

December 21, 2009

Charlie Clark, VP of HR & Government Affairs Martin Operating Partnership L. P. 484 East 6th Street Smackover, AR 71762

Dear Mr. Clark:

The enclosed Permit No. 1227-AR-17 is your authority to construct, operate, and maintain the equipment and/or control apparatus as set forth in your application initially received on 8/26/2009.

After considering the facts and requirements of A.C.A. §8-4-101 et seq., and implementing regulations, I have determined that Permit No. 1227-AR-17 for the construction, operation and maintenance of an air pollution control system for Martin Operating Partnership L. P. be issued and effective on the date specified in the permit, unless a Commission review has been properly requested under Arkansas Department of Pollution Control & Ecology Commission's Administrative Procedures, Regulation 8.603, within thirty (30) days after service of this decision.

All persons submitting written comments during the thirty (30) day, and all other persons entitled to do so, may request an adjudicatory hearing and Commission review on whether the decision of the Director should be reversed or modified. Such a request shall be in the form and manner required by Regulation 8.603, including filing a written Request for Hearing with the APC&E Commission Secretary at 101 E. Capitol Ave., Suite 205, Little Rock, Arkansas 72201. If you have any questions about filing the request, please call the Commission at 501-682-7890.

Sincerely,

Mike Bates Chief, Air Division

Enclosure

#### **RESPONSE TO COMMENTS**

#### MARTIN OPERATING PARTNERSHIP L. P. PERMIT #1227-AR-17 AFIN: 70-00039

On October 19, 2009, the Director of the Arkansas Department of Environmental Quality gave notice of a draft permitting decision for the above referenced facility. During the comment period, written comments on the draft permitting decision were submitted by the facility. The Department's response to these issues follows.

#### Comment #1:

The permittee requested to make the following changes to the permit:

• The facility ownership and name be changed from Cross Oil Refining and Marketing, Inc. to Martin Operating Partnership L. P

#### **Response to Comment #1:**

The facility ownership and name were revised in the permit.

# ADEQ MINOR SOURCE AIR PERMIT

Permit No. : 1227-AR-17

IS ISSUED TO:

Martin Operating Partnership L. P. 484 East 6th Street Smackover, AR 71762 Union County AFIN: 70-00039

THIS PERMIT IS THE ABOVE REFERENCED PERMITTEE'S AUTHORITY TO CONSTRUCT, MODIFY, OPERATE, AND/OR MAINTAIN THE EQUIPMENT AND/OR FACILITY IN THE MANNER AS SET FORTH IN THE DEPARTMENT'S MINOR SOURCE AIR PERMIT AND THE APPLICATION. THIS PERMIT IS ISSUED PURSUANT TO THE PROVISIONS OF THE ARKANSAS WATER AND AIR POLLUTION CONTROL ACT (ARK. CODE ANN. SEC. 8-4-101 *ET SEQ*.) AND THE REGULATIONS PROMULGATED THEREUNDER, AND IS SUBJECT TO ALL LIMITS AND CONDITIONS CONTAINED HEREIN.

Signed:

Mike Bates Chief, Air Division

December 21, 2009

Date

# Table of Contents

Section I: FACILITY INFORMATION	
Section II: INTRODUCTION	
Summary of Permit Activity	
Regulations	7
Total Allowable Emissions	
Section III: PERMIT HISTORY	9
Section IV: EMISSION UNIT INFORMATION	
Section V: INSIGNIFICANT ACTIVITIES	
Section VI: GENERAL CONDITIONS	
40 CFR Part 60, Subpart Ka	APPENDIX A
40 CFR Part 60, Subpart GG	APPENDIX B
40 CFR Part 61, Subpart FF	APPENDIX C
40 CFR Part 60, Subpart Kb	APPENDIX D
Example of Loading Rack Calculations	APPENDIX E

List of Acronyms and Abbreviations

A.C.A.	Arkansas Code Annotated
AFIN	ADEQ Facility Identification Number
CFR	Code of Federal Regulations
СО	Carbon Monoxide
HAP	Hazardous Air Pollutant
lb/hr	Pound Per Hour
No.	Number
NO <sub>x</sub>	Nitrogen Oxide
PM	Particulate Matter
PM <sub>10</sub>	Particulate Matter Smaller Than Ten Microns
$SO_2$	Sulfur Dioxide
Тру	Tons Per Year
UTM	Universal Transverse Mercator
VOC	Volatile Organic Compound

# Section I: FACILITY INFORMATION

PERMITTEE:	Martin Operating Partnership L. P.
AFIN:	70-00039
PERMIT NUMBER:	1227-AR-17
FACILITY ADDRESS:	484 East 6th Street Smackover, AR 71762
MAILING ADDRESS:	484 East 6th Street Smackover, AR 71762
COUNTY:	Union County
CONTACT NAME:	Charlie Clark
CONTACT POSITION:	VP of HR & Government Affairs
TELEPHONE NUMBER:	870-881-8700
REVIEWING ENGINEER:	Parviz Mokhtari
UTM North South (Y):	Zone 15: 3691692.87 m
UTM East West (X):	Zone 15: 526334.70 m

# Section II: INTRODUCTION

# Summary of Permit Activity

Martin Operating Partnership L. P (Martin) operates an oil refinery at 484 East Sixth Street, in Smackover, Union County, Arkansas, 71762. MOP submitted a de minimis application to make the following changes:

- Install an additional onsite distillate storage Tank #328 and lube oil storage Tanks #P033 through #P059 at SN-27
- Rename the Lube Stripper Reboiler (SN-12) to Lube Stripper Charge Heater (SN-12)
- Convert the existing lube oil storage Tanks #113 to a distillate storage tank at SN-27
- Install an offsite distillate Tank #116 at SN-29
- Install a new lube oil loading/unloading dock (SN-34)
- The permittee ownership and name be changed from Cross Oil Refining and Marketing, Inc. to Martin Operating Partnership L. P

The above listed changes could result in potential emission rate increases of 0.5 ton per year (tpy) of VOC, 0.05 tpy of Benzene, 0.1 tpy of Hexane, 0.02 of Toluene, and 0.01 tpy of Xylene.

# Process Description:

Martin is a refinery that processes crude oil into naptha, diesel fuel, lube oils, and asphalt. crude oil is charged from storage and is preheated with heat exchangers. Water is added to the crude oil to aid in removing salts. The crude passes through a series of electrostatic desalting units which separates the saltwater from the crude. From the desalters, the crude is heated through a series of heat exchangers and then through the Crude Charge Heater (SN-01). Finally, the crude is sent to the first atmospheric distillation tower where the oil is separated into naphtha, diesel fuel, No. 2 lube oil, No. 3 lube oil, and No. 4 lube oil.

Naphtha and other non-condensable gases flow overhead from the atmospheric tower. The naphtha is cooled in a condenser and then flows to an accumulator from which the liquid is pumped back to the tower as reflux. Excess naphtha product is drawn off of the accumulator, and then pumped to product storage for sales. Diesel fuel and Lube Oils No. 2, 3, and 4 are drawn off of the side of the tower, routed through strippers to remove non-condensable gases, and then pumped through heat exchangers and on to storage for product sales.

The bottoms stream off of the atmospheric tower is pumped through the Vacuum Tower Charge Heater (SN-02) before being charged to the vacuum distillation column. The reduced crude is separated in the vacuum tower to produce the heavier grades of lube oil, Nos. 7, 9, 10, and 11. The vacuum tower bottoms are asphalt flux, which is pumped through heat exchangers to storage for sales or for further processing in an asphalt blowstill.

Flux is charged from storage through the Asphalt Blowstill Charge Heater (SN-03) to one of two blowstills. While the flux is being pumped into the blowstill, air is blown into the bottom of the still through a distribution header. The air oxidizes the flux causing it to polymerize and thus increases the melting point and hardness of the asphalt. Oxygen, nitrogen, volatile organic

compounds (VOC), and sulfur dioxide  $(SO_2)$  are produced as byproducts of the operation. These byproducts are routed through an incinerator and the waste heat boiler (SN-04) before being vented to the atmosphere. The asphalt product is loaded into trucks at one of the two asphalt loading racks (SN-15 and SN-16) or loaded into tank cars at the Asphalt/Distillate Tank Car Loading Rack (SN-32).

The lube oils produced by both atmospheric and vacuum distillation are further processed in a set of heater exchangers and then are passed to the hydrotreaters. The oils are heated with heat exchangers and two hydrotreaters, the Hydrotreater Charge Heater (SN-07) and Lube Charge Pre-Heater (SN-30), before being pumped into the top of the reactor. The hot lube oils combine with hydrogen at the top of the reactor before passing through a catalyst bed. Sulfur in the oil reacts with the hydrogen to form hydrogen sulfide gas. The hydrogen sulfide gas also saturates the aromatic compounds in the oil, removes heavy metals, and converts some of the nitrogen to ammonia.

The reactor effluent flows to a high pressure separator where the excess hydrogen, hydrogen sulfide, and ammonia gases flash off. From the high pressure separator, the oil flows to a low pressure separator where additional light ends flash off. The oil then flows to a lube oil stripper where the remaining hydrogen sulfide is removed by steam stripping. The Lube Stripper Reboiler (SN-12) supplies heat to the lube oil stripper. From the lube oil stripper, the oil flows to a vacuum lube stripper where any entrained water is vacuum stripped from the product. The bottoms from the vacuum stripper are routed to finished lube storage for blending and sales. The finished lube products are loaded at one of the three lube oil loading racks (SN-17, SN-18 and SN-21). Martin also operates Lube Oil and Additive Loading Rack (SN-33) that transfers the contents back and forth between rail cars and truck cars.

The waste gas from the high pressure separator is routed to a caustic scrubber where the gas enters the bottom of the column. The gas flows countercurrent to a caustic solution, which removes the hydrogen sulfide in the gas. The waste gas from the low pressure separator is combined with the gases from the lube stripper and the lube vacuum stripper. The combined gas stream is then treated in a two stage caustic scrubber system. The clean hydrogen gas from the scrubbers is then sent back to the hydrotreater reactor.

The primary hydrogen is supplied to the hydrotreater by a steam/methane reformer. Natural gas is compressed and heated in a preheat exchanger, and combined with steam. The mixture is charged to the Hydrogen Plant Heater (SN-08) where it passes over a nickel catalyst and reacts to produce hydrogen and carbon oxides. The gases leaving the reactor are routed to a shift converter which contains an iron-chromium catalyst. Most of the carbon monoxide (CO) in the gas is converted to carbon dioxide (CO<sub>2</sub>) and hydrogen (H<sub>2</sub>). The CO<sub>2</sub> and H<sub>2</sub> gas then flow to a pressure swing absorption (PSA) system where the CO<sub>2</sub> and other impurities are removed.

