

Source Testing Manual

***Allegheny County Health Department
Air Quality Program
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Revised May 5, 2010

INTRODUCTION

This manual is provided by the Air Quality Program of the Allegheny County Health Department to assist the owners, managers, and operators of regulated sources, consultants, and members of this agency, to understand and comply with the procedures of source sampling.

Specific detail is included to guide source sampling through the required functions of the Air Quality Program. Organizational structures are presented with functional duties outlined to permit all concerned the necessary information as to all sampling responsibilities.

Sampling and analytical procedures likewise cannot always be exactly defined because they will vary with the purposes intended and the circumstances. There are specific procedures included, however, which are adaptable to most sampling requirements.

PURPOSES

Source sampling or emission testing, as applied to air pollution is the procedure whereby a representative sample is removed from some larger, contaminant-bearing gas stream confined in a duct or stack. This sample is then subjected to further analysis, and the contaminant concentrations are related to the parent gas stream to determine total quantities. Because the sample extracted from the main gas stream usually represents a very small fraction of the total volume, extreme care should be exercised in obtaining a representative sample. Additionally, because of the many and variable factors encountered in sampling gas streams, complex methods must frequently be used to obtain representative samples.

Source sampling frequently is employed to answer a variety of questions of which the main one is: What are the quantities and concentrations of emissions? Subsequent questions that can be answered from this basic determination include:

1. Is the process in compliance with the present or expected emission regulations?
2. What is the efficiency of existing pollution control equipment?
3. What effect do various process variables have on emissions?
4. Is a valuable product or by-product being emitted?
5. What are the potential (uncontrolled) emissions of various processes?
6. What further information is required, including legal enforcement actions, to attain compliance?
7. Is the operation of the process within approved surveillance limits?
8. Is continuous monitoring of emissions or parameter necessary?

P R E F A C E

The Air Quality Program regularly uses the test methods described in this manual. The Program may accept other test methods, as equivalent, suitable or required, in accordance with the provisions of the applicable regulations. Methods may be re-evaluated, revised, added, deleted or altered as more information becomes available and the applications are approved.

It is the intent of this manual to outline acceptable standard methods for determining compliance with the applicable regulations and for other purposes. All elements of a standard method may not always be exactly applicable or justified in a given situation; experience and intelligent judgment may be required to develop a usable sampling plan.

Since sampling results may have important legal consequences, and deviations from the standard methods set forth herein should be approved in advance by the Director in accordance with the applicable regulations. All such deviations should be thoroughly documented in the test protocol and reports.

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In determining compliance with the visible emission standards of the Rules and Regulations of the Allegheny County Health Department, the Following EPA Method 9 shall be used, except that the "averaging" provisions of paragraph 2.5 of Method 9 shall not apply.

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United States Environmental Protection Agency, 40 CFR 60 Appendix A (Method 29)

CHAPTER 40

"DETERMINATION OF INCINERATOR TEMPERATURES", Allegheny County Division of Air Quality

DETERMINATION OF INCINERATION TEMPERATURES

Insert a shielded thermocouple into the sample gas stream at a downstream point at least 2' from the center dimension of any burner. The location should be selected to be completely free of flame impingement and contact with walls or baffles. A normal location is near the effluent point of an afterburner section.

Contact with surfaces is to be avoided. A volume of sample gas is drawn through the shield from the incinerator effluent and temperatures are recorded after attaining thermal equilibrium or a constant temperature range cycle.

The suitable thermocouple is normally enclosed in a 1/8" diameter sheath and inserted in a 3/8" ID stainless steel tube. The tube is designed to protect the sheathed thermocouple from exposure to open sight of walls or flame, and reduce radiation effects. The tube (shield) is opened to the sample gas near the junction end of the thermocouple, but closed at the instrument end, except for a gas suction connection. A volume of gas is aspirated past the thermocouple, through the shield, at a 500 fps velocity (about 6.5 SCFM at 1400°F).

The temperature is read and recorded when stable or in a steady cycle.

CHAPTER 44

"DETERMINATION OF HYDROGEN SULFIDE FROM COKE OVEN GAS"

"Tutwiler Method for Hydrogen Sulfide", G.T. Altier, Gas Analysis and Testing of Gaseous Materials, N.Y. AGA (1945), page 339

and

"Standard Method of Test for Total Sulfur in Fuel Gases", American Society for Testing Materials, D 1072-56 (reapproved, 1970), 1916 Race Street, Philadelphia, PA.

CHAPTER 45

"DETERMINATION OF PARTICULATE MATTER FROM PRESSURIZED BAGHOUSES" Allegheny County Air Quality Program Positive Pressure Baghouse Test Procedure

- (1) The velocity and volumetric flow rate of gases going into the baghouse will be tested on the plenum connecting the fans to the baghouse. The number of traverse points will be selected according to EPA Method 1. EPA Method 2 will be used to determine the volumetric flow rate. The volumetric flow rate will be determined for each run of particulate testing.
- (2) The concentration of particulate matter in the baghouse exhaust gases will be determined by the use of high volume (hi-vol) samplers of the type described in 40 CFR, Part 50, Appendix B.
- (3)
 - (A) Each compartment will be sampled for 3 consecutive hours with 4 hi-vols per compartment.
 - (B) Each compartment will be divided in quadrants and one sampler located at the center of each quadrant. If desired, two or more compartments may be sampled simultaneously. All compartments must be sampled once during the compliance determination.
 - (C) The samplers will be located above the bag support mechanism. If temperature conditions preclude placement of the samplers at the locations specified above, flexible duct work will be used to duct the emissions to each sampler which will be located in a "safe" area. The inlet to the flexible duct will be positioned at the sampling locations specified above.
- (4) The starting flow rate through each high volume sampler shall be approximately 50 cfm. If the flow volume through any high volume sampler decreases by more than 20% from the starting flow rate, sampling with that hi-vol shall then be terminated. Record when the sampling period of each hi-vol sampler run commenced and terminated and record the physical location of each high volume sampler. These data shall be included in the test report. As soon as one such hi-vol sampler run is terminated, the filter shall be immediately replaced in that hi-vol and another run shall be started.

- (5) Any gratings of the baghouse compartments that may admit outside air into the baghouse will be sealed before the sampling period.
- (6) Temperature of the gases leaving the baghouse compartment will be measured by a temperature indicator located at each compartment being tested. This indicator will be located in the vicinity of one of the high volume samplers used to test the compartment.
- (7) Moisture content of the gases going to the baghouse will be measured at the plenum connecting the fans to the baghouse.
- (8) Test high volume sampler mass concentrations results will be determined and reported separately for each compartment.
- (9) In determining compliance with the emissions limitations these steps will be followed:
 - (A) A compartment average concentrations shall be computed by arithmetically averaging the concentrations from the four hi-vol sampler locations. If more than one run per hi-vol sampler is needed during any 3-hour test, then each of the individual concentrations per hi-vol sampler shall first be flow weight averaged to determine a concentration for that hi-vol sampler location.
 - (B) The separate compartment average concentrations shall be arithmetically averaged to determine an overall concentration for the entire test.
 - (C) In computing compliance, the overall concentration determined in (B) above shall be utilized, in conjunction with the flow rate measured.

PROCEDURE FOR QUALIFYING TEST HI-VOL

All test hi-vols used for testing pressurized baghouses should be qualified against a properly calibrated Bureau hi-vol sampler. The concentration variation of all test hi-vol samplers should indicate a concentration variation less than $\pm 15\%$ with the air quality program hi-vol.

Qualification of test samplers will take place in a clean plant area with the hi-vols placed at least 10 feet apart from each other and any other obstacles. Adequate power service should be provided (115 v. @ approximately 100 amp.) to prevent low flow rates at the individual samplers. Starting flow rates should be approximately 50 to 60 cfm. Final flow reduction for each individual sampler should be less than 20%. Any sampler showing a flow reduction greater than 20% should be recalibrated before the baghouse test is performed.

The samplers will run for four hours. Flow rate readings will be taken at the start and at one hour intervals during the test. Hi-vol filters will be supplied and weighed by the Allegheny County Laboratory.

CHAPTER 46

"DETERMINATION OF VOLATILE ORGANIC COMPOUND CONTENT OF EFFLUENT WATER"

Standard Methods for the Examination of Water from Wastewater, 14th edition, "Organic Carbon (Total), Combustion - Infrared Method", American Public Health Association, Washington, D.C.

