in either the mixing, weighing, or baking of silica gel.

3. MATERIALS

- Silica gel
- Indicator
- Leak free container
- Oven
- Gloves
- Baking dish
- Analytical Balance
- Sample bottles
- Duct tape
- Dust mask
- Funnel
- Large measuring cup
- Small measuring cup
- 500ml sample bottles

4. DEFINITIONS

4.1 Silica Gel: Granuales that absorb moisture

4.2 Indicator: Blue granuales that change color when moisture is present.

4.3 Leak free container: A bucket with a lid that is equipped with a rubber gasket which when tightened on the bucket, a leak free seal is made.

5. PROCEDURE

5.1 Mixing

- 5.1.1 Obtain a leak free container.
- 5.1.2 Put on dust mask.
- 5.1.3 Using large measuring cup inside 55 gallon drum of silica gel, dump silica gel into choosen leak free container.
- 5.1.4 Using small measuring cup inside indicator can, transfer approximately 1 cup of indicator into container with silica gel.
- 5.1.5 Mix together to evenly distribute indicator throughout gel.
- 5.1.6 Repeat last three steps until desired amount is prepared or container is full.

5.2 Weighing

- 5.2.1 Obtain 500ml sample bottles (Use the ones designated for this purpose).
- 5.2.2 Zero the analytical balance.
 - 5.2.2.1 Press the tare button.
- 5.2.3 Place the sample **bottle and the lid** on the balance pan.
- 5.2.4 Using a funnel, fill the bottle with silica gel until weight equals 300g.
- 5.2.5 Remove bottle from the balcance and put the lid on it.
- 5.2.6 Place a strip of duct tape over the top and down the sides of the bottle. (This tell anyone that the silica gel inside is ready for use, i.e., the total weight is 300g).
- 5.2.7 After silica gel has been used, it should be weighed without the tape.
 - 5.2.7.1 The duct tape may be placed around the side of the bottle to record test run and location.
 - 5.2.7.2 If little moisture was collected (less than 10ml) the silica may be used again. However, you must record the new gross weight of the silica and bottle (including the lid) after the first run, so that the moisture of the second run may be calculated corectly.
- 5.2.8 When the tape is around the bottle or totally removed, this indicates that the gel has been used and needs to be rebaked and refilled.

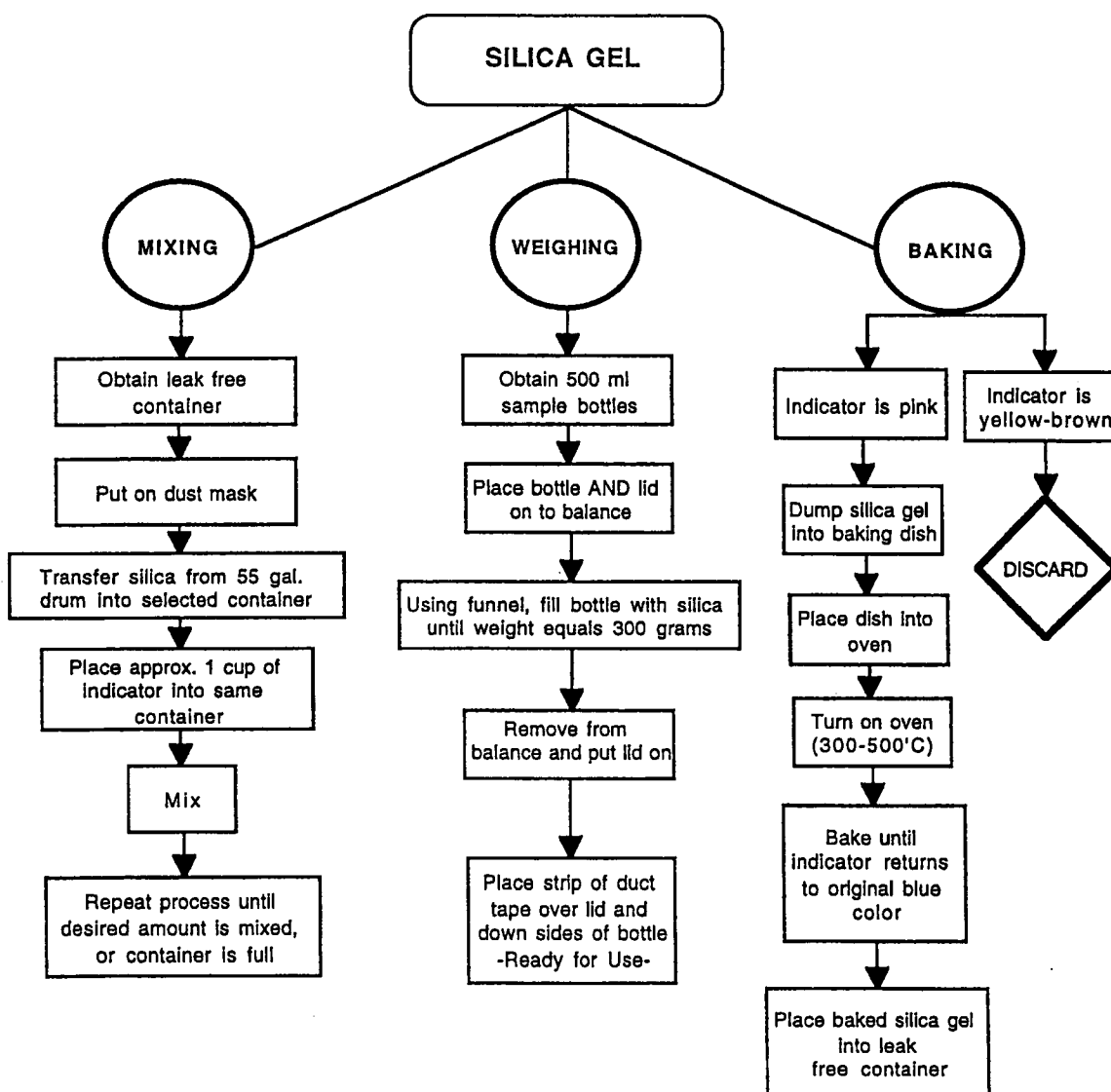
5.3 Baking

- 5.3.1 When silica gel indicator turns a pinkish color, it is time to bake the gel so it can be reused. (If indicator has turned yellow or black, silica gel should be discarded).
- 5.3.2 Obtain used silica gel.
- 5.3.3 Dump used silica gel into a baking dish (do not fill more than 1 inch

thick).

- 5.3.4 Place dish into oven that is designated for this purpose.
- 5.3.5 Turn on oven (300-500°C).
- 5.3.6 Bake for 20-45 minutes or until indicator has returned to its original blue color. (Time depends on oven temperature and the amount of gel. Adjust one or both parameters in order to keep the gel from burning).
- 5.3.7 Place baked silica gel into leak free container for later use.

6. PROCESS FLOW DIAGRAM



7. ATTACHMENTS

CAE STANDARD OPERATING PROCEDURE

**GLASSWARE PREPARATION PROCEDURE- EPA METHODS 5,
5A, 5B, 5D, 5F, 5G, 5H, 6, 8, 13A(B), 17, 26, 26A**

ISO NUMBER: LAB-4

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

Purpose: The purpose of this procedure is to document the steps taken in the preparation of glassware for Environmental Protection Agency air quality testing Methods 5, 5A, 5B, 5D, 5F, 5G, 5H, 6, 8, 13A(B), 17, 26, and 26A.

Scope: This procedure will be used by lab personnel (in the Glassware Prep./ Wet Chem. area) and will cover glassware preparation for EPA reference methods 5, 5A, 5B, 5D, 5F, 5G, 5H, 6, 8, 13A(B), 17, 26, and 26A.