Steam is produced in a boiler and a cogeneration unit at the facility. The cogeneration unit (SN-25) has a gas-fired turbine, which along with the boiler, uses natural gas as fuel.

# Regulations

The following table contains the regulations applicable to this permit.

Regulations			
Arkansas Air Pollu	Arkansas Air Pollution Control Code, Regulation 18, effective January 25, 2009		
Regulations of the	Arkansas Plan of Implementation for Air Pollution Control,		
Regulation 19, effe	ective July 18, 2009		
All Sources	Arkansas Air Pollution Control Code, Regulation 18, effective January 25, 2009		
All Sources Regulations of the Arkansas Plan of Implementation for Air Pollution Control, Regulation 19, effective July 18, 2009			
SNI 27	40 CFR Part 60, Subpart Ka – Standards of Performance for Storage Vessels for Petroleum Liquids for which Construction, Reconstruction, or Modification commenced after May 18, 1978, and prior to July 23, 1984		
SIN-27	40 CFR Part 60, Subpart Kb – Standards of Performance for Volatile Organic Liquid Storage Vessels (Including Petroleum Liquid Storage Vessels) for Which Construction, Reconstruction, or Modification Commenced After July 23, 1984		
SN-25 40 CFR Part 60, Subpart GG – Stationary Gas Turbines			
All Sources 40 CFR Part 61, Subpart FF – National Emission Standards for Hazardous Air Pollutants, Benzene Waste Operations			

Subpart FF applies because MOP is a petroleum refinery. The boiler SN-26 was manufactured in 1971 and is therefore not subject to the requirements of NSPS Subpart Dc. The reformer SN-08 burns pipeline quality gas so it is not subject to 40 CFR Part 60, Subpart J – *Standards of Performance for Petroleum Refineries*.

40 CFR Part 60, Subpart Kb applies for SN-27 distillate oil tank (Tank #319) because the storage capacity is in excess of 75 cubic meters (19813 gallons) and has a vapor pressure grater than 3.5 kilopascal (kPa). Although the lube oil tanks #026p, #027p, #028p, #029p (1,182 barrel), #031p, #032p, #229, #230, #231, #232, #233, #332, #333, #334, #335, #336, #337, #338, #339, #340, #P041, #P042, #P043, #P044, #P045, #P046, #P047, #P048, #P049, #P050, #P051, and #P052, are greater than 75 cubic meters (19813 gallons), the vapor pressure of the lube oil (1.33 kPa) is blow the NSPS threshold of 3.5 kPa, therefore, the lube oil tanks are not subject to any other requirements of Subpart Kb.

40 CFR Part 60 Subpart GGG – Standards Performance for Equipment Leaks of VOC in Petroleum Refineries. This regulation applies to certain compressors and other equipment in VOC service installed after January 4, 1983. The permittee consultant's letter dated April 25, 2007 states that all compressors installed after the effective date are in hydrogen service and are not in VOC service. Therefore, no compressors at this facility are subject to Subpart GGG.

× .

# Total Allowable Emissions

The following table is a summary of emissions from the facility. This table, in itself, is not an enforceable condition of the permit.

TOTAL ALLOWABLE EMISSIONS			
Delbutant	Emissi	sion Rates	
	lb/hr	tpy	
РМ	11.4	47.4	
PM <sub>10</sub>	11.4	47.4	
SO <sub>2</sub>	2.7	8.4	
VOC	52.3	66.6	
СО	21.8	93.0	
NO <sub>x</sub>	25.8	81.6*	
2,2,4- Trimethylpentane	0.20	0.13	
Benzene	1.49	3.70	
Cumene	0.06	0.09	
Ethylbenzene	0.14	0.35	
Hexane	5.40	8.70	
Naphthalene	0.01	0.01	
Phenol	0.01	0.01	
Styrene	0.01	0.01	
Toluene	0.91	1.65	
Xylene	0.36	0.71	

\* Includes 53.0 tpy emission bubble for SN-01, SN-25, and SN-26

#### Section III: PERMIT HISTORY

- 1227-A Issued on December 9, 1991, this was the first operating permit for CORC. This permit included the inclusion of a recently installed naphtha storage tank.
- 1227-AR-1 This modification, issued on July 14, 1992, covered the installation of a lube-oil stripper/reboiler/heater at the facility.
- 1227-AR-2 Issued on November 20, 1992, this permit allowed the installation of a replacement boiler. The installation of the replacement boiler classified the facility as a major source subject to Title V permitting since NO<sub>x</sub> emissions exceeded 100 tons per year.
- 1227-AR-3 This permit was issued on August 5, 1997 due to an emissions inventory that discovered that the facility did not have actual emissions greater than the major source threshold. Therefore, Cross Oil Refining and Marketing, Inc. was removed from major source status. Additionally, a cogeneration unit and the #4 boiler were added as sources at the facility.
- 1227-AR-4 This modification was issued on June 29, 2000 and covers the relocation of a 94.3 MMBTU/hr natural gas fired boiler to the facility. Several boilers at the plant had reached the end of their useful life. This new boiler incorporates a low NO<sub>x</sub> burner and flue gas recirculation to minimize emissions. Additionally, it was planned that a duct burner would work in conjunction with a cogeneration unit, but the duct burner was never installed and is being removed from the permit and the cogeneration unit calculations adjusted accordingly. In order for CORC to install the duct burner, a new application must be submitted. Also, the existing #3 Boiler (SN-06) has been retired from operation and so the emissions from this source have been removed.
- 1227-AR-5 This permit was issued on April 29, 2002 and addressed a proposal to make the following changes to some storage tanks:
  - 1. Two tanks which stored lube oil product were destroyed in a fire in 1999 and have not yet been replaced. The refinery plans to move two existing identical tanks to replace these tanks. These tanks will be designated as #328 and #329. Both tanks have a capacity of 1,000 barrels each (42,000 gallons) and will be subject to the record keeping provisions of 40 CFR Part 60, Subpart Kb, since they will store organic liquids;
  - Two tanks (#330 and #331), which will store lube oil product, are planned for installation. The tanks have a capacity of 10,000 barrels each (420,000 gallons) and will be subject to record keeping provisions of 40 CFR 60, Subpart Kb, since they will store organic liquids;
  - 3. Two tanks (#332 and #333), which will store lube oil product, are planned for installation. The tanks have a capacity of 500 barrels each (21,000 gallon) and will be subject to the record keeping provisions of 40 CFR 60, Subpart Kb, since they will store organic liquids; and

4. One existing tank (#284), which is currently permitted to store lube oil, will be removed from service.

The total emissions VOC from the six tanks amounted to less than 0.02 tons annually.

- 1227-AR-6 This permit was issued on August 2, 2002 and addressed the following modifications to the facility:
  - 1. Tank #329 was recently permitted as a lube oil storage tank. This tank will be equipped with an internal floating roof and will store naphtha. The tank will be subject to the provisions of NSPS Subpart Kb. A floating roof meeting the requirements of 40 CFR 60 60.112b (a) (1) will be installed;
  - 2. The existing naphtha tank #206 will be converted to a lube oil storage tank. It was constructed in 1980 and will not be modified with this project. Therefore, the tank will not be subject to NSPS Subpart Kb after the change of service;
  - 3. Tanks #291 and #292 will be changing service from diesel to lube oil storage. The tanks were constructed in 1980. Therefore, the tanks will not be subject to NSPS Subpart Kb after the change of service; and
  - 4. Tank #113 is currently permitted as a crude oil storage tank subject to NSPS Subpart Ka. It will be changing service to store Cross Oil's B Series lube oil (a mixture of lube oil and diesel). The tank was constructed in 1980. Therefore, the tank will not be subject to NSPS Subpart Kb (or NSPS Subpart Ka due to the low vapor pressure of the lube oil) after the change of service.

The above changes in tank service resulted in a decrease in VOC emissions of 2.9 tons per year. Without considering the reduction in emissions due to the change in service of the tanks, the total increase associated with this project is 0.74 tons VOC per year.

- 1227-AR-7 This permit was issued on October 29, 2002 and addressed the following modifications to the permit:
  - 1. Addition of six tanks (001 through 006), which will store lube oil product, are planned for installation. The tanks have a capacity of 15,250 gallons each and will be subject to the record keeping provisions of 40 CFR 60, Subpart Kb, since they will store organic liquids; and
  - 2. Addition of three tanks (007 through 009), which will store lube oil product, are planned for installation. The tanks have a capacity of 2,000 gallons each and will not be subject to the record keeping provisions of 40 CFR 60, Subpart K.

The above changes resulted in an increase of VOC emissions of 0.2 tons per year.

1227-AR-8 This permit was issued on May 30, 2003 and allowed the facility to modify its

existing permitted emission rates based upon emission factors, physical property data, facility operating conditions, and revised emissions modeling. In addition, the facility proposed to include hazardous air pollutant (HAP) emissions, which were not included in permit 1227-AR-7, to permit emissions from offsite storage tanks, and to correct opacity limits. No production increases were proposed. The proposed changes resulted in an increase of 0.7 tons per year of SO<sub>2</sub> emissions, 31.0 tons per year of CO emissions, and 15.8 tons per year of HAP emissions.