CHAPTER 47

"DETERMINATION OF PARTICULATE MATTER FROM MODULAR BAGHOUSES", Allegheny County Air Quality Program

Modular Baghouse Test Procedure

- (1) Stack sampling procedures for determining compliance for sources equipped the modular baghouses shall be the method set forth in chapter 5 of this manual.
- (2) If the baghouse contains more than five stacks, the averaging provisions shall not apply and one test run per stack shall be performed. Compliance shall be determined by (a) computing the mass emission rate, in lb. per hour, for each stack and then (b) summing each of these mass emission rates. During all sampling periods, the production rate shall be maintained within ten percent of the mean rate for the entire sampling period.

CHAPTER 48

"MEASUREMENT OF ODOR EMISSIONS BEYOND SOURCE BOUNDARY LINES",
Allegheny County Health Department, Air Quality Program Methodology

Purpose:

This method shall be used to determine compliance with **Section 2104.04 of Article XXI**, which prohibits the emission of malodorous matter that is perceptible beyond the property line of the emitting source.

Observers:

During or immediately prior to making odor observations, the person making the observation should not smoke, use tobacco, eat, drink or do anything that would significantly affect his sense of smell. Observers should not wear aftershave, cologne or other products having a significant odor.

Observations:

The following procedure shall be used insofar as possible in making odor observations. Information concerning the results of the observation shall be recorded in a form similar to that set forth in Appendix 1 attached hereto.

- (1) Observer should first patrol the area near the source. If odors are detected, the observer should immediately make an odor observation for approximately ten minutes and record the information obtained on the observation form.
- (2) The observer should then attempt to determine the source responsible for the odors. This scan can be done by tracing the odor upwind or, if a particular source is suspected, by going directly to that source. When the source is identified, the observer should make another odor observation of approximately ten minutes near the source boundary on the side on which the initial odor observation was made. Information obtained should be recorded on the observation form.
- (3) If possible, the observer should then go to the opposite side of the source, make an odor observation approximately 180° from the second observation point, and record the information obtained.
- (4) As time and circumstances permit, the observer should patrol the area around the source to determine the extent of the area affected by the odors. Additional observations can be made and recorded as described above.
- (5) The observer may interview persons present in the area affected by the odors to obtain background information. Relevant information includes the frequency and duration of the odors, whether odor incidents occur at certain times or days or under certain circumstances, the period over which odor incidents have occurred, a description of the odors, their strength, characteristics and effects, and the like.

Observation Forms:

Information for each observation should be recorded at the time of making the observation.

A. **Strength of Odors:** The range of odor strengths observed at each observation point should be recorded using the definitions set forth below. If one odor strength prevails, that strength should be circled or otherwise marked to indicate that most of the odors during the observation were of that strength.

No Odors:

Slight Odors: The level at which the odor becomes detectable and can be described by its characteristics or can be distinguished from other odors.

Moderate Odors: The level at which the odor becomes strong enough to block out other odors, but is not so strong as to make someone try to avoid the odor.

Strong Odors: The level at which a person would take positive action to attempt to avoid the odor.

Very Strong Odors: The level at which the odor produces physiological effects, such as nausea, difficulty of breathing, irritation of the eyes, nose, or throat, or the level at which a residual smell remains after the odor itself is gone.

B. **Odor Description:** The observer should describe as fully and objectively as possible the odors perceived, including as appropriate comparisons to other commonly-known odors. All physiological effects should be noted.

C. **Weather Information:** Should be obtained from a qualified meteorologist or from the National Weather Service. Any significant differences between actual weather conditions at the observation sites and the information obtained from such meteorologist should be noted on the observation form.

D. **Map:** The observation form should include a hand-drawn map showing all observation points, their approximate distance from the suspected source, wind direction as perceived at each observation point, other area odor sources, relevant topographical features and other pertinent information.

APPENDIX 1

ALLEGHENY COUNTY HEALTH DEPARTMENT
AIR QUALITY PROGRAM
ODOR OBSERVATION FORM

DATE: _____

SOURCE: _____

DAY: _____

OBSERVATION START: _____ AM/PM OBSERVATION COMPLETE: AM/PM

ODOR STRENGTH

WEATHER CONDITIONS

WIND SPEED DIR:

TEMPERATURE:

- 0 = NONE
- 1 = SLIGHT
- 2 = MODERATE
- 3 = STRONG
- 4 = VERY STRONG

=====

OBSERVATION STATION	TIME INTERVAL	ODOR STRENGTH	MIN. OD@ EA. STRENGTH	ODOR TYPE

=====

TOTAL MINUTES OF OBSERVATION: _____

TOTAL MINUTES OF ODOR DETECTION: _____

OBSERVER: _____

SIGNATURE:

STATIONS LOCATIONS:

Remarks: _____

PLEASE SKETCH AREA AND STATION LOCATIONS ON BACK

Odor Verified by Citizen Witness: _____ Yes _____ No

Signature of Citizen Witness: _____

CHAPTER 49

"DETERMINATION OF SULFUR IN COKE"

"Standard Test Method for Total Sulfur in the Analysis of Coal and Coke", American Society for Testing Materials, D 3177, 1916 Race Street, Philadelphia, PA

CHAPTER 50

CALIBRATION AND MAINTENANCE Of Sampling Equipment Shall be Performed According to: Jerome J. Rom, Maintenance, Calibration and Operation of Isokinetic Source Sampling Equipment, Environmental Protection Agency, Research Triangle Park, N.C., APTD-0576, March, 1972

High Volume Samplers used to determine particulate matter emissions from pressurized baghouses pursuant to Chapter 45 shall be calibrated, maintained and operated according to "Reference Method for the Determination of Suspended Particulates in the Atmosphere (High Volume Method)", 40 CFR 60 Appendix B

CHAPTER 51

"MONITORING TEST METHODS FOR ABRASIVE BLASTING"

MONITORING TEST METHODS FOR ABRASIVE BLASTING

<u>Parameter</u>	<u>Method No.</u>	<u>Method Name</u>	<u>Comments</u>
Lead	CFR 40 Part 50 Appendix G (EPA Method)	Reference Method for the determination of lead in suspended particulate matter collected from ambient air	8 Hours sampling or longer
Free Silica (Respirable Fraction)	NIOSH Method 7500	Free Silica (Quartz, Cristobalite, Tridymite) in atmospheric dust	8 Hours sampling or longer
TSP	CFR40 Part 50 Appendix B (EPA Method)	Reference method for the determination of suspended particulate matter in the atmosphere (high-volume method)	8 Hours sampling or longer
PM-10*	(EPA method) CFR40 Part 50 Appendix J as <u>proposed</u> in FR Vol. 49, No. 55 Tuesday, March 20, 1984 Optional method for PM10 NIOSH method 7500	Reference method for the determination of particulate matter as PM10 in the atmosphere Personal pump sampling for PM10 using a 37 mm PVC filter and a MSA nylon cyclone (Part No. 456228) to fractionate the particles	8 Hours sampling or longer Quartz filters meet EPA specifications for SSI PM 10 Samplers 8 Hours sampling for longer. High volume respirable dust sample (sampling rate of 9L/min.)

*** PM-10 sampling is not required for abrasive blasting monitoring**

CHAPTER 52

"METHODS FOR WASTE DERIVED LIQUID FUEL SPECIFICATION AND FLUE GAS ANALYSIS"

I. Sampling for Waste Derived Liquid Fuel Specification Analysis:

1. Sampling

- a. For a tank with a capacity of 1,000 gallons or less, a representative sample of waste-derived liquid fuel shall consist of at least a single sample of sufficient volume and weight for all analyses required by Section 2105.31 of Article XXI. Single samples shall be taken from the tank at a level of fifty percent (50%) of the liquid height from the bottom of the tank.
- b. For a tank with a capacity of greater than 1,000 gallons, a representative sample of waste-derived liquid fuel shall consist of a composite sample taken in a manner acceptable to the Director. A single sample taken in accordance with Subparagraph a. of this Paragraph shall constitute a composite sample only where the owner or operator of the waste-derived liquid fuel-burning operation can demonstrate to the Director's satisfaction that the contents of the tank were sufficiently agitated prior to and during the taking of the sample.

II. Equipment Testing by Direct Emission Reduction for the Waste-Derived Liquid Fuel Regulation:

1. For all equipment except equipment subject to §2105.31.a.4, Article XXI, direct emission reduction shall be determined by the following equation:

$$\text{Percent Direct Emission Reduction} = \frac{\text{CO}_2}{\text{CO} + \text{CO}_2} \times 100, \text{ where:}$$

- a. CO_2 = the proportion by volume of carbon dioxide (CO_2) in the flue gas (on a dry basis); and
- b. CO = the proportion by volume of carbon monoxide (CO) in the flue gas (on a dry basis).