Materials: The materials needed for this procedure may vary slightly depending on degree of initial cleaning required. They are as follows:

- Glassware
- Dishwasher
- Dry-Contrad, Alcojet, or equivalent non-foaming lab detergent
- A sink with hot running water
- Various size brushes
- D.I. water
- Omnisolve® (or equivalent) Acetone
- 30% Hydrogen Peroxide
- Large glass beaker
- Large plastic tray
- Kim-wipes
- Laboratory fume hood
- Teflon squeeze bottle
- Glass baking dish
- Polypropylene lab gloves
- Safety glasses
- Lab coat

Definitions: B/S: This abbreviation refers to ball and socket type glassware.

D.I.: This abbreviation refers to de-ionized water

Glassware: Any glass apparatus that is stored in the Glassware Prep/Pack area and is used for field testing. It may also include non-glass fittings (ex: caps or frits or rings) that are associated with certain glassware items.

GLASSWARE PREPARATION PROCEDURE- EPA METHODS 5, 5A, 5B, 5D, 5F, 5G, 5H, 6, 8, 13A(B), 17, 26, 26A**PAGE 2**

Lab: This term will be used to refer to the Glassware Prep/Packing area.

Prep.: This abbreviation refers to the word preparation.

Preparation: The actual cleaning, chemical rinsing, and capping process done to the various glassware components used for EPA air quality testing methods according to the applicable method.

Procedure:

Third.: This abbreviation refers to threaded type glassware. The following steps are taken in the preparation of glassware to be used for EPA reference methods 5, 5A, 5B, 5D, 5F, 5G, 5H, 6, 8, 13A(B), 17, 26, and 26A.:

Please note that all appropriate safety precautions must be followed for all glassware preparation steps, including use of a lab coat, polypropylene gloves, and safety glasses.

1. Removal of stains and/or grease. If the glassware is ball and socket type, the first step must be the complete removal of all silicone grease. Use kim-wipes or paper towels and completely remove all traces of grease from the glassware. There should be no visible grease left on any part of the glassware. Next, look for stained glassware. If there are brown or purple stains on the glassware, they must be removed with 30% hydrogen peroxide before any other steps can be taken. The glassware should be brought to the fume hood. 30% hydrogen peroxide (H_2O_2) found in the lab refrigerator can then be poured over the stained areas into a beaker until the stains are gone. Soaking or brushing may be necessary for tougher stains. A glass baking dish can also be used to catch the H_2O_2 . CAUTION: 30% H_2O_2 is harmful to human skin. Use gloves, safety glasses, and a lab coat or apron. Rinse everything with water when done.

2. Removal of Chemical residues. When the obvious dark stains have been removed from the glassware, look for other evidence of stains or chemicals in or on the glassware. Any glassware with this condition should be rinsed with tap water in the sink before moving on to step 3.

3. Loading the dishwasher. Once stains and chemicals have been removed from the glassware it can then be loaded into the dishwasher. A Labconco Flaskscrubber laboratory dishwasher is the preferred dishwasher. This unit has certain features which should be utilized. Gently place impinger bottles upside down over the stainless steel spindle jets. Other items can be loaded where they fit best. Do not overload the dishwasher to a point where glassware is squeezed in or it could break. Make sure no pieces are protruding below the tray or blocking the jet gasket in the rear of the tray.

4. **Using the dishwasher.** When the tray is full, gently slide the tray into the dishwasher until the gasket on the tray seats itself to the back of the unit. Do not use force to push in the tray. Once it is in, fill both detergent trays on the inside of the door up to the fill line with a non-foaming powder detergent; Dry-Contrad, Alcojet, or equivalent. If the scoop is used, one scoop is sufficient (half a scoop per tray- two trays, one scoop). Swing the detergent tray closed and close the dishwasher door. To date, it seems to work best with the steam function on, D.I. rinse on, normal cycle wash (heavy if you think it needs it), and air or heat dry.

4a. NOTE FOR CLEANING WITH STANDARD

AUTOMATIC DISHWASHER: After the glassware has gone through a complete washing, each piece must have all surfaces that come into contact with the sample brushed and rinsed three times with hot tap water and then rinsed three times with D.I. water.

4b. NOTE FOR CLEANING WITH NO AUTOMATIC

DISHWASHER: All glassware must be soaked in hot soapy tap water. All surfaces that come into contact with the sample must be brushed and rinsed three times with hot tap water, then rinsed three times with D.I. water. Better yet, order a dishwasher from Sears.

5. Unload dishwasher and inspect glassware. Unload the dishwasher and put the glassware on a cart or tray. Inspect each piece as it is being unloaded for dirt and stains or cracks and chips. Any dirty or stained pieces should be taken back to step 1. Any cracked, chipped, or broken glassware should be removed and repaired or replaced.

6. Acetone rinse. The glassware can now be brought to the hood for acetone rinsing. Fill a 500ml or larger Teflon squeeze bottle with acetone. (Nalgene bottles leave residue.) Omnisolve® grade acetone or equivalent is recommended, although A.C.S. grade is sufficient for Method 5. Place a large glass baking dish on the working surface in the fume hood to catch the used acetone. Proceed to rinse the glassware with the acetone. Each piece must be rinsed three times and all surfaces that come into contact with the sample must be rinsed. The used acetone should end up in the glass baking dish. (when dish gets about half full of acetone, carefully move the dish to the evaporation hood and place inside the hood. Take an empty baking dish back to the rinsing hood and continue.)

7. Dry and cap. Allow the glassware to air dry inside the fume hood. When the acetone has all evaporated and the glassware is dry, the final step is capping. All orifices must be stuffed with kim-wipes. Stems can be put in bottles using a nut and gasket if

job is not going to be shipped. If there are more impinger stems than impinger bottles, or if job is being shipped, stuff the threaded end of the stem with a kim-wipe and then wrap the stem in plastic wrap. Plastic wrap is a suitable cap, but please note that kim-wipes must be used somewhere on each piece of glassware to differentiate it between a method 5 prepped piece of glassware and a metals prepped piece of glassware.

GLASSWARE PREPARATION

ISO NUMBER: LAB-13

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

Glassware cleaning safety procedure

Due to the hazardous nature of the chemical used to clean glassware, strict adherence to the safety procedure must be observed. The following chemicals are used to clean glassware. Please see the recommended glassware cleaning procedure for their method specific use.

<u>CHEMICAL</u>	<u>HAZARD</u>
10% Potassium hydroxide	Corrosive
10% Nitric acid	Corrosive
50% Nitric acid	Corrosive
8N hydrochloric acid	Corrosive
Acetone	Volatile
Methylene Chloride	Volatile

When working with corrosive chemicals the following is to be worn:

- goggles or full face shield
- full body apron
- rubber gloves

All spills are to be neutralized and cleaned up at once. Boxes of baking soda are kept in the acid wash area for this reason.

When working with volatile compounds the following is to be worn:

- goggles or full face shield
- rubber gloves
- respirator with organic vapor cartridges

All volatile organics should be collected in a glass sample jar or waste jar for later disposal. Do not dump them down the drain.

All glassware cleaning areas should be well ventilated, preferably under the fume hood.

Glassware that is in-between rinses of DI and acetone or still needs rinsed with acetone or whatever, should be labeled as such. It helps to keep things organized.

a. GENERAL PREPARATION FOR ALL GLASSWARE**1. Removal of stains and/or grease**

If glassware is ball and socket type, the first step is to remove all silicone grease. Use Kim-wipes or paper towels and completely remove all traces of grease from glassware. Next, look for stained glassware. If there are brown or purple stains on the glassware, they must be removed with 3% Hydrogen

Peroxide (H_2O_2) before any other steps are taken. 3% H_2O_2 can be poured over stained area until stain is gone. Soaking or brushing may be necessary for tougher stains. Rinse everything with water when done.