- 1227-AR-9 This permit was issued on April 19, 2004, and it allowed the facility to install two new 3,500 gallon lube oil storage tanks (#010p and #011p). The proposed change resulted in no production and negligible annual emissions increases.
- 1227-AR-10 This permit was issued September 28, 2004. CORC's proposal included the installation of one new 21,000 gallon reclaimed oil storage tank, one new 42,000 gallon reclaimed oil storage tank, and six new 16,800 gallon lube oil storage tanks. In addition to installing the new storage tanks, Cross Oil requested to remove #500, re-designate #332 as #500, and to re-designate #312 and #333 as #512 and #513, respectively.
- 1227-AR-11 This permit was issued April 11, 2005. The permit revision contained the following changes: converted two tanks containing asphalt to lube oil (Tank #223 and #224); added a new lube oil tank (Tank #331); converted a tank currently containing lube oil to naptha (Tank #312); corrected the current tank numbering by shifting Tank #012P though #017P each up one number, resulting in Tank #013P through #018P; added two new lube oil tanks (Tanks #012P and #019P of SN-27); added a seasonal 50 horsepower (0.125 MMBTU/hr) low pressure boiler as an insignificant activity; added a pre-heat lube charge heater, with a design rating of 6.0 MMBTU/hr (SN-30) with low NO<sub>x</sub> burners; added 7 heat exchangers (no source number) to the process in order to increase efficiency and reduce reliance on the crude heaters; and removed the crude oil throughput limit. Total annual emission increases were 0.2 ton/yr PM/PM<sub>10</sub>, 0.1 ton/yr SO<sub>2</sub>, 0.4 ton/yr VOC, 1.3 ton/yr NO<sub>x</sub>, and 2.2 ton/yr CO.
- 1227-AR-12 This permit was issued on September 23, 2005. The facility modified their permit in order to increase the annual asphalt throughput at the Blow Still Incinerator Waste Heat Boiler (SN-04) and to remove the testing requirements for SO<sub>2</sub> at the Cogeneration Unit (SN-25), which is a natural gas source. Annual particulate emissions increased by 14.6 tons/year as a result of the asphalt throughput.
- 1227-AR-12 This permit was issued on October 24, 2006. The permit revision contained the following changes:
  - Installation of five 700 barrel (29,400 gallon) lube oil storage tanks at SN-27 Tanks #020p, #022p, #023p, #024p, and #025p;
  - Install one 500 barrel (21,000 gallon) lube oil storage tank at SN-27 Tank #021p;

- Remove the 1000 barrel (42,000 gallon) reclaimed oil storage tank and replace it with a 400 barrel (16,800 gallon) reclaimed oil storage tank at SN-27 Tank #503;
- Correct the permitted HAP lb/hr emission rate limits for the Diesel/Naphtha/Kerosene Loading Rack (SN-14);
- Correct various typographical errors; and
- Install one 5,000 barrel (210,000 gallon) lube oil storage tank at SN-27 Tank #030p.

The total permitted annual emission rate increases were:  $0.1 \text{ tpy NO}_x$ , 0.01 tpy cumene, 0.01 tpy phenol, and 0.01 tpy toluene.

1227-AR-13 This permit was issued October 26, 2006. The permit revision contained the following changes:

Cross Oil's proposal included the installation of five 700 barrel (29,400 gallons) lube oil storage tanks at SN-27 (Tanks #020p, #022p, #023p, #024p, and #025p), install one 500 barrel (21,000 gallons) lube oil storage tank at SN-27 (Tank #021p), Install one 5,000 barrel (210,000 gallons) lube oil storage tank at SN-27 (Tank #030p), and remove the 1000 barrel (42,000 gallon) reclaimed oil storage tank and replace it with a 400 barrel (16,800 gallon) reclaimed oil storage tank at SN-27 (Tank #503). In addition, the facility requested the permitted HAP lb/hr emission rate limits to be corrected for the Diesel/Naphtha/Kerosene Loading Rack (SN-14), and to correct various typographical errors. The typographical errors included adding SN-23's VOC emissions back to Specific Condition #1; adding naphthalene to the Total Allowable Emissions Table; and correcting the VOC, NO<sub>x</sub>, and toluene total emission rates in the Total Allowable Emissions Table.

The total permitted annual emission rate increases include: 0.1 tpy NO<sub>x</sub>, 0.01 tpy cumene, 0.01 tpy phenol, and 0.01 tpy toluene.

- 1227-AR-14 This permit was issued August 27, 2007. The permit revision contained the following changes:
  - Installation of Six (6) lube oil storage tanks at SN-27 with the following storage capacities: Tanks #026p (1,182 barrel), #027p (1,049 barrel), #028p (862 barrel), #029p (1,182 barrel), #031p (1,182 barrel), #032p (1,049 barrel);
  - Installation of one 1,000 barrel distillate oil storage tank (Tank #319) at SN-27;
  - Removing of the 1000 barrel reclaimed oil storage tank and replacing it with a 400 barrel reclaimed oil storage tank at SN-27 – Tank #504;
  - Installation of four (4) 280 barrel reclaimed oil storage tanks at SN-27; Tanks #514, #515, #516, and #517;
  - Add an asphalt tank car loading rack;
  - Add a distillate lube oil loading rack; and

• Revise insignificant activity list in the existing permit.

Cross Oil also submitted a DeMinimis application to perform the necessary piping modifications to allow for pipeline quality natural gas to be fired at Blow Still Incinerator Waste Heat Boiler (SN-04), Lube Stripper Reboiler (SN-12), and Lube Charge Heater (SN-30); to include use of mixed gas at the Crude Charge Heater (SN-01), Vacuum Tower Charge Heater (SN-02), the Asphalt Below Charge Heater (SN-03), and the Lube Precharge Heater (SN-07); to make necessary improvement to NASH plant to ensure the sulfur content in the waste gas produced onsite remain below 0.1 gr/dscf; and to change the source descriptions for SN-07 to the "Lube Precharge Heater" and SN-30 to the "Lube Charge Heater".

The permitted emission increases were due to these modifications: 3.8 tons per year (tpy) PM/PM10, 5.8 tpy SO<sub>2</sub>, 1.1 tpy VOC, 0.01 tpy 2,2,4-trimethylpentane, 0.18 tpy benzene, 0.01 tpy cumene, 0.01 tpy ethylbenzene, 0.34 tpy hexane, 0.06 tpy toluene, and 0.04 tpy xylene.

- 1227-AR-15 This permit was issued January 26, 2009. This permitting action was necessary to:
  - Add two (2) 400 barrel asphalt plasticizer tanks #227 and #228 at SN-27,
  - Replace three (3) 280 barrel reclaimed oil storage tanks #514, #515, and #516 (previously permitted) with three (3) reclaimed oil tanks of different size (400 barrel) and throughput at SN-27,
  - Remove the 280 barrel reclaimed oil storage tank #517at SN-27,
  - Add a lube oil and additive loading/unloading rack (SN-33), and
  - Add a lube oil packaging operation into insignificant activities list.

The permitted emission increased due to this De Minimis modification is 0.2 tons per year (tpy) VOC. The new tanks #227, #228, #514, #515, and #516 are not subject to 40 CFR 60 Subpart Ka and Kb.

Also, Cross Oil submitted another DeMinimis application on September 3, 2008 to modify its existing minor source to address the throughput at the Distillate Lube Oil Truck Loading Rack and associated storage tanks:

- Increase the throughput at the distillate lube oil storage tank (Tank # 319) from 1,302,000 gal/yr to 2,730,000 gal/yr.
- Increase the throughput at the Distillate Lube Oil loading Rack SN-31.
- Add a condition to address the throughput and tracking for the gasoline tank at SN-27.
- Record keeping requirements for SN-31 and SN-32 have been added to the permit.

The permitted emissions for SN-31 increased by: 1.2 tons per year (tpy) VOC, Benzene 0.15 tpy, Ethylbenzene 0.02 tpy, Hexane 0.3 tpy, Toluene 0.05, and Xylene 0.03 tpy.

Additionally, Cross Oil submitted a summary of all of the tanks with the revised capacities, turnovers, and emissions for Onsite Storage Tanks SN-27, Sandyland Storage Tanks SN-28, and Miller's Storage Tanks SN-29 by email dated June 26, 2008. Due to this revision the emission limits for these sources were decreased. The overall permitted emissions decreased due to this DeMinimis modification by: 16.2 tpy VOC, 2,2,4-Trimethylpentane 0.06 tpy, Benzene 0.6 tpy, Cumene 0.02 tpy, Ethylbenzene 0.04 tpy, Hexane 1.02 tpy, Toluene 0.48, and Xylene 0.17 tpy.

1227-AR-16 This permit was issued March 27, 2009. This permitting action was necessary to:

- Install fourteen (14) onsite lube oil storage tanks #229, #230, #231, #232, #233, #332, #333, #334, #335, #336, #337, #338, #339, and #340 at SN-27.
- Allow the distillate to be loaded at the Asphalt Tank Car Loading Rack SN-32.

There was an increase in lube oil throughput as a result of the additional storage tanks because the tanks were being installed to allow for reduced throughput at other lube oil tanks. The introduction of distillate to SN-32 resulted in an increase of 1,500,000 gallons per year of distillate. The permitted emissions increased 1.3 ton per year (tpy) of VOC, 0.02 tpy of 2,2,4-Trimethylpentane, 0.14 tpy of Benzene, 0.01 tpy Cumene, 0.02 tpy of Hexane, 0.07 tpy of Toluene, and 0.03 tpy of Xylene.

# Section IV: EMISSION UNIT INFORMATION

# Specific Conditions

1. The permittee shall not exceed the emission rates set forth in the following table. [Regulation 19, §19.501 et seq., and A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]

SN	Description	Pollutant	lb/hr	tpy
		PM <sub>10</sub>	0.3	1.0
		SO <sub>2</sub>	0.9	4.0
01	Crude Charge Heater	VOC	0.2	0.8
		CO	2.6	11.1
			2.8	*
		PM <sub>10</sub>	0.1	0.3
i		SO <sub>2</sub>	0.3	1.1
02	Vacuum Tower Charge Heater	VOC	0.1	0.2
		CO	0.8	3.3
		NO <sub>x</sub>	0.9	3.9
		PM <sub>10</sub>	0.1	0.3
1		SO <sub>2</sub>	0.3	0.3
03	Asphalt Blow Still Charge Heater	VOC	0.1	0.2
		CO	0.7	2.9
		NO <sub>x</sub>	0.8	3.5
		PM <sub>10</sub>	8.5	37.0
		SO <sub>2</sub>	0.1	0.1
04	Blow Still Incinerator Waste Heat Boiler	VOC	0.1	0.4
		CO	1.2	5.0
		NO <sub>x</sub>	1.4	5.9
05	Boiler #1		Retired	
06	Boiler #3		Retired	
		PM <sub>10</sub>	0.1	0.2
		SO <sub>2</sub>	0.2	0.8
07	Lube Precharge Heater	VOC	0.1	0.2
	5	CO	0.5	2.2
		NO <sub>x</sub>	0.6	2.6
		PM <sub>10</sub>	0.3	1.0
		$SO_2$	0.1	0.1
08	Hydrogen Plant Heater/Reactor	VOC	0.2	0.8
		CO	2.6	11.1
		NO <sub>x</sub>	1.3	5.6
00		PM <sub>10</sub>	0.1	0.1
09	High Pressure Flare	$SO_2$	0.1	0.1