2. For equipment subject to §2105.31.a.4 of Article XXI, direct emission reduction shall be determined by one (1) of the following equations:

a.
$$\text{Percent Direct Emission Reduction} = \frac{\text{CCl}_4(\text{in}) - \text{CCl}_4(\text{out})}{\text{CCl}_4(\text{in})} \times 100, \text{ where:}$$

- i. $\text{CCl}_4(\text{in})$ = the mass feed rate of carbon tetrachloride (CCl_4) going into the equipment in the fuel;
- ii. $\text{CCl}_4(\text{out})$ = the mass emission rate of carbon tetrachloride (CCl_4) coming from the equipment in the flue gas; and
- iii. The fuel being burned for the purpose of determining direct emission reduction under this Subparagraph, in addition to complying with Paragraph #3 of Section 2105.31. This test procedure is spiked so as to contain at least 1,000 ppm of carbon tetrachloride (CCl_4) by weight.

$$\text{b. Percent Direct Emission Reduction} = \frac{\text{POHC (in)} - \text{POHC (out)}}{\text{POHC (in)}} \times 100, \text{ where:}$$

- I. POHC (in) = the mass feed rate of principal organic halogenated compounds (POHC) going into the equipment in the fuel;
 - ii. POHC (out) = the mass emission rate of principal organic halogenated compounds (POHC) coming from the equipment in the flue gas; and
 - iii. The fuel being burned for the purpose of determining direct emission reduction under this Subparagraph in addition to complying with Paragraph #3 of Section 2105.31. This test procedure is spiked so as to contain at least 1,000 ppm of principal organic halogenated compounds (POHC) by weight.
3. For the purpose of determining direct emission reduction under Section 2105.31.:
- a. The owner or operator of the affected equipment shall conduct the tests required under testing requirements contained in Subsection §2108.02.e. of Article XXI unless otherwise specified by this Section or the Director; and
 - b. The fuel being burned for a test required under Section 2105.31 shall:
 - I. Contain the maximum routine percentage of waste-derived liquid fuel that is present when the equipment is operating at routine operating conditions; and
 - ii. Contain waste-derived liquid fuel, which is representative of the waste-derived liquid fuel burned when the equipment is operating at routine operating conditions.

<u>PARAMETER</u>	<u>METHOD NO.</u>	<u>METHOD NAME</u>	<u>COMMENTS</u>
Metals (Ar,Cd,Cr,Ph)	1) ASTM D2788-72	Trace metals in gas turbine fuels (Atomic absorption method)	Use with NJDEP digestion procedures in Appendix C
	2) NBS 1130 (1)	Test procedures for three recycled fuel oil impurities: Lead	Modified D2788 use with nitric/perchloric acid digestion as proposed in Engineering Science Study
	3) EPA Method 200.7	Inductively coupled plasma-atomic emission spectrometric method for trace element analysis of water and wastes	Appendix C to Part 136 Waste Water Method
	4) ASTM E 1097-86	Standard guide for direct current plasma emission spectrometry analysis	Used by PTM inspectorate
	5) APHA 305-85	Metals by emission spectroscopy using an inductively coupled plasma source.	Waste Water Method
	6) APPHA 304-85	Determination of micro quantities of aluminum, antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, iron, lead, manganese, molybdenum, selenium, silver, and tin by electrothermal atomic absorption spectrometry	Waste Water Method
	7) APA 303B	Determination of low concentration of cadmium, chromium, cobalt, copper, iron, lead, manganese, nickel, silver, and zinc by chelation with Ammonium Pyrrolidine Dithiocarbamate (APDC) and extraction into Methyl Isobutyl Ketone (MIBK)	Waste Water Method
<u>1)</u> (C C

<u>PARAMETER</u>	<u>METHOD NO.</u>	<u>METHOD NAME</u>	<u>COMMENTS</u>
Metals (Ar,Cd,Cr,Ph)	1) ASTM D2788-72	Trace metals in gas turbine fuels (Atomic Absorption method)	IsUse with NJDEP digestion procedures in Appendix C
	2) NBS 1130 (1)	Test procedures for three recycled fuel oil impurities: Lead	MModified D2788 use with nitric/perchloric acid digestion as proposed in Engineering Science Study.
	3) EPA Method 200.7	Inductively coupled plasma-atomic emission spectrometric method for trace element analysis of water and wastes	AAAppendix C to Part 136 Waste Water Method
	4) ASTM E 1097-86	Standard Guide for direct current plasma emission spectrometry analysis	UUUsed by PTM inspectorate
	5) APHA 305-85	Metals by emission spectroscopy using an inductively coupled plasma source	Waste Water Method
	6) APHA 304-85	Determination of micro quantities of aluminum, antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, iron, lead, manganese, molybdenum, selenium, silver, and tin by electrothermal atomic absorption spectrometry	Waste Water Method
	7) APA 303B	Determination of low concentration of cadmium, chromium, cobalt, copper, iron, lead, manganese, nickel, silver, and zinc by chelation with Ammonium Pyrrolidine Dithiocarbamate (APDC) and extraction into Methyl Isobutyl Ketone (MIBK)	Waste Water Method

<u>PARAMETER</u>	<u>METHOD NO.</u>	<u>METHOD NAME</u>	<u>COMMENTS</u>
PCB's	1)-----	Improved method for polychlorinated biphenyl determination in complex matrices (1)	EPA 608 used as framework (waste water method) Digestion of sample according to Copeland and Gohmann procedure.
	2) NBS 584 (2)	Determination of polychlorinated biphenyls in waste and lubricating oils	Utilized L C clean-up procedure followed by glass capillary GC method with electron capture detector.
	3) ASTM D4059-86	Analysis of polychlorinated biphenyls in insulating liquids by gas chromatography	Requires sample pre-clean up by NBS584 or Copeland and Gohmann procedure (used by PTM inspectorate)
BOTTOM SEDIMENT AND WATER	1) ASTM D 96-73	Water and sediment in crude oils	
	2) ASTM D 1796-83	Water and sediment in fuel oils by the Centrifuge method	Centrifuge method recommended by NBS 1130 for analysis of difficult types of oils.
WATER ONLY	3) ASTM D 95-83	Water in petroleum products and Bituminous material by distillation	Distillation method.
SEDIMENT ONLY	4) ASTM D 473-81	Sediment in crude oils and fuel oils by the extractive method	Extraction method
ASH	ASTM D 874-82	Sulfated ash from lubricating oils and additives	Sulfated ash
	ASTM D 482-80	Ash from petroleum products	Method is limited to petroleum products which are free from added ash forming additives, including certain phosphorus compounds.

(1) Copeland G.B. and Gohman, C.S., "Improved Method for Polychlorinated Biphenyl Determination in Complex Matrices", Environmental Science and Technology

(2) NBS Special Publication 58

METHODS FOR WASTE DERIVED LIQUID FUEL SPECIFICATION ANALYSIS, ANALYSIS BY WEIGHT

<u>PARAMETER</u>	<u>METHOD NO.</u>	<u>METHOD NAME</u>	<u>COMMENTS</u>
FLASH PIONT	ASTM D 93-85	Flash point by Pensky-Martens closed tester	
TOTAL HALOGENS	1) ASTM D 808-81	Chlorine in new and used petroleum (bomb method)	
	2) ASTM D 1317-83	Chlorine in new and used lubricants (Sodium Alcoholate method.	
	3) -----	X-Ray fluorescence or ion chromatography by ASTM D 808-63	
HEAT OF COMBUSTION	1) ASTM D 240-85	Heat of combustion of liquid hydrocarbon fuels by bomb Calorimeter	
	2) NBS 1130	Heating value	

**METHODS FOR FLUE GAS ANALYSIS APPLICABLE
TO WASTE-DERIVED LIQUID FUEL REGULATION**

A. Methods for Carbon Monoxide Analysis:

- 1) EPA method 10 – analysis using a luft-type NDIR analyzer or equivalent. This method allows either a continuous or integrated approach; the modification of the integrated approach using EPA method 25 sample collection tanks instead of tedlar bags allows safe storage of the undegraded CO sample to the laboratory for analysis.
- 2) ASTM D 1946-77 (uses thermal conductivity detector)

B. Methods for Carbon Dioxide Analysis:

- 1) EPA Method 3 (orsat or fyrite methods)
- 2) EPA Method 3A (instrument method) – concurrent measurements should be obtained using orsat or fyrite analyzers, instrument specifications are contained in Method 6C
- 3) EPA Method 6A (method 6 midget impinger train followed by ascarite II CO₂ absorber); acceptable for CO₂ values between 2.5% and 25%.

C. Methods for Principal Organic Halogenated Compound Analysis:

- 1) EPA Method 23 (determination of halogenated organics) – For analysis, gas chromatographic (GC) analysis using either a flame ionization detector (FID) or electron capture detector (EDC) is acceptable to the Administrator.