2. Removal of chemical residue

When the obvious dark stains have been removed, look for evidence of stains or chemicals in or on the glassware. Rinse off any other stains before continuing

3. Preparation

Wash each piece of glassware in hot soapy water. Rinse 3 times with hot tap water, 3 times with DI water, and Drain well.

b. PARTICULATE PREPARATION

If preparing glassware for EPA Methods 5,5A, 5B, 5D, 5F, 5G, 5H, 6, 8, 13A(B), 17, 26, or 26A

Perform general glassware prep. and then...

1. Rinse each piece of glassware 3 times with acetone. All surfaces that may come in contact with a sample must be rinsed.

NOTE: Acetone rinsing should be done under the laboratory hood. Used acetone should be collected in a glass baking dish. It then should be either poured into a 4L glass bottle and saved for cleaning purposes or it should be evaporated under the hood.

2. Allow to air dry
3. Cap all glassware openings with kimwipes.

c. METAL PREPARATION

If preparing glassware for EPA Methods 12, 101A, 104, 108, or multimetals(29)
Perform general glassware prep. and then ...

1. Remove large screw caps from impinger bottles. Submerge glassware in a 10 % Nitric acid bath that has been prepared earlier. Remove any air bubbles to make sure all surfaces are covered. Soak in bath for four (4) hours or more.

NOTE: 10% Nitric acid is corrosive. Use safety glasses, rubber gloves and labcoat or apron. Use caution.

2. Remove from Nitric acid bath and drain well. Rinse all surfaces with DI water and drain.
3. Rinse each piece of glassware 3 times with acetone. All surfaces that may come into contact with a sample must be rinsed.

NOTE: Acetone rinsing should be done under the laboratory hood. Used acetone should either be dumped into a glass baking dish and evaporated or it should be stored in a glass bottle and saved for cleaning purposes.

4. Allow to air dry
5. Cap all openings with parafilm.

d. DIOXIN PREPARATION

If preparing glassware for EPA Methods 23, 0010, or CARB 429
Perform general glassware preparation and then ...

1. Heat glassware in oven at 450 F for 2 hours. Allow to cool.
2. Rinse each piece of glassware 3 times with acetone (under the hood). All surfaces that may come into contact with a sample must be rinsed.
3. Allow to air dry
4. Rinse each piece 3 times with Methylene Chloride (MeCl_2). All surfaces that may come into contact with a sample must be rinsed. MeCl_2 rinsing should be done under the hood, and waste should be evaporated.
CAUTION: Methylene Chloride (MeCl_2) is a controlled substance. If you can smell it you are being over exposed. Refer to MSDS.
4. Allow to air dry.
5. Cover all glassware openings with MeCl_2 rinsed aluminum foil (dull side down).

e. HexCr Cr^{+6} PREPARATION

If preparing glassware for EPA Method Cr^{+6} , perform the general glassware procedure and then...

Follow Metals preparation procedures (c.)

f. METHOD 202 PREPARATION

If preparing glassware for EPA Method 202, perform the general glassware procedure and then...

1. Rinse each piece of glassware 3 times with acetone (under the hood).
2. Rinse each piece 3 times with Methylene Chloride (MeCl_2).

CAUTION: Methylene Chloride (MeCl_2) is a controlled substance. If you can smell it you are being over exposed. Refer to MSDS.

3. Allow to air dry.
4. Cover all glassware openings with MeCl_2 rinsed aluminum foil (dull side), and mark "202" on the foil with a sharpie.

g. METHOD BIF 0011 (FORMALDEHYDE) PREPARATION

If preparing glassware for EPA Method BIF 0011, perform the general glassware procedure and then...

1. Rinse each piece of glassware 3 times with acetone (under the hood).
2. Rinse each piece 3 times with Methylene Chloride (MeCl_2).

CAUTION: Methylene Chloride (MeCl_2) is a controlled substance. If you can smell it you are being over exposed. Refer to MSDS.

3. Allow to air dry.
4. Cover all glassware openings with MeCl_2 rinsed aluminum foil (dull side), and mark an "F" on the foil with a sharpie.

h. VOST PREPARATION

NOTE: Glassware for this method should not be cleaned or kept where organic solvents are used.

1. Wash with non-ionic detergent.
2. Rinse 3 times with HPLC water.
3. Heat at 230°F for one hour.
4. Wrap in aluminum foil.

i. CARB 428 PREPARATION

If preparing glassware for EPA Method CARB 428, perform the general glassware procedure and then...

1. Heat at 450°F for 2 hours.
2. Rinse each piece of glassware 3 times with acetone (under the hood).
3. Rinse each piece 3 times with Methylene Chloride (MeCl_2).
4. Rinse each piece 3 times with Toluene.
5. Allow to air dry.
6. Cover all glassware openings with MeCl_2 rinsed aluminum foil (dull side), and mark 428 on the foil with a sharpie.

CAE STANDARD OPERATING PROCEDURE

PREPARING PROBE BRUSHES

ISO NUMBER: LAB-16

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

Probe brushes that have a nylon brush on the end are to be used for particulate testing.

a. TO PREP. A PARTICULATE PROBE BRUSH...

1. Wash in hot soapy water.
2. Rinse 3 times with hot tap water
3. Rinse 3 times with DI water.
4. Rinse 3 times with acetone. (This may be done by squeezing acetone onto some kimwipes and running it along the length of the probe). By the end of the third rinse, no visible dirt or dust should be left on the kimwipes.

Probe brushes that have square teflon brushes on the ends may be used for either Metal or Dioxin testing.

b. TO PREP. A DIOXIN PROBE BRUSH...

1. Wash in hot soapy water.
2. Rinse 3 times with hot tap water.
3. Rinse 3 times with DI water.
4. Rinse 3 times with acetone. (Follow step 4 for particulate prepping).
5. Rinse 3 times with MeCl_2 (Use the same method as used with particulate acetone rinsing- Step 4).

c. TO PREP A METAL PROBE BRUSH...

1. Wash in hot soapy water.
2. Rinse 3 times with hot tap water.
3. Remove brush from probe.
4. Soak probe in 10% Nitric acid for 4 hours.
5. Remove from Nitric bath. Drain well and rinse with DI.
6. Rinse 3 times with acetone (Using the same method as used with particulate acetone rinsing -Step 4).

d. MEASURING PROBE BRUSHES

When measuring probe brushes, you must add 9-11" on to the probe length that is requested. This will be referred to as the "effective" length. EX. If asked for a 6 foot effective probe brush, the probe must actually measure between 6'9"- 6'11" . Refer to - "Liner Lengths for M5 Probes" (e) . After probe brushes are measured, place each in a ziplock bag, which is labeled with the effective length, method in which it is prepped for, and the date.

e. LINER LENGTHS FOR M-5 PROBES

2' EFFECTIVE	33 - 35"
3' EFFECTIVE	45 - 47"
4' EFFECTIVE	57 - 59"
5' EFFECTIVE	69 - 71"
6' EFFECTIVE	81 - 83"
7' EFFECTIVE	93 - 95"
8' EFFECTIVE	105 - 107"
9' EFFECTIVE	117 - 119"
10' EFFECTIVE	129 - 131"

CAE STANDARD OPERATING PROCEDURE

COMPOSITE COAL SAMPLES

ISO NUMBER: LAB-17

AUTHOR: Dept. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1. PURPOSE

To provide detailed information on the procedure for making a composite coal sample.

2. SCOPE

This procedure applies to those individuals involved in the making of composite coal samples.