SN	Description	Pollutant	lb/hr	tpy
		VOC	0.1	0.1
		CO	0.1	0.1
		NO <sub>x</sub>	0.1	0.1
		$PM_{10}$	0.1	0.1
		$SO_2$	0.1	0.1
10	Low Pressure Flare	VOC	0.1	0.1
		CO	0.1	0.1
		NO <sub>x</sub>	0.1	0.1
11	Naphtha Storage Tank		Retired	
		$PM_{10}$	0.1	0.5
		SO <sub>2</sub>	0.1	0.1
12	Lube Stripper Charge Heater	VOC	0.1	0.4
1		CO	1.1	4.7
		NO <sub>x</sub>	1.3	5.6
13	Boiler #2		Retired	
14	Diesel/Naphtha/Kerosene Loading Rack	VOC	37.4	4.1
15	Asphalt Truck Loading Rack #1	VOC	0.1	0.1
16	Asphalt Truck Loading Rack #2	VOC	0.1	0.1
17	Lube Oil Truck Loading Rack	VOC	0.1	0.1
18	Lube Oil Truck Loading Rack	VOC	0.1	0.1
21	Lube Oil Rail Car Loading Rack	VOC	0.2	0.2
23	Fugitive Emissions	VOC	3.6	15.7
24	Wastewater Emissions	VOC	2.0	8.6
		PM <sub>10</sub>	0.6	2.5
		SO <sub>2</sub>	0.1	0.3
25	Cogeneration Unit	VOC	0.7	2.8
		CO	5.5	23.8
		NO <sub>x</sub>	12.3	*
		PM <sub>10</sub>	1.0	4.2
		SO <sub>2</sub>	0.3	1.3
26	Boiler #4	VOC	0.4	1.7
		CO	6.1	26.5
ļ		NO <sub>x</sub>	3.8	*
	Onsite Storage Tanks			
27	(181 Tanks)	VOC	1.1	4.6
	Tanks Subject to 40 CFR Part 60, Subpart Ka:			
	113, 197, 287, 288, 289, 290, 296, 297, 298,			)

SN	Description	Pollutant	lb/hr	tpy
	299, 312, 313, 314, 315, 206, 266, 291, 292, 329, and Tank Subject to 40 CFR Part 60, Subpart Kb: Tank #319			
28	Sandyland Storage Tanks Tanks #104, #110, #111 Tanks Subject to 40 CFR Part 60, Subpart Ka: #111	VOC	3.6	15.7
29	Miller's Storage Tanks Tanks #114, #115, and #116	VOC	1.3	5.4
30	Lube Charge Heater	PM <sub>10</sub> SO <sub>2</sub> VOC CO NO <sub>x</sub>	0.1 0.1 0.5 0.3	0.2 0.1 0.1 2.2 1.3
31	Distillate Lube Oil Truck loading Rack	VOC	0.57	2.5
32	Asphalt/Distillate Tank Car Loading Rack	VOC	3.7	1.4
33	Lube oil/additive Loading/Unloading	VOC	0.1	0.1
34	Packaging Plant Lube Oil Loading/Unloading Docks	VOC	0.1	0.1

\* Single emission bubble for  $NO_x$  is 53 tpy for SN-01, 25, and 26

 The permittee shall not exceed the emission rates set forth in the following table. [Regulation 18, §18.801 and A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]

SN	Description	Pollutant	lb/hr	tpy
01 Crude Charge Heater		РМ	0.3	1.0
02 Vacuum Tower Charge Heater		РМ	0.1	0.3
03	Asphalt Blow Still Charge Heater	РМ	0.1	0.3
04	Blow Still Incinerator Waste Heat Boiler	PM	8.5	37.0
05 Boiler #1 Retired				
06	Boiler #3	Retired		
07	Lube Precharge Heater	РМ	0.1	0.2

SN	Description	Pollutant	lb/hr	tpy
08	Hydrogen Plant Heater/Reactor	РМ	0.3	1.0
09	High Pressure Flare	РМ	0.1	0.1
10	Low Pressure Flare	PM	0.1	0.1
11	Naphtha Storage Tank	Retired		
12	Lube Stripper Charge Heater	PM	0.1	0.5
13	Boiler #2	Retired		
14	Diesel/Naphtha/Kerosene Loading Rack	2,2,4-Trimethylpentane Benzene Cumene Ethylbenzene Hexane Naphthalene Phenol Styrene Toluene Xylene	$\begin{array}{c} 0.14 \\ 0.40 \\ 0.02 \\ 0.05 \\ 3.84 \\ 0.01 \\ 0.01 \\ 0.01 \\ 0.49 \\ 0.15 \end{array}$	$\begin{array}{c} 0.04\\ 0.63\\ 0.03\\ 0.08\\ 2.33\\ 0.01\\ 0.01\\ 0.01\\ 0.47\\ 0.24\\ \end{array}$
17	Lube Oil Truck Loading Rack	Benzene Hexane	0.01	0.01
18	Lube Oil Truck Loading Rack	Benzene Hexane	0.01	0.01
21	Lube Oil Rail Car Loading Rack	Benzene Hexane Toluene	0.04 0.06 0.02	0.05 0.06 0.02
23	Fugitive Emissions	Benzene Cumene Ethylbenzene Hexane Toluene Xylene	0.49 0.01 0.04 0.72 0.18 0.07	2.15 0.03 0.18 3.12 0.79 0.27
24	Wastewater Emissions	Hexane	0.06	0.24
25	Cogeneration Unit	РМ	0.6	2.5
26	Boiler #4	PM	1.0	4.2
27	Onsite Storage Tanks (181 Tanks) Tanks Subject to 40 CFR Part 60, Subpart Ka: 113, 197, 287, 288, 289, 290, 296, 297, 298, 299, 312, 313, 314, 315, 206, 266, 291, 292, 329, and	2,2,4-Trimethylpentane Benzene Cumene Ethylbenzene Hexane Toluene	$\begin{array}{c} 0.01 \\ 0.05 \\ 0.01 \\ 0.01 \\ 0.12 \\ 0.02 \end{array}$	$\begin{array}{c} 0.02 \\ 0.19 \\ 0.01 \\ 0.02 \\ 0.49 \\ 0.09 \end{array}$

SN	Description	Pollutant	lb/hr	tpy
	Tank Subject to 40 CFR Part 60, Subpart Kb: Tank #319	Xylene	0.01	0.04
	Sandyland Storage Tanks	2,2,4-Trimethylpentane	0.01	0.04
	Tank #104, #110, #111	Benzene	0.03	0.13
20		Ethylbenzene	0.01	0.01
20	Tanks Subject to 40 CFR Part 60, Subpart Ka:	Hexane	0.26	1.20
	#111	Toluene	0.02	0.08
		Xylene	0.01	0.04
		2,2,4-Trimethylpentane	0.01	0.02
-		Benzene	0.01	0.05
20	Miller's Storage Tanks	Ethylbenzene	0.01	0.01
29	Tank #114, #115, and #116	Hexane	0.09	0.38
		Toluene	0.01	0.03
		Xylene	0.01	0.02
30	Lube Charge Heater	РМ	0.1	0.2
		2,2,4-Trimethylpentane	0.01	0.01
		Benzene	0.07	0.31
		Cumene	0.01	0.01
31	Distillate Lube Oil Truck loading Rack	Ethylbenzene	0.01	0.03
		Hexane	0.14	0.61
		Toluene	0.03	0.11
		Xylene	0.02	0.06
		Benzene	0.45	0.17
		Cumene	0.02	0.01
22	A anhalt/Distillate Tenk Car Leading Back	Ethylbenzene	0.04	0.02
52	Asphan/Distillate Fank Car Loading Rack	Hexane	0.90	0.34
		Toluene	0.16	0.06
		Xylene	0.09	0.04

3. Visible emissions may not exceed the limits specified in the following table of this permit as measured by EPA Reference Method 9. [A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]

SN	Limit	Regulatory Citation
01, 02, 03, 04, 07, 08, 12, 25, 26, 30	5%	§18.501
14, 15, 16, 17, 18, and 21	20%	§19.503

4. The permittee will not cause or permit the emission of air contaminants, including odors or water vapor and including an air contaminant whose emission is not otherwise prohibited by Regulation #18, if the emission of the air contaminant constitutes air

pollution within the meaning of A.C.A. §8-4-303. [§18.801 of Regulation 18, and A.C.A. §8-4-203 as referenced by §8-4-304 and §8-4-31]

5. The permittee will not conduct operations in such a manner as to unnecessarily cause air contaminants and other pollutants to become airborne. [§18.901 of Regulation 18, and A.C.A. §8-4-203 as referenced by §8-4-304 and §8-4-311]

# SN-01, SN-25, and SN-26 Conditions

6. The permittee shall not emit more than 53 tons of NO<sub>x</sub> at SN-01, SN-25, and SN-26 combined per consecutive 12 month period. NO<sub>x</sub> emissions shall be calculated by monitoring fuel inlet flow to all three sources and applying the following emission factors, unless the Department determines that testing results or other credible evidence indicate that other factors should be used: [Regulation 19 §19.705 and A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]

Source Number	Emission Factor*	Emission Factor Source
SN-01	$(92.4 \text{ lb NO}_x) / (10^6 \text{ ft}^3 \text{ natural gas})$	Stack Test
SN-25, Cogeneration Unit	$(184.6 \text{ lb NO}_x) / (10^6 \text{ ft}^3 \text{ natural gas})$	Vendor Data
SN-26	$(40 \text{ lb NO}_x) / (10^6 \text{ ft}^3 \text{ natural gas})$	Vendor Data

#### Table 6 – NO<sub>x</sub> Emission Factors for SN-01, SN-25, and SN-26

\*Emission factors are calculated based on heat value of 1020 Btu/ scfm for natural gas.