CHAPTER 53

“DETERMINATION OF INHALABLE FUGITIVE PARTICULATE EMISSIONS
FROM AIR POLLUTION SOURCES WITHIN A STRUCTURE,” Allegheny County
Air Quality Program Methodology

CHAPTER 53

DETERMINATION OF INHALABLE FUGITIVE PARTICULATE EMISSIONS FROM AIR POLLUTION SOURCES WITHIN A STRUCTURE

Principle and Applicability and Limitations:

- a. The method measures the mass concentration of respirable dust smaller than 10 micron particulate size.
- b. This method is applicable to respirable dust from the fugitive emission sources enclosed within a structure.
- c. Depending on predominant wind direction during the sampling, air flow will be inward at some openings and outward at others. Only the openings with outward air flow will be responsible for fugitive emission and therefore, the area and air velocity through these openings will be taken into consideration for calculating emission rate.
- d. Since the wind direction may change during sampling, the air flow which was outward may become inward at the same openings where the test is being carried out. Under this situation, the sampling must continue at the same opening.

If the air flow remains inward for more than 50 percent of sampling time, then the sample must be discarded and another opening should be selected for sampling.

A. Sample Points:

By visual determination, select the openings with the most fugitive dust leakage out of enclosed structure.

Each sampling area should be divided into 23 equal areas with the sample point at the centroid of each area.

B. Velocity and Temperature:

The velocity of the air flowing through the sample point can be measured with a vane anemometer or Kurtz Model 441 or equivalent at the centroid of the sample point. The velocity should be measured at the beginning and end of the sampling period for each sample point. The total of 24 velocity measurements shall be made per two hour run.

The temperature of the air can be measured with a mercury thermometer before and after each test run.

C. **Moisture:**

The moisture in the sampled air will be the relative humidity of the ambient air. This can be determined with a sling psychrometer and should be measured at least once at each opening.

D. **Area of Openings:**

The area of every opening shall be measured in square feet.

1.0 Apparatus:

1.1 Sampling train: The sample train consists of a 10 millimeter Dorr-Oliver cyclone connected to a filter holder (37mm) loaded with a tared quartz filter and personal sampling pump.

I.I.1. Personal sampling pump: A personal sampling pump capable of sampling air at 1.7L/min $\pm 5\%$ with flexible Tygon connecting tubing.

I.I.2. Filter: Whatman type ZM-A, 37 mm diameter quartz filter or equivalent supported with backup pad in a two-piece, 37mm cassette filter holder held together by tape or cellulose shrink band.

I.I.3. Cyclone: 10 mm Dorr-Oliver nylon cyclone.

I.I.4. Sampling Head Holder: This holder must keep the cassette, cyclone and coupler together rigidly so that air enters only at the cyclone inlet.

2.0 Equipment for Analysis:

2.1 Environmental Chamber for Balance: It shall be maintained at 20 degrees C ± 0.3 degrees C and 50% $\pm 5\%$ humidity.

2.2 Vacuum desiccator:

3.0 **Filter Cassette Preparation and Pre-Weighing Before Sampling:**

- 3.1 Dry filters and backup pads under vacuum in the vacuum desiccator for at least 15 minutes. Release the vacuum, remove the desiccator cover, and equilibrate the filters in the environmental chamber for at least 1 hour. Or,
- 3.2 Desiccate the filters and backup pads in the desiccator for 24 hours.
- 3.3 Number the backup pads with a ballpoint pen and place one pad, the numbered side down, in the filter cassette bottom section.
- 3.4 Weigh the filters in the environmental chamber. Record the filter tare weight, W_1 (mg).

3.4.1 Zero the balance before each weighing.

3.4.2 Handle the filter with forceps (nylon forceps, if further analysis will be done).

- 3.5 Place a weighed filter on top of the backup pad in the filter cassette bottom section and allow to stand an additional 8 to 16 hours in the environmental chamber.
- 3.6 Reweigh the filters. If this tare weight differs by more than 0.01 mg from the first tare weight obtained in step 3.4 above, discard the filter.

NOTE: Insert a rod through the outlet hole of the filter cassette bottom section to raise the backup pad and filter so that the filter can be grasped with forceps.

- 3.7 Reassemble the filters in the filter cassettes and close firmly so that leakage around the filter will not occur. Place a plug in each opening of the filter cassette. Place a cellulose shrink band around the filter cassette, allow to dry, and mark with the same number as the backup pad.
- 3.8 Remove the cyclone's grit cap and vortex finder before use and inspect the cyclone interior. If the inside is visibly scored, discard this cyclone since the dust separation characteristics of the cyclone might be altered. Clean the interior of the cyclone to prevent re-entrainment of large particles.

- 3.9 Assemble the sampler head. Check alignment of filter holder and cyclone in the sampling head to prevent leakage.

4.0 Procedure

4.1 Calibration

- 4.1.1. Calibrate each personal sampling pump to 1.7 L/min. With a representative quartz filter in line.

4.2 Sampling Procedure

- 4.2.1. For door or window openings, sample at the centroid of each 12 equal areas used during the velocity traverse for 10 minutes per point for a total of 120 minutes per run. The sample flow rate shall be 1.7 L/min. Three runs shall comprise a test, one each at three different window or door openings. The cyclone air intake shall face the window or door opening and shall be no more than 6 inches outside the plane of the door or the window.

NOTE: *Do not allow the sample assembly to be inverted at any time. Turning the cyclone to anything more than a Horizontal orientation may deposit oversized material from the cyclone body onto the filter.*

5.0 Sample Preparation

- 5.1 Wipe dust from the external surface of the filter cassette with a moist paper towel to minimize contamination. Discard the paper towel.
- 5.2 Remove the top and bottom plugs from the filter cassette. Place the filter cassettes in a vacuum desiccator for at least 15 minutes followed by equilibration for at least 1 hour in the environmental chamber, or desiccate the filter in desiccator for 24 hours.

- 5.3** Remove the filter cassette band, pry open the filter cassette, and remove the filter by inserting a rod in the outlet hole of the filter cassette. Handle the filters very carefully by the edge to avoid loss of dust.
- 5.4** Zero the microbalance before all weighings. Use the same microbalance for weighing filters before and after sample collection. Calibrate the balance with National Bureau of Standards Class M. Weights.
- 5.5** Take two replicate blank filters for every batch of field samples for quality assurance on the sampling procedures. The set of replicate blank filters should be exposed to the same dust environment, either in a laboratory dust chamber or in the field. The quality control samples must be taken with the same equipment, procedures and personnel used in the routine field procedures. Circulate precision from these replicates. Take corrective action when the precision is out of control.
- 5.6** Weigh each filter, including field blanks. Record this post-sampling weight, W_2 (mg), beside its corresponding weight. Record anything remarkable about a filter (e.g., visible particulates, overloaded, leaking, wet, torn, etc.)

6.0 Calculations

- 6.1** Calculate the concentration of respirable dust, C (mg/m^3), in the air volume sampled, V (liters):
- $$C = \frac{(W_2 - W_1) + B}{V} \cdot 10^3, \text{ mg}/\text{m}^3 \quad (1)$$

Where: W_1 = tare weight of filter before sampling (mg).

W_2 = post-sampling weight of sample-containing Filter (mg).

B = mean change in field blank filter weights between tare and post-sampling (mg) (+or-)

V = Air volume sampled (liters)

- 6.2** Calculation of Air Volume rate through a door or window.

$$V_0 = V_s \times A_s \quad (2)$$

Where: V_0 = Volumetric flow rate from a single window or door opening of ft^3/min .

V_s = Average of 24 velocity measurements by anemometer (ft/min) for a single window or door opening.

A_s = Cross-sectional area of a single window or ft^2 door opening.

6.3 Calculation of Total Air Volume rate through the building.

$$V_{O1} = V_w = A_1 \quad (3)$$

Where: V_{O1} = Total volumetric outward flow rate from all building openings (ft^3/min).

V_w = Opening velocity averaged for 3 runs (ft/min).

A_1 = Cross sectional area (ft^2) of all building openings where air flow was outwards.

6.4 Emission Rate Calculation (1 lb/hour)

$$C = 1.3216 \times 10^4 C_{\text{avg}} \times \frac{V_{O1}}{3.53} \quad (4)$$

Where: C = Total concentration lb/hour from all openings.

C_{avg} = Average concentration mg/m^3 of 3 runs at 3 different openings.

$$C_{\text{avg}} = (C_1 + C_2 + C_3) / 3$$

C_1 = Concentration from run 1 mg/m^3

C_2 = Concentration from run 2 mg/m^3

V_{O1} = Total volumetric flow rate from all building openings. $\frac{\text{Ft}^3}{\text{min}}$.