3. MATERIALS

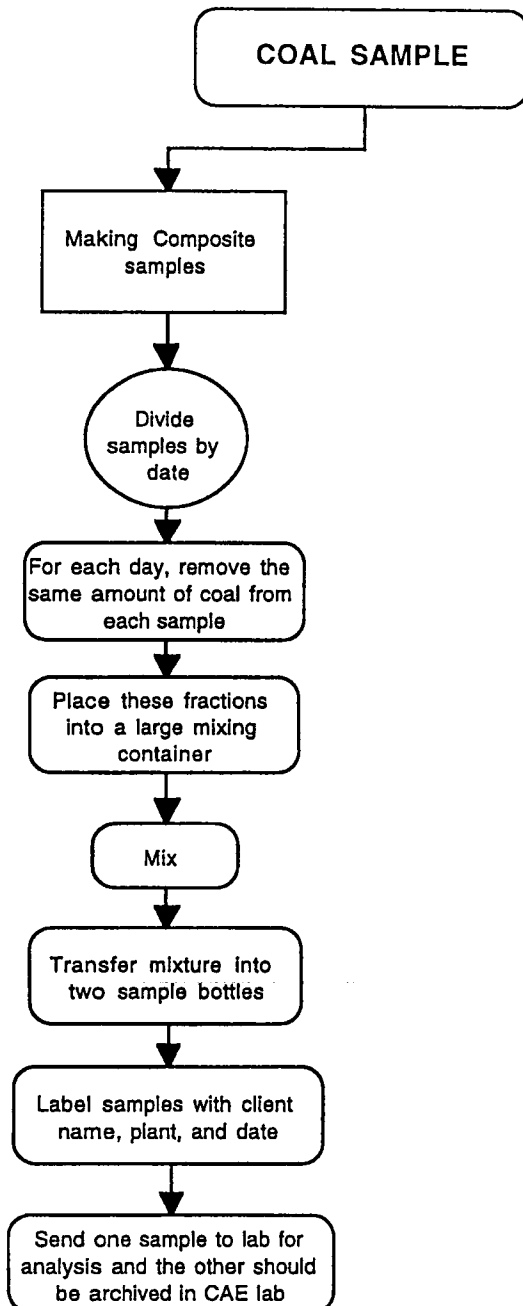
- Large mixing container with a lid
- Gloves
- Sample bottles
- Measuring cup
- Sharpie pen
- Teflon tape

4. PROCEDURE

- 4.1.1 Separate coal samples by what day they were collected.
- 4.1.2 For each day, remove approximately two cups of coal from each individual sample using a measuring cup. Samples from different locations may be mixed together if obtained on the same test date.
- 4.1.3 Place each measured amount into a large clean container with a lid.
- 4.1.4 Shake or stir to completely mix samples together.
- 4.1.5 Transfer this "composite" sample into two sample containers.
- 4.1.6 Label each container with a sharpie pen.
 - 4.1.6.1 Include Client name, Location of Plant and the date the sample was obtained.
- 4.1.7 Wrap lids on containers with teflon tape.
- 4.1.8 Send one sample to the lab for analysis and archive the other in the CAE lab.

Coal samples should not be brought into the lab. This process can be very messy and should be performed in the workshop area.

5. PROCESS FLOW DIAGRAM



ARCHIVING LABORATORY SAMPLES

ISO NUMBER: PSPL-1

AUTHOR: Dept. 64

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1.0 Purpose- The purpose of this procedure is to ensure that laboratory samples are archived in an accurate consistent manner.

2.0 Scope- This procedure applies to any employee archiving samples in the explosion room located behind sample receiving.

3.0 Materials-

- Sample Archive Log
- Black Pen
- Sample Archive Labels
- Cardboard Box of Suitable Size
- Black Marker

4.0 Definitions-

I.C.-Abbreviation for Ion Chromatography.

5.0 Procedure-

- 5.1. Place all samples to be archived in the cardboard box. Although it isn't required, and sometimes not even possible, it is convenient to archive all samples from the same job in one box.
- 5.2. Completely fill out a sample archive label. In the section of the label where it reads "Disposal Method" a colored dot should be drawn or a colored sticker should be placed to indicate one of three categories.
 - a) **Green-** A green label indicates that the sample is generally not considered hazardous and when disposed of, can be dumped down the drain if a liquid or thrown in the trash if a solid (Examples include most IC. samples, filters and thimble catches.)
 - b) **Yellow-** A yellow tag indicates that the sample should be disposed of by evaporation. (Examples include acetone rinses and dichloromethane rinses.)
 - c) **Red-** A red tag indicates that the sample is hazardous and needs to be disposed of either by neutralization or per special instructions. (If the sample needs to be disposed of specially, detailed instructions should be attached.) A red tag may indicate that the sample can not be disposed of by CAE. If this is the case a note should be attached to the archive tag and written in the Sample Archive Log.
- 5.3. Fill out the information on the line(s) in the sample archive log. Under box number, put the number corresponding with the current month, dash, the number

corresponding with the next year, dash, the sequential number of the box archived in that month.

(**Example 1:** The first box of samples archived in June of 1996 would be indicated by the number code "6-97-1".)

(**Example 2:** The seventh box of samples archived in December of 1997 would be indicated by the number code "12-98-7".)

(**Example 3:** The twenty-eighth box of samples archived in April of 2003 would be indicated by the number code: "4-04-28".)

- 5.4. Write the number code on the corresponding box of samples, so it can be easily identified.
- 5.5. Put the box of samples in the explosion room on a shelf near samples archived during the same month.
- 5.6. Return sample archive log to the explosion room.

6.0 Attachments-

- 6.1 Blank Page from the Sample Archive Log

CAE STANDARD OPERATING PROCEDURE

DISPOSAL OF LABORATORY SAMPLES

ISO NUMBER: PSPL-2

AUTHOR: Dept. 64

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1.0 Purpose- The purpose of this procedure is to ensure that any employee disposing of laboratory samples does so in a safe, consistent manner that will impact the environment as little as possible.

2.0 Scope- This procedure applies to most of the samples generated in CAE's Gravimetric and Ion Chromatography Laboratories.

3.0 Materials-

- Lab Gloves
- Lab Coat
- Lab Apron
- Safety Glasses (Goggles if Needed)
- Laboratory Fume Hood
- Large Plastic Bucket or Cooler
- pH Paper
- Sodium Bicarbonate (Baking Soda)
- Acid

4.0 Definitions-

I.C. - Abbreviation for Ion Chromatography

5.0 Procedure-

Note¹: These procedures are to be performed only after samples have been in storage for at least one year. See the Procedure for Archiving Laboratory Samples for more information or to answer any questions about sample storage.

Note²: The disposal of samples should be recorded in the Sample Archive Log under the appropriate heading and for the corresponding CAE project.

5.0 Procedure 1 - Disposal of Green Tagged Samples

- 5.0.2. Samples with a green disposal tag are usually water samples, dilute solutions of acids and bases (i.e. 0.1 Normal or less) dilute solutions of hydrogen peroxide, filters, road silt and thimble catches.
- 5.0.2. Water samples and dilute solutions of acids, bases and hydrogen peroxide can be poured down the drain and flushed with water.
- 5.0.3. Filters, road silt and thimble catches can be disposed of into a landfill.

5.1 Procedure 2 - Disposal of Yellow Tagged Samples

- 5.1.1. Samples with a yellow disposal tag are usually organic solvents such as acetone, dichloromethane, ether, toluene and isopropyl alcohol.
- 5.1.2. Acetone, dichloromethane and other solvents can be evaporated under a fume hood.
- 5.1.3. Isopropyl alcohol can be evaporated under a fume hood, or, since it is water soluble, it can be poured down the drain and flushed with water.