- 7. The permittee shall maintain monthly records of fuel usage and NO<sub>x</sub> emissions for sources SN-01, SN-25, and SN-26 which demonstrates compliance with Specific Condition #6. Records shall be updated by the fifteenth day of the month following the month to which the records pertain. A twelve month rolling total and each individual month's data shall be kept on site, and shall be made available to Department personnel upon request. [Regulation 19 §19.705 and A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]
- 8. The permittee shall install and maintain dedicated meters on the natural gas piping which feeds each emission unit specified in Specific Condition #6. [Regulation 19 §19.703 and A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]

#### **SN-04** Condition

9. The permittee shall maintain monthly records providing PM emissions from asphalt blowing at the Blow Still Incinerator Waste Heat Boiler. These emissions shall be calculated using the production rate multiplied by the PM emission factor of 0.81 lb/ton of asphalt. These records shall include ton per year PM calculations and thus demonstrate compliance with the limits in Specific Condition #1. Records shall be updated by the fifteenth day of the month following the month to which the records

pertain. A twelve month rolling total and each individual month's data shall be kept on site, and shall be made available to Department personnel upon request. [Regulation 19 §19.705 and A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]

#### **SN-09 and SN-10 Conditions**

10. The permittee shall report an upset condition any time that the flares are used. The permittee shall report the upset (by telephone, facsimile, or overnight delivery) to the Department by the end of the next business day after the occurrence or the discovery of the occurrence. The permittee shall submit to the Department, within five business days after the occurrence or the discovery of the occurrence, a full, written report of such occurrence, including a statement of all known causes and of the scheduling and nature of the actions to be taken to minimize or eliminate future occurrences, including, but not limited to, action to reduce the frequency of occurrence of such conditions, to minimize the amount by which said limits are exceeded, and to reduce the length of time for which said limits are exceeded. If the information is included in the initial report, it need not be submitted again. [Regulation 19 §19.601 and A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]

#### SN-14 through SN-21 Conditions

11. The permittee shall maintain monthly records providing VOC and HAP emissions from the loading of diesel, naphtha, kerosene, asphalt, and lube oil at the loading racks. These records shall include ton per year VOC and HAP calculations and thus demonstrate compliance with the VOC limits in Specific Condition #1 and the HAP limits in Specific Condition #2. A record of VOC and HAPs calculations for each one of the loading/unloading racks must be presented in a similar format to the spread sheet in the Attachment F. Records shall be updated by the fifteenth day of the month following the month to which the records pertain. A twelve month rolling total and each individual month's data shall be kept on site, and shall be made available to Department personnel upon request. [Regulation 19 §19.705 and A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]

#### SN-27 Onsite Storage Tanks

- The permittee shall not process more than 24,066 gallons per year of gasoline in gasoline tank located at the Onsite Storage Tanks (SN-27) in any consecutive 12 month period.
   [§19.705 of Regulation 19 and A.C.A. §8-4-203 as referenced by §8-4-304 and §8-4-311]
- 13. The permittee shall maintain monthly records which demonstrate compliance with Specific Condition # 12. These records shall include the gallons per month of gasoline received. These records shall also include the cumulative 12 month rolling totals of liquid received for each tank. Records shall be updated by the fifteenth day of the month following the month for which the records pertain. These records shall be kept on site,

and shall be made available to Department personnel upon request. [§19.705 of Regulation 19 and A.C.A. §8-4-203 as referenced by §8-4-304 and §8-4-311]

14. The permittee shall store lube oil or a liquid with maximum true vapor pressure less than 3.5 kPa include storage tanks #229, #230, #231, #232, #233, #332, #333, #334, #335, #336, #337, #338, #339, #340, #P041, #P042, #P043, #P044, #P045, #P046, #P047, #P048, #P049, #P050, #P051, and #P052. [§19.304 of Regulation 19 and 40 CFR Part 60, Subpart Kb, §60.110b(b)]

**NSPS Kb Conditions:** Tank #319 (distillate oil storage tank) is Subject to 40 CFR Part 60, Subpart Kb:

- 15. SN-27 (Tank #39) shall meet all applicable requirements of 40 CFR Part 60, Subpart Kb (Appendix D) Standards of Performance for Volatile Organic Liquid Storage Vessels (Including Petroleum Liquid Storage Vessels) for Which Construction, Reconstruction, or Modification Commenced After July 23, 1984. These requirements include, but are not limited to the following: [§19.304 of Regulation 19 and 40 CFR Part 60, Subpart Kb]
  - a. The owner or operator shall keep copies of all records required by § 60.116b, except for the record required by paragraph of §60.116b(b), for at least 2 years. The record required by paragraph (b) of §60.116b will be kept for the life of the source.
  - b. The owner or operator of each storage vessel as specified in §60.110b(a) shall keep readily accessible records showing the dimension of the storage vessel and an analysis showing the capacity of the storage vessel.
  - c. The owner or operator of each storage vessel either with a design capacity greater than or equal to 151 m<sup>3</sup> storing a liquid with a maximum true vapor pressure greater than or equal to 3.5 kPa or with a design capacity greater than or equal to 75 m<sup>3</sup> but less than 151 m<sup>3</sup> storing a liquid with a maximum true vapor pressure greater than or equal to 15.0 kPa shall maintain a record of the VOL stored, the period of storage, and the maximum true vapor pressure of that VOL during the respective storage period.
  - d. The owner or operator of each storage vessel either with a design capacity greater than or equal to 151 m<sup>3</sup> storing a liquid with a maximum true vapor pressure that is normally less than 5.2 kPa or with a design capacity greater than or equal to 75 m<sup>3</sup> but less than 151 m<sup>3</sup> storing a liquid with a maximum true vapor pressure that is normally less than 27.6 kPa shall notify the Administrator within 30 days when the maximum true vapor pressure of the liquid exceeds the respective maximum true vapor pressure values for each volume range.
  - e. Available data on the storage temperature may be used to determine the maximum true vapor pressure as determined below.

- (1) For vessels operated above or below ambient temperatures, the maximum true vapor pressure is calculated based upon the highest expected calendar-month average of the storage temperature. For vessels operated at ambient temperatures, the maximum true vapor pressure is calculated based upon the maximum local monthly average ambient temperature as reported by the National Weather Service.
- (2) For refined petroleum products the vapor pressure may be obtained by the following:
- i. Available data on the Reid vapor pressure and the maximum expected storage temperature based on the highest expected calendar-month average temperature of the stored product may be used to determine the maximum true vapor pressure from nomographs contained in API Bulletin 2517 (incorporated by reference—see §60.17), unless the Administrator specifically requests that the liquid be sampled, the actual storage temperature determined, and the Reid vapor pressure determined from the sample(s).
- ii. The true vapor pressure of each type of crude oil with a Reid vapor pressure less than 13.8 kPa or with physical properties that preclude determination by the recommended method is to be determined from available data and recorded if the estimated maximum true vapor pressure is greater than 3.5 kPa.

#### SN-31, SN-32, SN-33, and SN-34

16. The permittee shall maintain monthly records providing VOC and HAPs emissions from the Distillate Lube Oil Truck Loading Rack (SN-31), Asphalt Tank Car Loading Rack (SN-32), Loading/Unloading of Lube Oil and Additive Rack (SN-33), and Packaging Plant Lube Oil Loading/Unloading Docks (SN-34). These VOC and HAPs records shall include ton per year VOC and HAPs calculations and thus demonstrate compliance with the VOC and HAPs limits in Special Conditions #1 and #2. A record of VOC and HAPs calculations for each one of the loading/unloading racks must be presented in a similar format to the spread sheet in the Attachment E of the permit. Records shall be updated by the fifteenth day of the month following the month to which the records pertain. A twelve month rolling total and each individual month's data shall be kept on site, and shall be made available to Department personnel upon request. [Regulation 19 §19.705 and A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]

# **NSPS GG Conditions**

The cogeneration unit (SN-25) is an affected source of 40 CFR Part 60, Subpart GG (Appendix B) – Standards of Performance for Stationary Gas Turbines. [Regulation 19 §19.304 and 40 CFR Part 60, Subpart GG]

- a. The turbine shall not discharge any gases which contain nitrogen oxides in excess of 209 ppm by volume at 15 percent oxygen on a dry basis. [40 CFR Part 60, Subpart GG, §60.332 (a) (2)]
- b. The turbine is exempt from Specific Condition #17(a) when ice fog is deemed a traffic hazard by the owner or operator of the gas turbine. [40 CFR Part 60, Subpart GG, §60.332 (f)]
- c. The cogeneration unit shall only be fired with pipeline quality natural gas. [Regulation No. 19 §19.705, A.C.A. §8-4-203 as referenced by §8-4-304 and §8-4-311, and 40 CFR 70.6]
- d. No owner or operator shall burn in any stationary gas turbine any fuel which contains sulfur in excess of 0.8 percent by weight. [40 CFR Part 60, Subpart GG, §60.332 (b)]
- e. Analysis for fuel sulfur content of the natural gas shall be conducted using one of the approved ASTM reference methods for the measurement of sulfur in gaseous fuels, or an approved alternative method. The approved reference methods are: ASTM D1072-80; ASTM D3031-81; ASTM D3246-81; and ASTM D4084-82 as referenced in 40 CFR 60.335 (b) (2). [40 CFR Part 60, Subpart GG, §60.335 (d)]
- f. The fuel supply shall be initially sampled daily for a period of two weeks to establish that the pipeline quality natural gas fuel supply is low in sulfur content. [Regulation No. 19 §19.705, A.C.A. §8-4-203 as referenced by §8-4-304 and §8-4-311, and 40 CFR 70.6]
- g. After the monitoring required in Specific Condition #17 (e), sulfur monitoring shall be conducted twice monthly for six months. If this monitoring shows little variability in the fuel sulfur content, and indicates consistent compliance with 40 CFR 60.333, then sulfur monitoring shall be conducted once per quarter for six quarters. [40 CFR Part 60, Subpart GG, §60.335 (d)]
- h. If after the monitoring required in Specific Condition #17 (f), the sulfur content of the fuel shows little variability and, calculated as sulfur dioxide, represents consistent compliance with the sulfur dioxide emission limits specified under 40 CFR 60, Subpart GG, §60.333, sample analysis shall be conducted twice per annum. This monitoring shall be conducted during the first and third quarters of each calendar year. [40 CFR 60, Subpart GG, §60.333]
- i. Should any sulfur analysis as required in Specific Condition #17(f) or (g) indicate noncompliance with 40 CFR 60, Subpart GG, §60.333, the owner or operator shall notify ADEQ of such excess emissions and the custom schedule shall be re-examined. Sulfur monitoring shall be conducted weekly during the interim period

when this custom schedule is being re-examined. [Regulation No. 19 §19.303 and A.C.A. §8-4-203 as referenced by §8-4-304 and §8-4-311]