35.3 = Conversion factor for converting cubic feet to cubic meters.

CHAPTER 54

“DETERMINATION OF PARTICULATE EMISSIONS FOR COKE OVEN PUSHING SOURCES,” Allegheny County Health Department, Air Quality Program.

Stack testing procedure for determining compliance of particulate emissions for coke oven pushing sources shall be the methodology specified in Chapter 5 of this manual with the following back-half analysis to be used:

1. Identify and desiccate for 24-hours sufficient filters to filter all liquid collectors and washing.
2. Weigh and record the weights of the filters in addition to the identification number.
3. Before filtering, measure to the nearest millimeter, the volume of liquid in each impinger and record the measurement.
4. Place the impinger solutions in a labeled container and wash any residuals in the impingers into the container using distilled water and a policeman.
5. Add the water washings from the back half of the filter holder and interconnecting glassware to the container in (4).
6. Wash the impingers and the connecting glassware with acetone and place in a labeled container.
7. Filter solutions from steps (4) and (5) using pre-weighed and identified Millipore .22 micron Catalog # GSPW-047-00
.45 micron Catalog # HAWP-047-00
.80 micron Catalog # AAWP-047-00
8. Measure the filtrate from (7) to the nearest millimeter and record the volumes.
9. Air dry, then desiccate for 24-hours all filters used in step 7. Weigh to the nearest .1 mg., and record the weight. As an alternative, the filters can be oven dried at 160-180° F, cooled in a desiccator, and weighed to a constant weight.
10. Identify and dry for two hours at 105° C as many 350 milliliter evaporating dishes as needed to evaporate the filtrates. Then desiccate until cool and weigh to the nearest .1 mg. Record each weight with its identification number.
11. Impinger solution after filtration – follow steps a through d below:

- a. Evaporate impinger (sample) solution to a minimum weight using evaporating dishes prepared in step 10 and an equal amount of distilled water used in the impingers at 160-180° F. Record the weight of the distilled water residue. This will be the distilled water blank.
 - b. Re-dissolve the sample solution residue in water and neutralize using NaOH used. Calculate weight of Na⁺ ion. (The NaOH should react with any free H₂SO₄ to tie up the SO₄⁼ present in sample as well as other condensibles).
 - c. Evaporate neutralized solution to constant weight at 160-180° F. Record the weight. (This weight should include Na⁺, added plus the SO₄⁼ present in sample, as well as other condensibles).
 - d. Re-dissolve in distilled water and use turbidimetric method for determination of sulfate ion (SO₄⁼). Calculate weight of SO₄⁼. An acceptable alternative for determining sulfate ion would be the Barium-Thorninn titration method (EPA Method 6).
12. Transfer the acetone washing from step 6 into a pre-weighed evaporating dish after measuring and recording the volume of acetone.
 13. Place a measured quantity of unused acetone into pre-weighed evaporating dish. This is the acetone blank.
 14. Evaporate both blank (13) and unknowns (12) at less than 60° C. in a vacuum.
 15. Dessicate to a constant weight and reweigh. Then record the weight to the nearest 0.1 milligram.
 16. Calculation of back half particulate catch – determine the total back half particulate catch from the sum of the weights obtained from steps (9), (11c), and the unknown in step (15) minus the weights obtained from steps (11a), (11b), and (11d), and the blank from step (15).

Compliance with the particulate mass emission standard for coke oven pushing shall be calculated based upon the front half of the EPA train and insoluble weights in the impinger solution and on sample exposed surfaces subsequent to the final filtration media. Insoluble weights shall be determined by 0.22 micron filtration

CHAPTER 55

“DETERMINATION OF VOLATILE ORGANIC COMPOUND EMISSIONS FROM VAPOR RECOVERY SYSTEMS FOR GASOLINE LOADING OPERATIONS”

Emission Test Procedures for Tank Truck Gasoline Loading Terminals (Appendix A) “Control of Hydrocarbons from Tank Truck Gasoline Loading Terminals,” EPA-450/2-77-026 United States Environmental Protection Agency, Oct. 1977.

CHAPTER 56

“DETERMINATION OF THE LEAK TIGHTNESS OF GASOLINE TANK TRUCKS AND VAPOR RECOVERY SYSTEMS”

Pressure-Vacuum Test Procedures for Leak Tightness of Truck Tanks (Appendix A), “Control of Volatile Organic Compound Leaks From Gasoline Tank Trucks and Vapor Collection Systems,” EPA-450/2-78-051, United States Environmental Protection Agency, December, 1978.

CHAPTER 57

“DETERMINATION OF THE MAGNITUDE OF LEAKS OF VOLATILE ORGANIC COMPOUNDS FROM GASOLINE TANK TRUCKS AND VAPOR RECOVERY SYSTEMS”

Gasoline Vapor Leak Detection Procedure by Combustible Gas Detector (Appendix B), “Control of Volatile Organic Compound leaks from Gasoline Tank Trucks and Vapor Collection Systems,” EPA-450/2-78-051, United States Environmental Protection Agency, December, 1978.

CHAPTER 58

“DETERMINATION OF COMPLIANCE OF PERCHLOROETHYLENE DRY CLEANING FACILITIES”

Appendix V, “Compliance Test Method and Leak Detection Equipment for Perchloroethylene Dry Cleaners,” Measurement of Volatile Organic Compounds, United States Environmental Protection Agency, EPA-450/2-78-041, Washington, D.C.

CHAPTER 59

“DETERMINATION OF COMPLIANCE OF PETROLEUM SOLVENT DRY CLEANING FACILITIES EMPLOYING A PETROLEUM SOLVENT FILTRATION SYSTEM BUT NOT EMPLOYING CARTRIDGE FILTERS”

ASTM Method D322-901 (Standard Test Method for Gasoline Diluents in Used Gasoline Engine Oils by Distillation).

CHAPTER 60

“DETERMINATION OF ASBESTOS CONTENT OF BULK SAMPLES”

“Guidance for Controlling Asbestos-Containing Materials In Buildings,” EPA 560/5-85-024
United States Environmental Protection Agency, June 1985.

CHAPTER 101

“DETERMINATION OF PARTICULATE AND GASEOUS MERCURY EMISSIONS FROM CHLOR-ALKALI PLANTS (AIR STREAMS),” United States Environmental Protection Agency, 40 CFR 61 Appendix B (Method 101 and 101A)

CHAPTER 102

“DETERMINATION OF PARTICULATE AND GASEOUS MERCURY EMISSIONS FROM CHLOR-ALKALI PLANTS (HYDROGEN STREAMS),” United States Environmental Protection Agency, 40 CFR 61 Appendix B (METHOD 102)

CHAPTER 103

“BERYLLIUM SCREENING METHOD,” United States Environmental Protection Agency, 40
CFR 61 Appendix B (METHOD 103)

CHAPTER 104

“DETERMINATION OF BERYLLIUM EMISSIONS FROM STATIONARY SOURCES,”
United States Environmental Protection Agency, 40 CFR 61 Appendix B (METHOD 104)

CHAPTER 105

“METHOD FOR DETERMINATION OF MERCURY IN WASTE WATER TREATMENT PLANT SEWAGE SLUDGE,” United States Environmental Protection Agency, 40 CFR 61 Appendix B (METHOD 105)

CHAPTER 106

“DETERMINATION OF VINYL CHLORIDE FROM STATIONARY SOURCES,” United States Environmental Protection Agency, 40 CFR 61 (Appendix B (Method 106))

CHAPTER 107

“DETERMINATION OF VINYL CHLORIDE CONTENT OF INPROCESS WASTES, WATER SAMPLES, AND VINYL CHLORIDE CONTENT OF POLYVINYL CHLORIDES IN, SLURRY, WETCAKE AND LATEX SAMPLES,” United States Environmental Protection Agency, 40 CFR 61 Appendix B (METHOD 107 and 107A)

CHAPTER 108

“DETERMINATION OF PARTICULATE AND GASEOUS ARSENIC EMISSIONS,” United States Environmental Protection Agency, 40 CFR 61 Appendix B (METHOD 108 and 108A)

CHAPTER 109

“DETERMINATION OF VISIBLE EMISSIONS FROM COKE OVEN BATTERIES,” United States Environmental Protection Agency, 40 CFR 61 (Appendix B (Method 109), as modified by the Allegheny County Health Department, Air Quality Program.

CHAPTER 109

COKE-OVEN INSPECTION PROCEDURES

- A. **Charging:** For purposes of determining compliance with the coke oven charging standard, the following inspection technique will be utilized.