5.2 Procedure 3 - Disposal of Red Tagged Samples

- 5.2.1. Samples with a red disposal tag are usually stronger acids and bases (i.e. greater than 0.1 Normal) which can be neutralized, or samples which can not be disposed of at CAE. Further information should be obtained with these samples.

Neutralization

- 5.2.2. Neutralization should be performed under a fume hood. This reaction generates heat and may splatter, so it should be performed slowly in a plastic bucket or cooler and while wearing the proper PPE.
- 5.2.3. Test the pH of the sample.
 - a) If the pH is less than 7, proceed to Step 5.2.4.
 - b) If the pH is greater than 7, proceed to Step 5.2.6.
- 5.2.4. Neutralize acidic samples with sodium bicarbonate (baking soda). Slowly add sodium bicarbonate to the acidic sample.
- 5.2.5. After adding an amount of sodium bicarbonate, test the pH.
 - a) If the pH is less than 7 repeat Step 5.2.4.
 - b) If the pH is greater than 7 proceed to Step 5.2.6.
 - c) If the pH is equal to 7 flush the sample down the drain with water.
- 5.2.6. Neutralize basic samples with waste acid (This can usually be obtained from the Glassware Preparation Lab.) Slowly add acid to the basic sample.

5.2.7. After adding an amount of acid, test the pH.

a) If the pH is less than 7 go to Step 5.2.4.

b) If the pH is greater than 7 repeat Step 5.2.6.

c) If the pH is equal to 7 flush the sample down the drain with water.

6.0 References

- 6.1 Prudent Practices for Disposal of Chemicals from Laboratories, Committee on Hazardous Substances in the Laboratory, National Academy Press, Washington D.C., 1983

7.0 Attachments-

- 7.1 Page From Sample Archive Log

CAE STANDARD OPERATING PROCEDURE

SAMPLE RECEIVING

ISO NUMBER: PSPL-3

AUTHOR: Dept. 64

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1.0 Purpose- The purpose of this procedure is to provide steps for the accurate processing and distribution of samples received by CAE's Gravimetric and Ion Chromatography Laboratories.

2.0 Scope- This procedure applies to any employee receiving samples for analysis in the Gravimetric or Ion Chromatography laboratories.

3.0 Materials-

- Sample Receiving Logs (Internal & External)
- Samples
- Scissors (Knife)
- Three Hole Punch
- Black Pen
- Lab Cart
- Black Marker
- Analysis Tracking Sheets
- File Folders
- Dust Masks
- Chain of Custody Log

4.0 Definitions-

I.C. - Abbreviation for Ion Chromatography

Internal Samples - These are samples generated from CAE's own projects and sent to CAE's laboratories for analysis.

External Samples - These are samples generated by a company other than CAE, but were sent to CAE laboratories for analysis.

5.0 Procedure 1 - Internal Samples

Note: Samples will either be shipped or driven to CAE and should be left in the sample receiving area. If expected samples do not arrive, check with the project manager and/or the shipping department.

- 5.0.1. Open the sample box and unpack the samples. Vermiculite and cardboard should be recycled and reused. (If the samples are packed in vermiculite, it is a good idea to wear a dust mask.)
- 5.0.2. Place the chain of custody form(s) (Attachment 6.1) on the desk. (If there are no chain of custodies with the samples contact the project manager or the test leader.)
- 5.0.3. Obtain an Analysis Tracking Form (Attachment 6.2) from the lower right hand drawer of the desk in sample receiving. Fill out the information on the top of the form.
- 5.0.4. Examine the samples. Make sure each sample is:
 - a) Sealed with Teflon tape, if not , note it on the analysis tracking form.
 - b) Marked with a liquid level line, if not, or if the sample has leaked, note it on the analysis tracking form. If any sample leaked a noticeable amount of its volume, contact the project manager.
- 5.0.5. Compare the description and the number of samples to what is indicated on the chain of custody form. If the number of samples does not match what is indicated on the chain of custody, contact the project manager. If any sample description does not match the label on the sample, try to figure out which sample it really is by comparing it to the chain of custody form. If the sample still can not be correctly identified, contact the project manager. Note any irregularities on the analysis tracking form.
- 5.0.6. Each sample from a project is to be assigned its own unique number on the chain of custody form. Start with (-01) and number sequentially, using the following as a guide to order the samples based upon the type of analysis requested. Fill out the number on both the chain of custody form and write it , with black marker, on the sample itself.

Note: If one sample is in more than one container, assign both containers the same number.

- a) Sample types are: Thimbles, Filters, Acetone Rinses, Impinger Catches (Gravimetric), Impinger Catches (I.C.), Method 6/8 Titration Samples, Vacuum Bags and Others.
- b) Sample types should be grouped together by location. Blanks should be the first sample numbered in a set.

Example: A sample group of filters could be: the blank followed by Unit 1 Inlet Runs 1, 2, 3 followed by Unit 1 Outlet Runs 1, 2, 3 followed by Unit 2 Inlet Runs 1, 2, 3 followed by Unit 2 Outlet Runs 1, 2, 3 etc.

Note: Most projects will not have all types of samples, ignore the sample types when they are not present.

Example: If a project only has vacuum bags number them beginning with (-01).

5.0.7. Fill out the information requested in the next available space in the internal sample receiving log.(See Attachment 6.3).

Note: If samples from the same project have been received already, begin numbering the next set of samples starting after the last numbered sample.

Examples: Filters received for Gravimetric analysis on March 20, 1996 are numbered -01-08.

Acetone rinses received for Gravimetric analysis on March 20, 1996 are numbered -09-16.

Filters received for Gravimetric analysis on March 25, 1996 would be numbered -17-24.

Acetone rinses received for Gravimetric analysis on March 25, 1996 would be numbered -25-32.

This only applies to samples from the same project.

5.0.8. Once all samples have been assigned a number and everything is acceptable, sign off on the chain of custody form under the heading "Received for Laboratory By". Write in the date and time.

5.0.9. Separate the chain of custody forms and distribute them in the following way.

- a) White (Original) - Keep in project lab folder
- b) Canary - Three hole punch and place in the chain of custody log
- c) Pink - Give to project manager or file in explosion room
- d) Gold (If any) - Give to test leader or project manager or file in explosion room

5.0.10. On the tab of a new file folder, write the project name, project number and the date the samples were received. Put the white (original) copy of the chain of custody and the analysis tracking form in the folder and keep with the samples.

5.0.11. Give the samples to the proper laboratory for analysis.

5.1 Procedure 2 - External Samples

Note: External Samples will be shipped to CAE. If expected samples do not arrive, contact the shipping department or the external client.

5.1.1. Unpack the samples as in Step 5.0.1.

5.1.2. Locate a chain of custody. (Most clients will send a chain of custody with their samples, although it may look a lot different than CAE's form, the same information should be provided. If there are any questions whatsoever, the client should be contacted directly.)

- 5.1.3. Follow Step 5.0.3. Under project manager put the salesman.
- 5.1.4. Follow Step 5.0.4.
- 5.1.6. Compare the description and the number of samples to what is indicated on the chain of custody. Any questions or differences should be resolved by talking to the client or CAE salesman before proceeding.
- 5.1.7. Follow Step 5.0.6.
- 5.1.8. Fill out the information requested in the next available space in the external sample log (See Attachment 6.4). Assign the next sequential five digit lab number to the project (i.e. 26657). Write this number at the top of the external chain of custody and write "CAE Lab Number" next to it.
- 5.1.9. Once all samples have been assigned a number and everything is acceptable, sign and date the chain of custody form.
- 5.1.10. Make a copy of the chain of custody, three hole punch it and put the copy in the chain of custody log.
- 5.1.11. Follow Steps 5.0.10 and 5.0.11.