- j. If there is a change in fuel supply (supplier), the fuel shall be sampled daily for a period of two weeks to re-establish for the record that the fuel supply is low in sulfur content. If the fuel supply's low sulfur content is re-established, then the custom fuel monitoring schedule can be resumed. [Regulation No. 19 §19.705, 40 CFR Part 52, Subpart E, and A.C.A. §8-4-203 as referenced by §8-4-304 and §8-4-311]
- k. Records of sample analysis and fuel supply pertinent to this custom schedule shall be retained for a period of three years, and be available for inspection.
  [Regulation No. 19 §19.705, 40 CFR Part 52, Subpart E, and A.C.A. §8-4-203 as referenced by §8-4-304 and §8-4-311]
- Any one hour period during which the average water-to-fuel ratio, as measured by the continuous monitoring system, falls below the water-to-fuel ratio determined to demonstrate compliance with §60.332 by the performance test required in §60.8 or any period during which the fuel-bound nitrogen of the fuel is greater than the maximum nitrogen content allowed by the fuel-bound nitrogen allowance used during the performance test required in §60.8 shall be recorded. Each record entry shall include the average water-to-fuel ratio, average fuel consumption, ambient conditions gas turbine load, and nitrogen content of the fuel during the period of excess emissions, and the graphs or figures developed under §60.335(a). These records shall be retained for a period of five years, and be available for inspection. [40 CFR Part 60, Subpart GG, §60.334]
- m. Records shall be kept of any daily period during which the sulfur content of the fuel being fired in the gas turbine exceeds 0.8 percent. These records shall be retained for a period of five years, and be available for inspection. [40 CFR Part 60, Subpart GG, §60.334]
- n. Records shall be kept for each period which Specific Condition #17 (b) applies. For each period the ambient conditions existing during the period, the date and time the air pollution control system was deactivated, and the date and time the air pollution control system was reactivated shall be recorded. These records shall be retained for a period of five years, and be available for inspection. [40 CFR Part 60, Subpart GG, §60.334]
- o. The permittee shall perform an initial test of  $NO_x$  and  $SO_2$  to verify emissions. EPA Method 20 shall be used to determine the nitrogen oxides and oxygen concentrations. The span values shall be 300 ppm of nitrogen oxide and 21 percent oxygen.  $NO_x$  emissions shall be determined at each of the load conditions specified in §60.335(c) (2). The testing shall be coordinated in advance with the

Compliance Inspector Supervisor. This initial test was performed in March 2005. [40 CFR Part 60, Subpart GG, §60.335]

18. The permittee shall test NO<sub>x</sub> emissions once every five years to verify that the unit is operating within permitted limits. The permittee shall utilize the procedure outlined in 40 CFR Part 60, Subpart GG §60.335 and as previously conducted in the initial test required by Specific Condition #17(o). The testing shall be coordinated in advance with the Compliance Inspector Supervisor. [Regulation No. 19 §19.702, 40 CFR Part 52, Subpart E and 40 CFR Part 60, Subpart GG, §60.335]

# **NSPS Ka Conditions**

 The onsite storage tanks of SN-27 which are subject to provisions for 40 CFR Part 60, Subpart Ka (Appendix A) and their requirements are listed in the following table. [Regulation No. 19 §19.304 and 40 CFR Part 60, Subpart Ka]

Tank	Contents	NSPS Requirement
113, 197, 287, 288, 289, 290, 296, 297, 298, 299, 313, 314, 315	Lube Oils	Pursuant to 40 CFR 60.115a (a), maintain a record of the petroleum liquid stored, the
206, 266, 291, 292	Diesel	vener program of that liquid during the
329, 312	Naphtha	respective storage period
111	Crude	respective storage period.

# Table 7 – Storage Tanks Subject 40 CFR Part 60, Subpart Ka

# **NESHAP FF Conditions**

- 20. The facility is an affected source according to 40 CFR Part 61, Subpart FF (Appendix C)
    *National Emission Standard for Benzene Waste Operations*. [Regulation No. 19 §19.304 and 40 CFR Part 61, Subpart FF]
  - a. The owner and operator shall determine the total annual benzene quantity from facility waste by the procedures outlined in §61.355 (a). [40 CFR Part 61, Subpart FF, §61.355]
  - b. The facility shall comply with all record keeping requirements outlined in §61.356 (b). [40 CFR Part 61, Subpart FF, §61.355 (a)]
  - c. The facility shall submit reports to the Department by following the procedures of §61.357 (a) (1)-(4). In cases where the total annual benzene quantity is less than 1 Mg/yr [as determined in Specific Condition #20 (a)], reports will comply with §61.357 (b). In cases where the total annual benzene quantity is greater than 1 Mg/yr but less than 10 Mg/yr, reports will comply with §61.357 (c). And when

the total annual benzene quantity is greater than 10 Mg/yr, reports will comply with §61.357 (d). [40 CFR Part 61, Subpart FF, §61.357]

The facility is an affected source according to 40 CFR Part 61, Subpart FF – National Emission Standards for Hazardous Air Pollutants, Benzene Waste Operations

# Section V: INSIGNIFICANT ACTIVITIES

The Department deems the following types of activities or emissions as insignificant on the basis of size, emission rate, production rate, or activity in accordance with Group A of the Insignificant Activities list found in Regulation 18 and 19 Appendix A. Insignificant activity emission determinations rely upon the information submitted by the permittee in an application dated November 29, 2004, February 23, 2007, and October 16, 2007.

Description	Category
50 horsepower (0.125 MMBTU/hr) Low Pressure Boiler	Group A, No. 1
Recycle Water Evaporators (2)	Group A, No. 13
Packaging operation	Group A, No. 13

#### Section VI: GENERAL CONDITIONS

- 1. Any terms or conditions included in this permit that specify and reference Arkansas Pollution Control & Ecology Commission Regulation 18 or the Arkansas Water and Air Pollution Control Act (A.C.A. §8-4-101 et seq.) as the sole origin of and authority for the terms or conditions are not required under the Clean Air Act or any of its applicable requirements, and are not federally enforceable under the Clean Air Act. Arkansas Pollution Control & Ecology Commission Regulation 18 was adopted pursuant to the Arkansas Water and Air Pollution Control Act (A.C.A. §8-4-101 et seq.). Any terms or conditions included in this permit that specify and reference Arkansas Pollution Control & Ecology Commission Regulation 18 or the Arkansas Water and Air Pollution Control Act (A.C.A. §8-4-101 et seq.) as the origin of and authority for the terms or conditions are enforceable under this Arkansas statute.
- 2. This permit does not relieve the owner or operator of the equipment and/or the facility from compliance with all applicable provisions of the Arkansas Water and Air Pollution Control Act and the regulations promulgated under the Act. [A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]
- 3. The permittee shall notify the Department in writing within thirty (30) days after commencement of construction, completion of construction, first operation of equipment and/or facility, and first attainment of the equipment and/or facility target production rate. [Regulation 19, §19.704 and/or A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]
- 4. Construction or modification must commence within eighteen (18) months from the date of permit issuance. [Regulation 19, §19.410(B) and/or Regulation 18, §18.309(B) and A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]
- 5. The permittee must keep records for five years to enable the Department to determine compliance with the terms of this permit such as hours of operation, throughput, upset conditions, and continuous monitoring data. The Department may use the records, at the discretion of the Department, to determine compliance with the conditions of the permit. [Regulation 19, §19.705 and/or Regulation 18, §18.1004 and A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]
- 6. A responsible official must certify any reports required by any condition contained in this permit and submit any reports to the Department at the address below. [Regulation 19, §19.705 and/or Regulation 18, §18.1004 and A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]

Arkansas Department of Environmental Quality Air Division ATTN: Compliance Inspector Supervisor

> 5301 Northshore Drive North Little Rock, AR 72118-5317

- 7. The permittee shall test any equipment scheduled for testing, unless stated in the Specific Conditions of this permit or by any federally regulated requirements, within the following time frames: (1) newly constructed or modified equipment within sixty (60) days of achieving the maximum production rate, but no later than 180 days after initial start up of the permitted source or (2) existing equipment already operating according to the time frames set forth by the Department. The permittee must notify the Department of the scheduled date of compliance testing at least fifteen (15) days in advance of such test. The permittee must submit compliance test results to the Department within thirty (30) days after the completion of testing. [Regulation 19, §19.702 and/or Regulation 18, §18.1002 and A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]
- 8. The permittee shall provide: [Regulation 19, §19.702 and/or Regulation 18, §18.1002 and A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]
  - a. Sampling ports adequate for applicable test methods;
  - b. Safe sampling platforms;
  - c. Safe access to sampling platforms; and
  - d. Utilities for sampling and testing equipment
- 9. The permittee shall operate equipment, control apparatus and emission monitoring equipment within their design limitations. The permittee shall maintain in good condition at all times equipment, control apparatus and emission monitoring equipment. [Regulation 19, §19.303 and/or Regulation 18, §18.1104 and A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]
- 10. If the permittee exceeds an emission limit established by this permit, the permittee will be deemed in violation of said permit and will be subject to enforcement action. The Department may forego enforcement action for emissions exceeding any limits established by this permit provided the following requirements are met: [Regulation 19, §19.601 and/or Regulation 18, §18.1101 and A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]
  - a. The permittee demonstrates to the satisfaction of the Department that the emissions resulted from an equipment malfunction or upset and are not the result of negligence or improper maintenance, and the permittee took all reasonable measures to immediately minimize or eliminate the excess emissions.
  - b. The permittee reports the occurrence or upset or breakdown of equipment (by telephone, facsimile, or overnight delivery) to the Department by the end of the next business day after the occurrence or the discovery of the occurrence.
  - c. The permittee must submit to the Department, within five business days after the occurrence or the discovery of the occurrence, a full, written report of such occurrence, including a statement of all known causes and of the scheduling and

> nature of the actions to be taken to minimize or eliminate future occurrences, including, but not limited to, action to reduce the frequency of occurrence of such conditions, to minimize the amount by which said limits are exceeded, and to reduce the length of time for which said limits are exceeded. If the information is included in the initial report, the information need not be submitted again.

- 11. The permittee shall allow representatives of the Department upon the presentation of credentials: [A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]
  - a. To enter upon the permittee's premises, or other premises under the control of the permittee, where an air pollutant source is located or in which any records are required to be kept under the terms and conditions of this permit;
  - b. To have access to and copy any records required to be kept under the terms and conditions of this permit, or the Act;
  - c. To inspect any monitoring equipment or monitoring method required in this permit;
  - d. To sample any emission of pollutants; and
  - e. To perform an operation and maintenance inspection of the permitted source.
- 12. The Department issued this permit in reliance upon the statements and presentations made in the permit application. The Department has no responsibility for the adequacy or proper functioning of the equipment or control apparatus. [A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]
- 13. The Department may revoke or modify this permit when, in the judgment of the Department, such revocation or modification is necessary to comply with the applicable provisions of the Arkansas Water and Air Pollution Control Act and the regulations promulgated the Arkansas Water and Air Pollution Control Act. [Regulation 19, §19.410(A) and/or Regulation 18, §18.309(A) and A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]
- 14. This permit may be transferred. An applicant for a transfer must submit a written request for transfer of the permit on a form provided by the Department and submit the disclosure statement required by Arkansas Code Annotated §8-1-106 at least thirty (30) days in advance of the proposed transfer date. The permit will be automatically transferred to the new permittee unless the Department denies the request to transfer within thirty (30) days of the receipt of the disclosure statement. The Department may deny a transfer on the basis of the information revealed in the disclosure statement or other investigation or, deliberate falsification or omission of relevant information. [Regulation 19, §19.407(B) and/or Regulation 18, §18.307(B) and A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]
- 15. This permit shall be available for inspection on the premises where the control apparatus is located. [A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]

- 16. This permit authorizes only those pollutant emitting activities addressed herein. [A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]
- This permit supersedes and voids all previously issued air permits for this facility.
   [Regulation 18 and 19 and A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311]
- 18. The permittee must pay all permit fees in accordance with the procedures established in Regulation No. 9. [A.C.A §8-1-105(c)]
- 19. The permittee may request in writing and at least 15 days in advance of the deadline, an extension to any testing, compliance or other dates in this permit. No such extensions are authorized until the permittee receives written Department approval. The Department may grant such a request, at its discretion in the following circumstances:
  - a. Such an extension does not violate a federal requirement;
  - b. The permittee demonstrates the need for the extension; and
  - c. The permittee documents that all reasonable measures have been taken to meet the current deadline and documents reasons it cannot be met.