Observations of charging emissions shall be made from any point or points on the topside of a coke oven battery from which an observer can obtain an unobstructed view of the charging operation. The observer will determine and record the total number of seconds that charging emissions are visible during the charging of coal to be the coke oven. The observer shall time the visible emissions with a stopwatch while observing the charging operation. Simultaneous emissions from more than one emission point shall be timed and recorded as one emission and shall not be added individually to the total time. Open charging emissions shall not include any emissions observed after all the charging port covers have been firmly seated following the removal of the Larry car, such as emissions occurring when a cover is temporarily removed to permit the sweep-in of spilled coal. The total number of seconds of visible emissions observed, clock time for the initiation and completion of the charging operation, battery identification and oven number for each charge shall be recorded by the observer. In the event that observations of emissions from a charge are interrupted due to events beyond the control of the observer, the data from that charge shall be invalidated and the observer shall note on his observation sheet the reason for invalidating the data. The observer shall then resume observation of the next consecutive charge or charges, and continue until he has obtained a set of four charges for comparison with the emission standard. Compliance shall be determined by summing the seconds of charging emission observed during each of the four charges.

- B. **Doors:** Compliance with the 40% opacity limitation for the door area emissions shall be determined in accordance with the following method.

Observations of door area emissions shall be made at a point above the top, or at the top of the door, but below the battery top, or at the top of any local door area emission control hood. The observer shall place himself no less than 25 feet from the face of the door in a location where his view of the door area is unobstructed.

- C. **Doors:** Compliance with the door area leakage standard shall be determined in accordance with the following method:

Observations of door area emissions shall be made from a minimum distance of 25 feet from each door. Each door area shall be observed in sequence for only that period necessary to determine whether or not, at the time, there are visible emissions from any point on the door area while the

observer walks along side of the battery. If the observer's view of a door area is more than momentarily obstructed, as, for example, by door machinery, pushing machinery, coke guide, Luther truck, or opaque steam plumes, he shall record the door area obstructed and the nature of the obstruction and continue the observations with the next door area in sequence which is not obstructed. The observer shall continue this procedure along the entire length of the battery for both sides and shall record the battery identification, battery side, and oven door identification number of each door area exhibiting visible emissions. Before completing the traverse or immediately thereafter he shall attempt to re-observe the obstructed doors. Compliance with this section shall be calculated by application of the following formula, which excludes two door areas representing the last oven charged from the numerator and obstructed door areas from the denominator:

$$\frac{(\# \text{ of door areas with visible emission}) - 2}{(\# \text{ of door areas on operating ovens in battery}) - (\# \text{ of door areas obstructed from view})} \times 100 = 10\% \text{ or less.}$$

D. Charging Ports: For purposes of determining compliance with the percent charging port leakage standard, observations of any visible emissions from coke oven topside, other than charging or pushing emissions, shall be made and recorded during the time an observer walks the topside of a battery from one end to the other. Each oven shall be observed in sequence. The observer shall record the battery identification, the points of topside emissions from each oven, and the oven number, and whether an oven was dampered off. Compliance shall be determined by application of the following formula:

$$\frac{(\# \text{ of charging ports with visible emissions})}{(\# \text{ of charging ports on operating ovens})} \times (100) = 2\% \text{ or less.}$$

E. Offtake Piping:

For purposes of determining compliance with the percent off take piping leakage standard, observations of any visible emissions from the off take piping shall be made by traversing the topside of the battery near the centerline. During the traverse, the observer may stray from near the centerline of battery and walk as close as possible to the offtake piping to determine whether an observed emission is emanating from the offtake piping. The observer shall traverse the battery once per each collector main. Therefore, to observe a battery with two collector mains, one observer may traverse the battery in one direction for one offtake system and traverse the battery in one direction for the second offtake system or two observers can traverse the battery in one direction. Each oven shall be observed in sequence. The observer shall record the battery identification, the points of offtake piping emission from any oven and the

oven number. Compliance shall be determined by application of the following formula:

$$\frac{(\# \text{ of offtake piping with visible emissions})}{(\# \text{ of offtake piping on operating ovens})} \times (100) = 5\% \text{ or less.}$$

F. **Pushing:** Compliance with the visible emission standards for pushing shall be determined in accordance with the following methods:

- (1) Visible emission observers shall be certified in accordance with the procedures specified at 40 C.F.R. Part 60 Appendix A. Method 9.
- (2) The provisions of Method 9 Section 2.5 shall not apply in that averaging shall not be used to determine compliance with the visible emissions performance standards.
- (3) In making observations of any pushing emissions control device outlet the observer shall be positioned in accordance with the provisions of Section 2.1 of Method 9 except that if it is an overcast day and the reader need not position himself with his back to the sun.
- (4) In viewing the pushing operation the observer shall stand on the coke side of the battery where a clear view of the push can be obtained. This generally should be a location on the ground, in the coke side yard, outside the hot car tracks approximately perpendicular to the observed oven. However, the observer is not restricted to the ground level, but may make the observation from some elevated level. If it is an overcast day or if the plume is in a shadow, the reader need not follow the requirements about positioning his back to the sun.
- (5) During the pushing operation, the reader shall observe all the pushing emissions including, but not limited to, fugitive emissions from the pushing emission control device and from open quench cars during travel.
- (6) Except as provided in paragraph (7) below, the reader upon observing any visible emissions with opacity equal to or greater than 20% opacity, as determined against any contrasting background, shall start an accumulating stopwatch. The reader shall stop the watch whenever the visible emissions are less than 20% opacity. Observations shall not be made until the coke side door machine is in the final spotted position and ready to receive coke at the oven to be pushed. The reader continues this procedure for the entire pushing operation. The reader shall independently observe emission from the pushing emission control device gas cleaning outlet and fugitive emissions from the pushing operation.

- (7) Pushing emissions during the transport of coke to quench tower shall be evaluated separately. In this case, the readers shall be positioned in accordance with paragraph 4 above.

G. **Combustion Stacks:** Compliance with the visible emission standard for combustion stacks shall be determined in accordance with the provisions of chapter 9.

CHAPTER 201

“DETERMINATION OF PM10 EMISSIONS EXHAUST GAS RECYCLE PROCEDURE,”
United States Environmental Protection Agency, 40 CFR 51 Appendix M (Method 201 and
201A)

CHAPTER 202

“DETERMINATION OF CONDENSIBLE PARTICULATE EMISSIONS FROM STATIONARY SOURCES,” United States Environmental Protection Agency, 40 CFR 51 Appendix M (Method 202)

CHAPTER 205

“VERIFICATION OF GAS DILUTION SYSTEMS FOR FIELD INSTRUMENT CALIBRATIONS,” United States Environmental Protection Agency, 40 CFR 51 Appendix M (Method 202)

CHAPTER 303

“DETERMINATION OF VISIBLE EMISSIONS FROM COKE OVEN BATTERIES,”
United States Environmental Protection Agency, 40 CFR 61 Appendix A (Method 303 thru
303A)

APPENDIX A

SUMMARY OF EPA EMISSION TEST METHODS
Title 40
Office of Air Quality Planning and Standards

<u>Method</u>	<u>Reference</u>	<u>Description</u>
<u>Part 51, Appendix M</u>		
201	55 FR 14246 04/17/90	PM-10 (EGR procedure).
201A	55 FR 14246 04/17/90	PM-10 (CSR procedure).
201A	55 FR 24687 06/18/90	Correction of equations.
201	55 FR 37606 09/12/90	Correction of equations.
202	56 FR 65433 12/17/91	Condensable PM.
203 P	57 FR 46114 10/07/92	COMS QA for compliance. (See Part 60, App F, Proc 3)
203A P	58 FR 61640 11/22/93	Visible Emissions - 2-6 min avg.
203B P	58 FR 61640 11/22/93	Visible Emissions - time exceptions.
203C P	58 FR 61640 11/22/93	Visible Emissions - instantaneous.
204	62 FR 32500 06/16/97	VOC Capture Efficiency.
204A	62 FR 32500 06/16/97	VOC Capture Efficiency.
204B	62 FR 32500 06/16/97	VOC Capture Efficiency.
204C	62 FR 32500 06/16/97	VOC Capture Efficiency.
204D	62 FR 32500 06/16/97	VOC Capture Efficiency.
204E	62 FR 32500 06/16/97	VOC Capture Efficiency.
204F	62 FR 32500 06/16/97	VOC Capture Efficiency.
205	59 FR 19590 05/30/94	Dilution calibration verification.
206	(Now CTM-027)	Ammonia (NH ₃)
207 P	62 FR 64532 12/08/97	Isocyanates.
<u>Part 60, Appendix A</u>		
1-8	42 FR 41754 08/18/77	Velocity, Orsat, PM, SO ₂ , NO _x , etc.
	43 FR 11984 03/23/78	Corr. and amend. to M-1 thru 8.
1-24	52 FR 34639 09/14/87	Technical corrections.
	52 FR 42061 11/02/87	Corrections.
2-25	55 FR 47471 11/14/90	Technical amendments.
1-29 P	62 FR 45639 08/27/97	Reformat and revise methods.