6.0 Attachments-

- 6.1 CAE Chain of Custody Form
- 6.2 Analysis Tracking Form
- 6.3 Page From the Internal Sample Log
- 6.4 Page From the External Sample Log

CAE STANDARD OPERATING PROCEDURE

CHEMICAL DISPOSAL

ISO NUMBER: LAB-2

AUTHOR: DEPT. 68

EFFECTIVE DATE: Pending

APPROVED BY: Pending

1. PURPOSE

To provide detailed information on the procedure for chemical disposal.

2. SCOPE

This procedure applies to those individuals involved in the disposal of used chemical solvents, containers, archived samples, mercury waste, and spent oxorbent.

3. MATERIALS

- Baking dish
- Hot plate
- Hood
- Tap water
- Trash can

4. PROCEDURE

4.1 Used Chemical Solvents

4.1.1 Pour into baking dish under the hood.

4.1.2 Evaporate.

4.2 Empty Chemical Containers

4.2.1 Remove lid.

4.2.2 Rinse with tap water.

4.2.3 Do not replace lid, and dispose of container in the trash.

4.3 Year Old Archival Samples

4.3.1 Weak Acids and Bases

4.3.1.1 Remove lid.

4.3.1.2 While running tap water, dump sample into laboratory sink.

4.3.1.3 Rinse container with tap water.

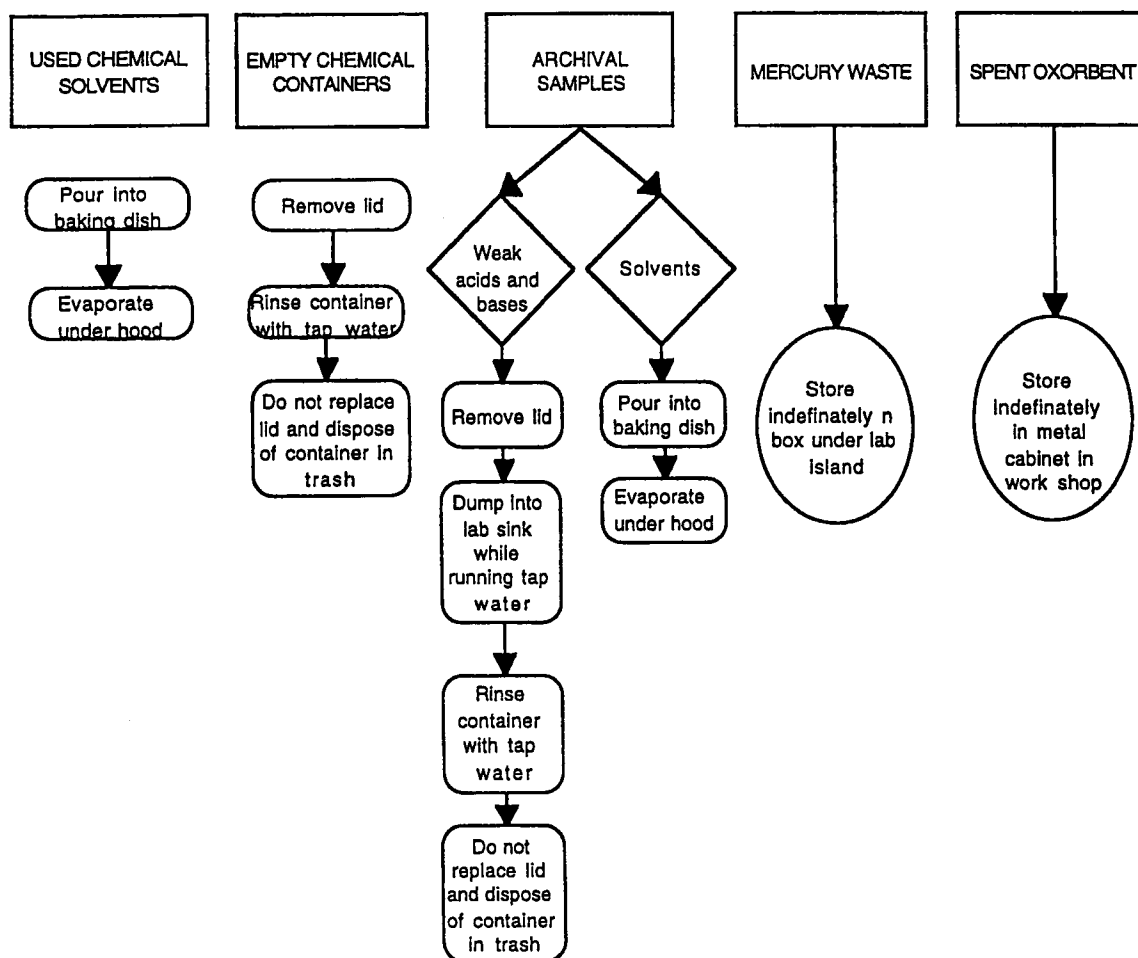
4.3.1.4 Do not replace lid, and dispose of container in the trash.

4.3.2 Solvents

- 4.3.2.1 Pour into baking dish under the hood.
- 4.3.2.2 Evaporate.
- 4.3.2.3 Rinse container with tap water.
- 4.3.2.4 Do not replace lid, and dispose of container in the trash.
- 4.3.3 Mercury Waste
 - 4.3.3.1 Store indefinitely in the box under the laboratory island.
- 4.3.4 Spent Oxorbent
 - 4.3.4.1 Store indefinitely in the metal cabinet in the workshop.

For more information consult MSDS or the Chemical Hygiene and Safety Plan.

5. PROCESS FLOW DIAGRAM



6. ATTACHMENTS

ISO NUMBER: LAB-3
EFFECTIVE DATE: Pending

AUTHOR: Dept. 68
APPROVED BY: Pending

This procedure provides the steps for the proper completion of a Chain of Custody Record Form.

This procedure applies to all Clean Air Engineering personnel who are responsible for generating Chain of Custody Record Forms to document the accountability of any lab media, reagents, or samples.

The following materials are needed to properly execute this procedure:

- Chain of Custody Record Form
- Black Ball Point Pen

Chain of Custody Record Form (COC) - A
form which serves as a permanent record of accountability and traceability for samples. The COC is divided into four major sections as shown in the accompanying figure. These sections are as follows:

- **Section I : Project Information** - this section covers project specific information which will be used for all lab data and analytical reports.
- **Section II : Sample Information** - this section covers the identification of collected samples or other media.
- **Section III : Analytical Information** - this section involves accounting for the number of sample containers, recording sample volumes (if applicable), designating the type of analysis for each sample, and relaying any additional information related to the proper analysis of the samples.
- **Section IV: Handling** - this section covers signatures of individuals relinquishing and receiving samples, the signature of the individual who completed the form, and a space for any special handling instructions or forwarding lab information.

Sample - Any solid, liquid or gaseous material which is collected during a source test for analysis.

Matrix - Refers to the media in which the samples are collected (i.e. acetone, 80% isopropanol, 0.1N sulfuric acid, deionized water, etc.)

[illegible]

Fraction - Refers to the individual portion of a sample recovery (i.e. thimbles, filters, front-half rinse, impinger condensate and rinse, etc.)

5.0 PROCEDURE

5.1 Make sure that all samples to be recorded are properly labeled.

5.2 Complete Section I : Project Information

5.2.1 Fill out the Project Information section as shown in the example below.

5.2.2 If any information is not available or not applicable, print "N/A" in the space.

CLIENT <u>ABC Manufacturing</u>	PROJECT NO. <u>5555</u>
PLANT <u>Wheeling , West Virginia</u>	DEPT. <u>C</u>
PROJECT MANAGER <u>Bob Gadget</u>	RECOVERY PERSON:
JOB LEADER <u>Jim Nelson</u>	<u>Joseph Labman</u>

Section I: Project Information

5.3 Complete Section II : Sample Information

5.3.1 Include the following information for each sample in this section:

- RUN NO. - a unique identification of the test run sequence. If the sample is a blank, then write in the word "Blank".
- TEST LOCATION - name and unit number (if applicable) of sample collection point.
- DATE - include the month and day that samples were collected (note that this may be different than the date which you are filling out the form).
- SAMPLE MATRIX - description of type of sample, including: sample fraction, matrix, and if necessary, any preassigned sample number (e.g., filter number).