[Regulation 18, §18.314(A), Regulation 19, §19.416(A), A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311, and 40 CFR Part 52, Subpart E]

- 20. The permittee may request in writing and at least 30 days in advance, temporary emissions and/or testing that would otherwise exceed an emission rate, throughput requirement, or other limit in this permit. No such activities are authorized until the permittee receives written Department approval. Any such emissions shall be included in the facilities total emissions and reported as such. The Department may grant such a request, at its discretion under the following conditions:
  - a. Such a request does not violate a federal requirement;
  - b. Such a request is temporary in nature;
  - c. Such a request will not result in a condition of air pollution;
  - d. The request contains such information necessary for the Department to evaluate the request, including but not limited to, quantification of such emissions and the date/time such emission will occur;
  - e. Such a request will result in increased emissions less than five tons of any individual criteria pollutant, one ton of any single HAP and 2.5 tons of total HAPs; and
  - f. The permittee maintains records of the dates and results of such temporary emissions/testing.

[Regulation 18, §18.314(B), Regulation 19, §19.416(B), A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311, and 40 CFR Part 52, Subpart E]

- 21. The permittee may request in writing and at least 30 days in advance, an alternative to the specified monitoring in this permit. No such alternatives are authorized until the permittee receives written Department approval. The Department may grant such a request, at its discretion under the following conditions:
  - a. The request does not violate a federal requirement;
  - b. The request provides an equivalent or greater degree of actual monitoring to the current requirements; and
  - c. Any such request, if approved, is incorporated in the next permit modification application by the permittee.

[Regulation 18, §18.314(C), Regulation 19, §19.416(C), A.C.A. §8-4-203 as referenced by A.C.A. §8-4-304 and §8-4-311, and 40 CFR Part 52, Subpart E]

#### **APPENDIX A**

40 CFR Part 60, Subpart Ka

Standards of Performance for Storage Vessels for Petroleum Liquids for which Construction, Reconstruction, or Modification commenced after May 18, 1978, and prior to July 23, 1984
# e-CFR Data is current as of October 6, 2009

## **Title 40: Protection of Environment**

PART 60—STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES

Browse Previous | Browse Next

# Subpart Ka—Standards of Performance for Storage Vessels for Petroleum Liquids for Which Construction, Reconstruction, or Modification Commenced After May 18, 1978, and Prior to July 23, 1984

# § 60.110a Applicability and designation of affected facility.

(a) Affected facility. Except as provided in paragraph (b) of this section, the affected facility to which this subpart applies is each storage vessel with a storage capacity greater than 151,416 liters (40,000 gallons) that is used to store petroleum liquids for which construction is commenced after May 18, 1978.

(b) Each petroleum liquid storage vessel with a capacity of less than 1,589,873 liters (420,000 gallons) used for petroleum or condensate stored, processed, or treated prior to custody transfer is not an affected facility and, therefore, is exempt from the requirements of this subpart.

(c) Alternative means of compliance —(1) Option to comply with part 65. Owners or operators may choose to comply with 40 CFR part 65, subpart C, to satisfy the requirements of §§60.112a through 60.114a for storage vessels that are subject to this subpart that store petroleum liquids that, as stored, have a maximum true vapor pressure equal to or greater than 10.3 kPa (1.5 psia). Other provisions applying to owners or operators who choose to comply with 40 CFR part 65 are provided in 40 CFR 65.1.

(2) Part 60, subpart A. Owners or operators who choose to comply with 40 CFR part 65, subpart C, must also comply with §§60.1, 60.2, 60.5, 60.6, 60.7(a)(1) and (4), 60.14, 60.15, and 60.16 for those storage vessels. All sections and paragraphs of subpart A of this part that are not mentioned in this paragraph (c)(2) do not apply to owners or operators of storage vessels complying with 40 CFR part 65, subpart C, except that provisions required to be met prior to implementing 40 CFR part 65 still apply. Owners and operators who choose to comply with 40 CFR part 65, subpart C, must comply with 40 CFR part 65, subpart A.

[45 FR 23379, Apr. 4, 1980, as amended at 65 FR 78275, Dec. 14, 2000]

## § 60.111a Definitions.

In addition to the terms and their definitions listed in the Act and subpart A of this part the following definitions apply in this subpart:

(a) Storage vessel means each tank, reservoir, or container used for the storage of petroleum liquids, but does not include:

(1) Pressure vessels which are designed to operate in excess of 204.9 kPa (15 psig) without emissions to the atmosphere except under emergency conditions.

(2) Subsurface caverns or porous rock reservoirs, or

(3) Underground tanks if the total volume of petroleum liquids added to and taken from a tank annually does not exceed twice the volume of the tank.

(b) *Petroleum liquids* means petroleum, condensate, and any finished or intermediate products manufactured in a petroleum refinery but does not mean Nos. 2 through 6 fuel oils as specified in ASTM D396–78, 89, 90, 92, 96, or 98, gas turbine fuel oils Nos. 2–GT through 4–GT as specified in ASTM D2880–78 or 96, gas turbine fuel oils Nos. 2–GT through 4–GT as specified in ASTM D2880–78 or 96, or diesel fuel oils Nos. 2–D and 4–D as specified in ASTM D975–78, 96, or 98a. (These three methods are incorporated by reference—see §60.17.)

(c) *Petroleum refinery* means each facility engaged in producing gasoline, kerosene, distillate fuel oils, residual fuel oils, lubricants, or other products through distillation of petroleum or through redistillation, cracking, extracting, or reforming of unfinished petroleum derivatives.

(d) Petroleum means the crude oil removed from the earth and the oils derived from tar sands, shale, and coal.

(e) Condensate means hydrocarbon liquid separated from natural gas which condenses due to changes in the temperature or pressure, or both, and remains liquid at standard conditions.

(f) *True vapor pressure* means the equilibrium partial pressure exerted by a petroleum liquid such as determined in accordance with methods described in American Petroleum Institute Bulletin 2517, Evaporation Loss from External Floating-Roof Tanks, Second Edition, February 1980 (incorporated by reference—see §60.17).

(g) *Reid vapor pressure* is the absolute vapor pressure of volatile crude oil and nonviscous petroleum liquids, except liquified petroleum gases, as determined by ASTM D323–82 or 94 (incorporated by reference—see §60.17).

(h) Liquid-mounted seal means a foam or liquid-filled primary seal mounted in contact with the liquid between the tank wall and the floating roof continuously around the circumference of the tank.

(i) *Metallic shoe seal* includes but is not limited to a metal sheet held vertically against the tank wall by springs or weighted levers and is connected by braces to the floating roof. A flexible coated fabric (envelope) spans the annular space between the metal sheet and the floating roof.

(j) Vapor-mounted seal means a foam-filled primary seal mounted continuously around the circumference of the tank so there is an annular vapor space underneath the seal. The annular vapor space is bounded by the bottom of the primary seal, the tank wall, the liquid surface, and the floating roof.

(k) Custody transfer means the transfer of produced petroleum and/or condensate, after processing and/or treating in the producing operations, from storage tanks or automatic transfer facilities to pipelines or any other forms of transportation.

[45 FR 23379, Apr. 4, 1980, as amended at 48 FR 3737, Jan. 27, 1983; 52 FR 11429, Apr. 8, 1987; 65 FR 61756, Oct. 17, 2000]

### § 60.112a Standard for volatile organic compounds (VOC).

(a) The owner or operator of each storage vessel to which this subpart applies which contains a petroleum liquid which, as stored, has a true vapor pressure equal to or greater than 10.3 kPa (1.5 psia) but not greater than 76.6 kPa (11.1 psia) shall equip the storage vessel with one of the following:

(1) An external floating roof, consisting of a pontoon-type or double-deck-type cover that rests on the surface of the liquid contents and is equipped with a closure device between the tank wall and the roof edge. Except as provided in paragraph (a)(1)(ii)(D) of this section, the closure device is to consist of two seals, one above the other. The lower seal is referred to as the primary seal and the upper seal is referred to as the secondary seal. The roof is to be floating on the liquid at all times (i.e., off the roof leg supports) except during initial fill and when the tank is completely emptied and subsequently refilled. The process of emptying and refilling when the roof is resting on the leg supports shall be continuous and shall be accomplished as rapidly as possible.

(i) The primary seal is to be either a metallic shoe seal, a liquid-mounted seal, or a vapor-mounted seal. Each seal is to meet the following requirements:

(A) The accumulated area of gaps between the tank wall and the metallic shoe seal or the liquid-mounted seal shall not exceed 212  $\text{cm}^2$  per meter of tank diameter (10.0 in<sup>2</sup> per ft of tank diameter) and the width of any portion of any gap shall not exceed 3.81 cm (11/2in).

(B) The accumulated area of gaps between the tank wall and the vapor-mounted seal shall not exceed  $21.2 \text{ cm}^2$  per meter of tank diameter (1.0 in<sup>2</sup> per ft of tank diameter) and the width of any portion of any gap shall not exceed 1.27 cm (1/2in).

(C) One end of the metallic shoe is to extend into the stored liquid and the other end is to extend a minimum vertical distance of 61 cm (24 in) above the stored liquid surface.

(D) There are to be no holes, tears, or other openings in the shoe, seal fabric, or seal envelope.

(ii) The secondary seal is to meet the following requirements:

(A) The secondary seal is to be installed above the primary seal so that it completely covers the space between the roof edge and the tank wall except as provided in paragraph (a)(1)(ii)(B) of this section.