1-29	65 FR 61744	10/17/02	Performance specs.
1	48 FR 45034	09/30/83	Reduction of number of traverse points.
1	51 FR 20286	06/04/86	Alternative procedure for site selection.
1A	54 FR 12621	03/28/89	Traverse points in small ducts.
2A	48 FR 37592	08/18/83	Flow rate in small ducts - vol. meters.
2B	48 FR 37594	08/18/83	Flow rate - stoichiometry.
2C	54 FR 12621	03/28/89	Flow rate in small ducts - std. pitot.
2D	54 FR 12621	03/28/89	Flow rate in small ducts - rate meters.
2E	61 FR 9929	03/12/96	Flow rate from landfill wells.
2F	64 FR 26484	05/14/99	3D pitot for velocity.
2G	64 FR 26484	05/14/99	2D pitot for velocity.
2H	64 FR 26484	05/14/99	Velocity decay near the stack wall.
3	55 FR 05211	02/14/90	Molecular weight.
3/3B	55 FR 18876	05/07/90	Method 3B applicability.
3A	51 FR 21164	06/11/86	Instrumental method for O ₂ and CO ₂ .
3B	55 FR 05211	02/14/90	Orsat for correction factors and excess air.
3C	61 FR 9929	03/12/96	Gas composition from landfill gases.
3	48 FR 49458	10/25/83	Addition of QA/QC.
4	48 FR 55670	12/14/83	Addition of QA/QC.
5	48 FR 55670	12/14/83	Addition of QA/QC.
5	45 FR 66752	10/07/80	Filter specification change.
5	48 FR 39010	08/26/83	DGM revision.
5	50 FR 01164	01/09/85	Incorp. DGM and probe cal. procedures.
5	52 FR 09657	03/26/87	Use of critical orifices as cal stds.
5	52 FR 22888	06/16/87	Corrections.
5A	47 FR 34137	08/06/82	PM from asphalt roofing (Prop. as M-26).
5A	51 FR 32454	09/12/86	Addition of QA/QC.
5B	51 FR 42839	11/26/86	Nonsulfuric acid PM.
5C	Tentative		PM from small ducts.
5D	49 FR 43847	10/31/84	PM from fabric filters.
5D	51 FR 32454	09/12/86	Addition of QA/QC.
5E	50 FR 07701	02/25/85	PM from fiberglass plants.

5F	51 FR 42839	11/26/86	PM from FCCU.
5F	53 FR 29681	08/08/88	Barium titration procedure.
5G	53 FR 05860	02/26/88	PM from wood stove - dilution tunnel.
5H	53 FR 05860	02/26/88	PM from wood stove - stack.
5I	64 FR 53027	09/30/99	PM for RATA of PM CEMS.
6	49 FR 26522	06/27/84	Addition of QA/QC.
6	48 FR 39010	08/26/83	DGM revision.
6	52 FR 41423	10/28/87	Use of critical orifices for FR/vol meas.
6A	47 FR 54073	12/01/82	SO ₂ /CO ₂ - manual method.
6B	47 FR 54073	12/01/82	Auto SO ₂ /CO ₂ .
6A/B	49 FR 09684	03/14/84	Incorp. coll. test changes.
6A/B	51 FR 32454	09/12/86	Addition of QA/QC.
6C	51 FR 21164	06/11/86	Instrumental method for S ₀₂ .
6C	52 FR 18797	05/27/87	Corrections.
7	49 FR 26522	06/27/84	Addition of QA/QC.
7A	48 FR 55072	12/08/83	Ion chromatograph NO _x analysis.
7A	53 FR 20139	06/02/88	ANPRM.
7A	55 FR 21752	05/29/90	Revisions.
7B	50 FR 15893	04/23/85	UV NO _x analysis for nitric acid plants.
7A/B	Tentative		High SO ₂ interference.
7C	49 FR 38232	09/27/84	Alkaline permanganate/colorimetric for NO _x .
7D	49 FR 38232	09/27/84	Alkaline permanganate/IC for NO _x .
7E	51 FR 21164	06/11/86	Instrumental method for NO _x .
8	36 FR 24876	12/23/71	Sulfuric Acid mist and SO ₂ .
8	42 FR 41754	08/18/77	Addition of particulate and moisture.
8	43 FR 11984	03/23/78	Miscellaneous corrections
9	39 FR 39872	11/12/74	Opacity.
9A	46 FR 53144	10/28/81	Lidar opacity; called Alternative 1.
10	39 FR 09319	03/08/78	CO.
10	53 FR 41333	10/21/88	Alternative trap.
10A	52 FR 30674	08/17/87	Colorimetric method for PS-4.
10A	52 FR 33316	09/02/87	Correction notice.
10B	53 FR 41333	10/21/88	GC method for PS-4.
11	43 FR 01494	01/10/78	H ₂ S.
12	47 FR 16564	04/16/82	Pb.

12	49 FR 33842	08/24/84	Incorp. method of additions.
13A	45 FR 41852	06/20/80	F - colorimetric method.
13B	45 FR 41852	06/20/80	F - SIE method.
13A/B	45 FR 85016	12/24/80	Corr. to M-13A and 13B.
14	45 FR 44202	06/30/80	F from roof monitors.
14A	62 FR 52383	10/07/97	F from roof monitors- Alcan cassettes.
15	43 FR 10866	03/15/78	TRS from petroleum refineries.
15	54 FR 46236	11/02/89	Revisions.
15	54 FR 51550	12/15/89	Correction notice.
15A	52 FR 20391	06/01/87	TRS alternative/oxidation.
16	43 FR 07568	02/23/78	TRS from kraft pulp mills.
16	43 FR 34784	08/07/78	Amend. to M-16, H ₂ S loss after filters.
16	44 FR 02578	01/12/79	Amend. to M-16, SO ₂ scrubber added.
16	54 FR 46236	11/02/89	Revisions.
16	55 FR 21752	05/29/90	Correction of figure.(10%).
16A	50 FR 09578	03/08/85	TRS alternative.
16A	52 FR 36408	09/29/87	Cylinder gas analysis alternative method.
16B	52 FR 36408	09/29/87	TRS alternative/GC analysis of SO ₂ .
16A/B	53 FR 02914	02/02/88	Correction 16A/B.
17	43 FR 07568	02/23/78	PM, in-stack.
18	48 FR 48344	10/18/83	VOC, general GC method.
18	49 FR 22608	05/30/84	Corrections to M-18.
18	52 FR 51105	02/19/87	Revisions to improve method.
18	52 FR 10852	04/03/87	Corrections.
18	59 FR 19308	04/22/94	Revisions to improve QA/QC.
19	44 FR 33580	06/11/79	F-factor, coal sampling.
19	52 FR 47826	12/16/87	M-19A incorp, into M-19
19	48 FR 49460	10/25/83	Corr. to F factor equations and F _c value.
20	44 FR 52792	09/10/79	NO _x from gas turbines.
20	47 FR 30480	07/14/82	Corr. and amend.
20	51 FR 32454	09/12/86	Clarifications.
21	48 FR 37598	08/18/83	VOC leaks.
21	49 FR 56580	12/22/83	Corrections to Method 21.

21	55 FR 25602	06/22/90	Clarifying revisions.
22	47 FR 34137	08/06/82	Fugitive VE.
22	48 FR 48360	10/18/83	Add smoke emission from flares.
23	56 FR 5758	02/13/91	Dioxin/dibenzo furan.
23 P	60 FR 28378	05/31/95	Revisions and corrections.
24	45 FR 65956	10/03/80	Solvent in surface coatings.
24A	47 FR 50644	11/08/82	Solvent in ink (Prop. as M-29).
24	Tentative		Solvent in water-borne coatings.
24	57 FR 30654	07/10/92	Multicomponent coatings.
24	60 FR 47095	09/11/95	Radiation-cured coatings.
25	45 FR 65956	10/03/80	TGNMO.
25	53 FR 04140	02/12/88	Revisions to improve method.
25	53 FR 11590	04/07/88	Correction notice.
25A	48 FR 37595	08/18/83	TOC/FID.
25B	48 FR 37597	08/18/83	TOC/NDIR.
25C	61 FR 9929	03/12/96	VOC from landfills.
25D	59 FR 19311	04/22/94	VO from TSDF - purge procedure.
25E	59 FR 62896	12/06/94	VO from TSDF - vapor pressure procedure.
26	56 FR 5758	02/13/91	HCl.
26	57 FR 24550	06/10/92	Corrections to Method 26.
26	59 FR 19309	04/22/94	Add 26 HCl, halogens, other hydrogen halides.
26A	59 FR 19309	04/22/94	Isokinetic HCl, halogens, hydrogen halides.
27	48 FR 37597	08/18/83	Tank truck leaks.
28	53 FR 05860	02/26/88	Wood stove certification.
28A	53 FR 05860	02/26/88	Air to fuel ratio.
29	61 FR 18262	04/25/96	Multiple metals.
29	63 FR 06493	02/09/98	Amends promulgation dates.