5.3.2 Do not write in the CAE LAB NO. column. This is an ID number assigned by the analytical lab when samples are received. The laboratory sample custodian will normally complete this column.

5.3.3 When filling in sample information, group samples according to fraction and location. Always begin with reagent blanks or field blanks (when appropriate) followed by collected samples for each fraction (see the following examples).

CLEAN AIR ENGINEERING GENERATING A CHAIN OF CUSTODY RECORD FORM

SOP:LAB-3
Page 3

CAE LAB NO.	RUN NO.	TEST LOCATION	DATE	SAMPLE MATRIX
	Blank	Stack unit 1	7/30	Filter # 12345
	1	Stack unit 1	7/30	Filter # 12346
	2	Stack unit 1	7/30	Filter # 12347
	3	Stack unit 1	7/30	Filter # 12348
	Blank	Stack unit 1	7/30	Acetone Blank
	1	Stack unit 1	7/30	F1/2 Acetone Rinse
	2	Stack unit 1	7/30	F1/2 Acetone Rinse
	3	Stack unit 1	7/30	F1/2 Acetone Rinse
	Blank	Stack unit 1	7/30	0.1N H ₂ SO ₄ Blank
	1	Stack unit 1	7/30	B1/2 0.1N H ₂ SO ₄ C&R
	2	Stack unit 1	7/30	B1/2 0.1N H ₂ SO ₄ C&R
	3	Stack unit 1	7/30	B1/2 0.1N H ₂ SO ₄ C&R

Example of M5 Particulate with M26

CAE LAB NO.	RUN NO.	TEST LOCATION	DATE	SAMPLE MATRIX
	Blank	Stack unit 1	7/30	Filter Blank (3)
	1	Stack unit 1	7/30	Filter
	2	Stack unit 1	7/30	Filter
	3	Stack unit 1	7/30	Filter
	Blank	Stack unit 1	7/30	0.1N HNO ₃ Blank
	1	Stack unit 1	7/30	F1/2 0.1N HNO ₃ Rinse
	2	Stack unit 1	7/30	F1/2 0.1N HNO ₃ Rinse
	3	Stack unit 1	7/30	F1/2 0.1N HNO ₃ Rinse
	Blank	Stack unit 1	7/30	DI Water Blank
	Blank	Stack unit 1	7/30	5%HNO ₃ /10%H ₂ O ₂ Blank
	1	Stack unit 1	7/30	B1/2 5%HNO ₃ /10%H ₂ O ₂ C&R
	2	Stack unit 1	7/30	B1/2 5%HNO ₃ /10%H ₂ O ₂ C&R
	3	Stack unit 1	7/30	B1/2 5%HNO ₃ /10%H ₂ O ₂ C&R

Example of M29 Multi-

CAE LAB NO.	RUN NO.	TEST LOCATION	DATE	SAMPLE MATRIX
	Field Blank	Outlet unit 3	7/30	Filter
	1	Outlet unit 3	7/30	Filter
	2	Outlet unit 3	7/30	Filter
	3	Outlet unit 3	7/30	Filter
	Field Blank	Outlet unit 3	7/30	XAD Trap
	1	Outlet unit 3	7/30	XAD Trap
	2	Outlet unit 3	7/30	XAD Trap
	3	Outlet unit 3	7/30	XAD Trap
	Field Blank	Outlet unit 3	7/30	F1/2 Acetone/McCl ₃ Rinse
	1	Outlet unit 3	7/30	F1/2 Acetone/McCl ₃ Rinse
	2	Outlet unit 3	7/30	F1/2 Acetone/McCl ₃ Rinse
	3	Outlet unit 3	7/30	F1/2 Acetone/McCl ₃ Rinse

Example of M23 Dioxins

5.4 Complete Section III : Analytical Information

5.4.1 NO. OF CONTAINERS - Mark the number of containers required to hold the sample.

5.4.2 ORIGINAL VOLUME - For liquid samples, indicate the volume of the sample in milliliters (ml). This should be the volume before any aliquots are removed. In some circumstances, the weight of the substance may be measured in lieu of the volume. In these cases, indicate that the value is actually in grams (gm).

5.4.3 ANALYSIS REQUESTED - Indicate the type of analysis(es) required in the columns provided under this heading.

5.4.3.1 List one analytical method per column.

5.4.3.2 If there is not enough room to list specific analytes, add an asterisk and list the analytes in the ADDITIONAL INFORMATION column.

5.4.3.3 Mark "Archive" in a column if samples require storage only.

5.4.3.4 Place an "X" in the corresponding box for each of the samples to be analyzed by that specific method or to be archived.

- 5.4.4 ADDITIONAL INFORMATION - Use this column to indicate any information that may be useful for the proper analysis, storage, or handling of the samples listed. This may include requests for rush analysis or any analytical variations.
- 5.4.5 REVISION NO. - The blank for revision number is filled in only if any changes are made by an individual other than the originator of the document. When changes are made, that individual must initial and date the change. The first revision is REVISION NO. 1, and all subsequent revisions are numbered sequentially.


NO. OF CONTAINERS	ORIGINAL VOLUME	ANALYSIS REQUESTED				PAGE <u>1</u> OF <u>1</u> REVISION NO. _____ ADDITIONAL INFORMATION
		<i>Gravimetrics</i>	<i>M-26 HCl by IC</i>			
1		X				
1		X				Need preliminary
1		X				particulate numbers
1		X				by 8/1/96
						Give results to Proj
1		X				
1		X				
1		X				
1		X				
1	300ml		X			
2	856ml		X			
2	799ml		X			
2	823ml		X			

5.5 Complete Section IV: Handling

- 5.5.1 Once all of Sections I through III are completed, the individual who filled out the form must sign and date their name in the "This form was completed by:" space.
- 5.5.2 If samples are going to be forwarded to another lab after analysis at a CAE lab, fill in the forwarding information in the "Special Handling Instructions" space.
- 5.5.3 If samples are being shipped by courier (e.g., Federal Express), the individual responsible for packaging the samples must sign and date in the first "Relinquished by" space. This individual must also enter the name of the courier, the date, and the approximate time of receipt, in the corresponding boxes.


**CLEAN AIR ENGINEERING
GENERATING A CHAIN OF CUSTODY RECORD FORM**

**SOP:LAB-3
Page 5**

Relinquished by:(Signature) Joseph Labman	Date/Time 7/30/96 16:00	Received by:(Signature)	Date/Time	Relinquished by:(Signature)	Date/Time
Courier: Fed Ex	Date/Time 7/30/96 18:00	Relinquished by:(Signature)	Date/Time	Rec'd for Analysis by:	Date/Time
Special Handling Instructions Send filters and FI/2 0.1n nitric rinses to Square Labs metal analysis. Attn. Brenda Boast ph# (432) 555-6789 Forwarding Lab: Square Labs 666 Chemical lane Durham N.C. 30402 PO Number: 6789-345-61		This form was completed by: Joseph Labman 7/30/96 Signature Date		 500 West Wood Street Palatine, IL 60067 (847) 991-3300 phone (847) 991-3385 fax Clean Air Engineering <small>25 COC Palatine 8/20/96-4/97</small>	

Example for samples shipped by courier

- 5.5.4 If samples are being shipped by CAE vehicle, the individual responsible for packaging the samples must sign and date in the first "Relinquished by" space. The individual who takes responsibility of the samples (e.g., the driver) must sign and date/time in the first "Received by" box.

Relinquished by:(Signature) Joseph Labman	Date/Time 7/30/96 16:00	Received by:(Signature) Jack Roadwarrior	Date/Time 7/30/96 16:30	Relinquished by:(Signature)	Date/Time
Courier:	Date/Time	Relinquished by:(Signature)	Date/Time	Rec'd for Analysis by:	Date/Time
Special Handling Instructions Send filters and FI/2 0.1n nitric rinses to Square Labs metal analysis. Attn. Brenda Boast ph# (432) 555-6789 Forwarding Lab: Square Labs 666 Chemical lane Durham N.C. 30402 PO Number: 6789-345-61		This form was completed by: Joseph Labman 7/30/96 Signature Date		 500 West Wood Street Palatine, IL 60067 (847) 991-3300 phone (847) 991-3385 fax Clean Air Engineering <small>25 COC Palatine 8/20/96-4/97</small>	

Example of samples shipped by CAE

5.6 Distribute the COC

- 5.6.1 After all sections are completed, retain the gold and pink copy and give them to the test leader.
- 5.6.2 Put the white copy of the COC in a plastic bag and place inside the box with the samples.

CAE STANDARD OPERATING PROCEDURE

**CLEANING AND CALIBRATING THE SUPPRESSED CONDUCTIVITY
DETECTOR CELL OF THE DIONEX SERIES 2000I ION CHROMATOGRAPH**

ISO NUMBER: PICL-1

AUTHOR: Dept. 64

EFFECTIVE DATE: Pending

APPROVED BY: Pending

AUTHOR: Timothy A. Wojtach

REVISION: 1.1

APPROVED BY: Gary Zapel, Doug Rhoades

1.0 PURPOSE - The purpose of this procedure is to provide steps for the cleaning and calibration of the suppressed of the Dionex 2000I Ion Chromatograph.

2.0 SCOPE - This procedure applies to laboratory technicians operating the Ion Chromatograph.

3.0 MATERIALS

The following materials are needed to properly execute this procedure:

- Dionex Series 2000I Ion Chromatograph (Including all lines, the appropriate column, and the appropriate suppressor installed)
- Disposable 10 mL polystyrene Beakers
- Disposable Luer Lock 10 mL Syringes
- Disposable PTFE 0.2 µm syringe filters

4.0 DEFINITIONS -

IC - Abbreviation for the Dionex Series 2000I Ion Chromatograph

5.0 PROCEDURE

5.1 Set the local helium pressure to 4 psi using the valve on top of the IC.

5.2 Prepare the appropriate eluent and regenerant solutions.

5.2a For anions

eluent - Dissolve 0.5713 grams of sodium bicarbonate and 0.7613 grams of sodium carbonate into two liters of de-ionized water. Dilute to four liters with de-ionized water. Filter the eluent solution through a 0.2 μ m filter.

regenerant - Add three milliliters of concentrated sulfuric acid to two liters of de-ionized water. Dilute to four liters with de-ionized water.

5.2b For cations

eluent - Add 1600 milliliters of 0.1 N HCl to two liters of de-ionized water. Dilute to four liters with de-ionized water.

regenerant - Add 188.56 milliliters of tetrabutylammonium hydroxide to two liters of de-ionized water. Dilute to four liters with de-ionized water.

- 5.3 De-gas the eluent and regenerant solutions for 20 minutes.
- 5.4 Turn on the main power by pressing the blue button on the upper left side of the front panel of the IC.
- 5.5 Set eluent flow rate using the swithes on the lower right side of the front panal of the IC.
 - 5.5a Set the flow rate to 2.0 ml/min for anion applications.
 - 5.5b Set the eluent floe rate to 1.0 ml/min for cation applications.
- 5.6 Turn off the cell by pressing the square tan button until the red light indicates that the cell is off.
- 5.6 Turn on valve number 2 by pressing the the button next to its position on the center of the lower panel on the front of the IC.
- 5.7 Allow the eluent to run throught the valve for 2 minutes into a beaker by turning the lever above the Luer adapter on the lower left side of the front panel of the IC a quarter turn. After two minutes return the lever to its original position.
- 5.8 Turn on the pump by pressing the button on the lower right side of the front panel on the IC until the red light indicates the start position.
- 5.9 When the pump pressure is steady and the ready indicator light is on, turn on the cell.

- 5.10 Using a 10 mL syringe fitted with a syringe filter, inject de-ionized water into the sample port of the IC.
- 5.11 Repeat injections until a stable baseline is achieved.
- 5.12 IC is now ready for calibration.

Dilute to four liters with de-ionized water. Filter the eluent solution through a 0.2 μ m filter.

regenerant - Add three milliliters of concentrated sulfuric acid to two liters of de-ionized water. Dilute to four liters with de-ionized water.

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- 5.10 Using a 10 mL syringe fitted with a syringe filter, inject de-ionized water into the sample port of the IC.

5.11 Repeat injections until a stable baseline is achieved.

5.12 IC is now ready for calibration.

Clean Air Engineering Trademark Case Summary

Clean Air Engineering is currently involved in litigation over its "CAE" service mark. Clean Air Engineering applied for the use of 'CAE' as a service mark in November of 1991. During the opposition period, CAE, Inc. of Canada filed an opposition to our use of "CAE" in our "CAE Clean Air Engineering" logo. CAE, Inc. (the Canadian firm) lost its claim in the initial trademark judicial hearing. In 1997 CAE, Inc. filed suit in the US District Court for the Northeastern District of Illinois.

In January 2000, Judge Warren K. Urbom overturned the decision of the trademark board and awarded the mark to CAE, Inc. This decision would require Clean Air Engineering to alter or destroy of all marketing materials, products, signs, etc. displaying Clean Air Engineering's "CAE Clean Air Engineering" logo.

Clean Air Engineering filed a Motion with the trial Judge requesting reconsideration of his original decision. On August 4, 2000 Judge Urbom, though denying the Motion to Reconsider, agreed to stay a portion of his original decision which would allow Clean Air Engineering to continue using the "CAE Clean Air Engineering Logo" if our use of same is accompanied by the following statement: "CAE Clean Air Engineering, Inc. (or the appropriate division) is not affiliated with CAE, Inc." in conjunction with the distribution of all products, services and marketing materials which contain the current CAE Logo.

Clean Air Engineering has taken an appeal of Judge Urbom's rulings to the U.S. Court of Appeals for the Seventh Circuit. Clean Air Engineering is seeking to reverse the ruling of the Trial Court, and reinstatement of the original decision of the Trademark Board. Clean Air Engineering will ask the U.S. Court of Appeals for the Seventh Circuit to stay the use of any disclaimer as otherwise required by Judge Urbom's last order. It is possible that the Court of Appeals will not yet have acted on our request by the deadline date set by Judge Urbom, being October 11, 2000. If this is the case, all Clean Air Engineering products, services and marketing materials will be accompanied by the above disclaimer.

We ask for your patience and understanding during our ongoing litigation. Any questions or comments please contact Bill Walker or Frank Kilvinger.

Clean Air Engineering, Inc. has served the air quality management industry since 1972 - providing consulting, monitoring and testing services, instrumentation rental and equipment sales. Clean Air Engineering has a staff of over 80 environmental engineers, equipment specialists, manufacturing/electronics technicians, and regulatory analysts and support personnel. <http://www.cleanair.com>

**CAE Clean Air Engineering, Inc. is not
affiliated with CAE, Inc. of Toronto, Canada**