Part 60, Appendix B

PS-2 - 9 P	62 FR 45639	08/27/97	Reformat and revise performance specs.
PS-2 - 9	65 FR 61744	10/17/02	Reformat and revise performance specs.
PS-1	48 FR 13322	03/30/83	Opacity.
PS-1 P	59 FR 60585	11/25/94	Revisions.
PS-1	65 FR 48914	08/10/00	Revisions, final rule.

PS-2	48 FR 23608	05/25/83	SO ₂ and NO _x .
PS 1-5	55 FR 47471	11/14/91	Technical amendments.
PS-3	48 FR 23608	05/25/83	CO ₂ and O ₂ .
PS-4	50 FR 31700	08/05/85	CO.
PS-4A	56 FR 5526	02/11/91	CO for MWC.
PS-4B P	61 FR 17495	04/19/96	CO and O ₂ for HWI (BIF rules)
PS-4B	64 FR 53032	09/30/99	Final rule.
PS-5	48 FR 32984	07/20/83	TRS.
PS-6	53 FR 07514	03/09/88	Velocity and mass emission rate.
PS-7	55 FR 40171	10/02/90	H ₂ S.
PS-8	59 FR 64580	12/15/94	VOC CEMS performance specifications.
PS-8A P	61 FR 17495	04/19/96	VOC CEMS for HWI (BIF rules)
PS-8A	64 FR 53033	09/30/99	Final rule.
PS-9	59 FR 64580	12/15/94	GC CEMS performance specifications.
PS-10 P	61 FR 17495	04/19/96	Metals CEMS (HWC MACT)
PS-11	69 FR 1786	01/12/04	PM CEMS.
PS-11A P	61 FR 17495	04/19/96	PM CEMS (HWC MACT).
PS-12A	70 FR 28606	05/18/05	Hg CEMS (Coal-fired Boilers)
PS-12 P	61 FR 17495	04/19/96	Hg CEMS (HWC MACT).
PS-13 P	61 FR 17495	04/19/96	HCl CEMS (HWC MACT).
PS-14 P	61 FR 17495	04/19/96	Cl CEMS (HWC MACT).
PS-15 P	62 FR 45372	08/27/97	FTIR CEMS.
PS-16 P	70 FR 45608	08/08/05	Predicative Emissions Monitoring Systems

Part 60, Appendix F

Proc 1	52 FR 21003	06/04/87	Quality assurance for CEMS.
Proc 1	56 FR 5527	02/11/91	Revision.
Proc 2	69 FR 1768	01/12/04	QA for PM CEMS
Proc 3 P	68 FR 24692	05/08/03	QA for COMS

Part 60, Appendix J

App-J	55 FR 33925	08/20/90	Wood stove thermal efficiency.
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Alternative Procedures and Miscellaneous

48 FR 44700	09/29/83	S-Factor method for sulfuric acid plants.
48 FR 48669	10/20/83	Corrections to S-Factor publication.
49 FR 30672	07/31/84	Add fuel analysis procedures for gas turbines.
51 FR 21762	06/16/86	Alternative PST for low level concentrations.
54 FR 46234	11/02/89	Misc. revisions to Appendix A, 40 CFR Part 60.
55 FR 40171	10/02/90	Monitoring revisions to Subpart J (Petr. Ref.).

Part 60

54 FR 06660	02/14/89	Test methods & procedures rev. (40 CFR 60).
54 FR 21344	05/17/89	Correction notice.
54 FR 27015	06/27/89	Correction notice.

Part 61, Appendix B

101-111 P	62 FR 45639	08/27/97	Reformat, revise, amend methods.
101	47 FR 24703	06/08/82	Hg in air streams.
101A	47 FR 24703	06/08/82	Hg in sewage sludge incinerators.
101A	61 FR 18262	04/25/96	Revisions - Consistency with Method 29.
101	49 FR 35768	09/12/84	Corrections to M-101 and 101A.
102	47 FR 24703	06/08/82	Hg in H ₂ streams.
103	48 FR 55266	12/09/83	Revised Be screening method.
104	48 FR 55268	12/09/83	Revised beryllium method.
105	40 FR 48299	10/14/75	Hg in sewage sludge.
105	49 FR 35768	09/12/84	Revised Hg in sewage sludge.
106	47 FR 39168	09/07/82	Vinyl chloride.
107	47 FR 39168	09/07/82	VC in process streams.
107	52 FR 20397	06/01/87	Alternative calibration procedure.
107A	47 FR 39485	09/08/82	VC in process streams.
108	51 FR 28035	08/04/86	Inorganic arsenic.
108A	51 FR 28035	08/04/86	Arsenic in ore samples.
108B	51 FR 22026	05/31/90	Arsenic in ore alternative.
108C	55 FR 22026	05/31/90	Arsenic in ore alternative.
108B/C	55 FR 32913	08/13/90	Correction notice.
111	50 FR 05197	02/06/85	Polonium-210.

114	54 FR 51695	12/15/89	Monitoring of radio nuclides.
115	54 FR 51702	12/15/89	Radon-222.

Part 61

53 FR 36972	09/23/88	Corrections.
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Part 63 Appendix A

303-306 P	62 FR 45639	08/27/97	Reformat and revise methods.
303-306	65 FR 61744	10/17/02	Reformat and revise methods.
301	57 FR 61970	12/29/92	Field data validation protocol.
301 P	69 FR 76642	12/22/04	Revisions
302			(Reserved)
303	58 FR 57898	10/27/93	Coke Oven Door Emissions.
304A	62 FR 2793	01/17/97	Biodegradation rate (vented).
304B	62 FR 2793	01/17/97	Biodegradation rate (enclosed).
305	59 FR 19590	04/22/94	Compound specific liquid waste.
306	60 FR 4948	01/25/95	Chromium from electroplaters/anodizers.
306A	60 FR 4948	01/25/95	Simplified Chromium sampling.
306B	60 FR 4948	01/25/95	Surface tension of chromium suppressors.
307	59 FR 61801	12/02/94	Solvent Degreaser VOC.
308 P	58 FR 66079	12/17/93	Methanol
308	63 FR 18504	04/15/98	Methanol (Pulp and Paper MACT I)
309 P	59 FR 29216	06/04./94	Aerospace solvent recovery material balance.
310A	62 FR 12546	03/17/97	Residual hexane in EPDM rubber.
310B	62 FR 12546	03/17/97	Residual hexane in EPDM rubber.
310C	62 FR 12546	03/17/97	Residual hexane in EPDM rubber.
311	60 FR 62930	12/07/95	VOC HAPS in furniture coatings.
312A	62 FR 12546	03/17/97	Residual styrene in SBR rubber.
312B	62 FR 12546	03/17/97	Residual styrene in SBR rubber.
312C	62 FR 12546	03/17/97	Residual styrene in SBR rubber.
313A	62 FR 12546	03/17/97	Residual styrene in PBR rubber.
313B	62 FR 12546	03/17/97	Residual styrene in PBR rubber.
314	Tentative		Halogenated compounds in solvents.
315	62 FR 52418	10/07/97	MeCl Extractable organic matter.
316 P	62 FR 15257	03/31/97	Formaldehyde - manual method.
317	Tentative		Phenol - manual method.
318 P	62 FR 52266	03/31/97	Formaldehyde, phenol, methanol with FTIR.

319	P	61 FR 55862 10/29/96	Filter efficiency; paint over-spray
319		63 FR 46525 03/27/98	Filter efficiency, paint over-spray.
320		64 FR 31898 06/14/99	Extractive FTIR.
321		64 FR 31898 06/14/99	FTIR for HCl from Portland Cement kilns.
322	P	63 FR 14244 03/24/98	GFC/IR for HCl from Portland cement kilns.
323	P	68 FR 1925 01/14/03	Formaldehyde from Natural Gas-Fired sources.

P = Proposal

Tentative = Under evaluation