(B) The accumulated area of gaps between the tank wall and the secondary seal used in combination with a metallic shoe or liquid-mounted primary seal shall not exceed  $21.2 \text{ cm}^2$  per meter of tank diameter (1.0 in<sup>2</sup> per ft. of tank diameter) and the width of any portion of any gap shall not exceed 1.27 cm (1/2in.). There shall be no gaps between the tank wall and the secondary seal used in combination with a vapor-mounted primary seal.

(C) There are to be no holes, tears or other openings in the seal or seal fabric.

(D) The owner or operator is exempted from the requirements for secondary seals and the secondary seal gap criteria when performing gap measurements or inspections of the primary seal.

(iii) Each opening in the roof except for automatic bleeder vents and rim space vents is to provide a projection below the liquid surface. Each opening in the roof except for automatic bleeder vents, rim space vents and leg sleeves is to be equipped with a cover, seal or lid which is to be maintained in a closed position at all times (i.e., no visible gap) except when the device is in actual use or as described in pargraph (a)(1)(iv) of this section. Automatic bleeder vents are to be closed at all times when the roof is floating, except when the roof is being floated off or is being landed on the roof leg supports. Rim vents are to be set to open when the roof is being floated off the roof legs supports or at the manufacturer's recommended setting.

(iv) Each emergency roof drain is to be provided with a slotted membrane fabric cover that covers at least 90 percent of the area of the opening.

(2) A fixed roof with an internal floating type cover equipped with a continuous closure device between the tank wall and the cover edge. The cover is to be floating at all times, (i.e., off the leg supports) except during initial fill and when the tank is completely emptied and subsequently refilled. The process of emptying and refilling when the cover is resting on the leg supports shall be continuous and shall be accomplished as rapidly as possible. Each opening in the cover except for automatic bleeder vents and the rim space vents is to provide a projection below the liquid surface. Each opening in the cover, seal, or lid which is to be maintained in a closed position at all times (i.e., no visible gap) except when the device is in actual use. Automatic bleeder vents are to be closed at all times when the cover is floating except when the cover is being floated off or is being landed on the leg supports. Rim vents are to be set to open only when the cover is being floated off the leg supports or at the manufacturer's recommended setting.

(3) A vapor recovery system which collects all VOC vapors and gases discharged from the storage vessel, and a vapor return or disposal system which is designed to process such VOC vapors and gases so as to reduce their emission to the atmosphere by at least 95 percent by weight.

(4) A system equivalent to those described in paragraphs (a)(1), (a)(2), or (a)(3) of this section as provided in §60.114a.

(b) The owner or operator of each storage vessel to which this subpart applies which contains a petroleum liquid which, as stored, has a true vapor pressure greater than 76.6 kPa (11.1 psia), shall equip the storage vessel with a vapor recovery system which collects all VOC vapors and gases discharged from the storage vessel, and a vapor return or disposal system which is designed to process such VOC vapors and gases so as to reduce their emission to the atmosphere by at least 95 percent by weight.

[45 FR 23379, Apr. 4, 1980, as amended at 45 FR 83229, Dec. 18, 1980]

### § 60.113a Testing and procedures.

(a) Except as provided in §60.8(b) compliance with the standard prescribed in §60.112a shall be determined as follows or in accordance with an equivalent procedure as provided in §60.114a.

(1) The owner or operator of each storage vessel to which this subpart applies which has an external floating roof shall meet the following requirements:

(i) Determine the gap areas and maximum gap widths between the primary seal and the tank wall and between the secondary seal and the tank wall according to the following frequency:

(A) For primary seals, gap measurements shall be performed within 60 days of the initial fill with petroleum liquid and at least once every five years thereafter. All primary seal inspections or gap measurements which require the removal or dislodging of the secondary seal shall be accomplished as rapidly as possible and the secondary seal shall be replaced as soon as possible.

(B) For secondary seals, gap measurements shall be performed within 60 days of the initial fill with petroleum liquid and at least once every year thereafter.

(C) If any storage vessel is out of service for a period of one year or more, subsequent refilling with petroleum liquid shall be considered initial fill for the purposes of paragraphs (a)(1)(i)(A) and (a)(1)(i)(B) of this section.

(D) Keep records of each gap measurement at the plant for a period of at least 2 years following the date of measurement. Each record shall identify the vessel on which the measurement was performed and shall contain the date of the seal gap measurement, the raw data obtained in the measurement process required by paragraph (a)(1)(ii) of this section and the calculation required by paragraph (a)(1)(iii) of this section.

(E) If either the seal gap calculated in accord with paragraph (a)(1)(iii) of this section or the measured maximum seal gap exceeds the limitations specified by §60.112a of this subpart, a report shall be furnished to the Administrator within 60 days of the date of measurements. The report shall identify the vessel and list each reason why the vessel did not meet the specifications of §60.112a. The report shall also describe the actions necessary to bring the storage vessel into compliance with the specifications of §60.112a.

(ii) Determine gap widths in the primary and secondary seals individually by the following procedures:

(A) Measure seal gaps, if any, at one or more floating roof levels when the roof is floating off the roof leg supports.

(B) Measure seal gaps around the entire circumference of the tank in each place where a1/8&inch; diameter uniform probe passes freely (without forcing or binding against seal) between the seal and the tank wall and measure the circumferential distance of each such location.

(C) The total surface area of each gap described in paragraph (a)(1)(ii)(B) of this section shall be determined by using probes of various widths to accurately measure the actual distance from the tank wall to the seal and multiplying each such width by its respective circumferential distance.

(iii) Add the gap surface area of each gap location for the primary seal and the secondary seal individually. Divide the sum for each seal by the nominal diameter of the tank and compare each ratio to the appropriate ratio in the standard in §60.112a(a)(1)(i) and §60.112a(a)(1)(ii).

(iv) Provide the Administrator 30 days prior notice of the gap measurement to afford the Administrator the opportunity to have an observer present.

(2) The owner or operator of each storage vessel to which this subpart applies which has a vapor recovery and return or disposal system shall provide the following information to the Administrator on or before the date on which construction of the storage vessel commences:

(i) Emission data, if available, for a similar vapor recovery and return or disposal system used on the same type of storage vessel, which can be used to determine the efficiency of the system. A complete description of the emission measurement method used must be included.

(ii) The manufacturer's design specifications and estimated emission reduction capability of the system.

(iii) The operation and maintenance plan for the system.

(iv) Any other information which will be useful to the Administrator in evaluating the effectiveness of the system in reducing VOC emissions.

[45 FR 23379, Apr. 4, 1980, as amended at 52 FR 11429, Apr. 8, 1987]

§ 60.114a Alternative means of emission limitation.

(a) If, in the Administrator's judgment, an alternative means of emission limitation will achieve a reduction in emissions at least equivalent to the reduction in emissions achieved by any requirement in §60.112a, the Administrator will publish in theFederal Registera notice permitting the use of the alternative means for purposes of compliance with that requirement.

(b) Any notice under paragraph (a) of this section will be published only after notice and an opportunity for a hearing.

(c) Any person seeking permission under this section shall submit to the Administrator a written application including:

(1) An actual emissions test that uses a full-sized or scale-model storage vessel that accurately collects and measures all VOC emissions from a given control device and that accurately simulates wind and accounts for other emission variables such as temperature and barometric pressure.

(2) An engineering evaluation that the Administrator determines is an accurate method of determining equivalence.

(d) The Administrator may condition the permission on requirements that may be necessary to ensure operation and maintenance to achieve the same emissions reduction as specified in §60.112a.

(e) The primary vapor-mounted seal in the "Volume-Maximizing Seal" manufactured by R.F.I. Services Corporation is approved as equivalent to the vapor-mounted seal required by §60.112a(a)(1)(i) and must meet the gap criteria specified in §60.112a(a)(1)(i)(B). There shall be no gaps between the tank wall and any secondary seal used in conjunction with the primary seal in the "Volume-Maximizing Seal".

[52 FR 11429, Apr. 8, 1987]

## § 60.115a Monitoring of operations.

(a) Except as provided in paragraph (d) of this section, the owner or operator subject to this subpart shall maintain a record of the petroleum liquid stored, the period of storage, and the maximum true vapor pressure of that liquid during the respective storage period.

(b) Available data on the typical Reid vapor pressure and the maximum expected storage temperature of the stored product may be used to determine the maximum true vapor pressure from nomographs contained in API Bulletin 2517, unless the Administrator specifically requests that the liquid be sampled, the actual storage temperature determined, and the Reid vapor pressure determined from the sample(s).

(c) The true vapor pressure of each type of crude oil with a Reid vapor pressure less than 13.8 kPa (2.0 psia) or whose physical properties preclude determination by the recommended method is to be determined from available data and recorded if the estimated true vapor pressure is greater than 6.9 kPa (1.0 psia).

(d) The following are exempt from the requirements of this section:

(1) Each owner or operator of each storage vessel storing a petroleum liquid with a Reid vapor pressure of less than 6.9 kPa (1.0 psia) provided the maximum true vapor pressure does not exceed 6.9 kPa (1.0 psia).

(2) The owner or operator of each storage vessel equipped with a vapor recovery and return or disposal system in accordance with the requirements of §60.112a(a)(3) and (b), or a closed vent system and control device meeting the specifications of 40 CFR 65.42(b)(4), (b)(5), or (c).

[45 FR 23379, Apr. 4, 1980, as amended at 65 FR 78275, Dec. 14, 2000]

Browse Previous | Browse Next

For questions or comments regarding e-CFR editorial content, features, or design, email ecfr@nara.gov.

For questions concerning e-CFR programming and delivery issues, email webteam@gpo.gov.

Section 508 / Accessibility

# **APPENDIX B**

40 CFR Part 60, Subpart GG Stationary Gas Turbines

# e-CFR Data is current as of October 6, 2009

## **Title 40: Protection of Environment**

PART 60-STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES

Browse Previous | Browse Next

## Subpart GG—Standards of Performance for Stationary Gas Turbines

## § 60.330 Applicability and designation of affected facility.

(a) The provisions of this subpart are applicable to the following affected facilities: All stationary gas turbines with a heat input at peak load equal to or greater than 10.7 gigajoules (10 million Btu) per hour, based on the lower heating value of the fuel fired.

(b) Any facility under paragraph (a) of this section which commences construction, modification, or reconstruction after October 3, 1977, is subject to the requirements of this part except as provided in paragraphs (e) and (j) of §60.332.

[44 FR 52798, Sept. 10, 1979, as amended at 52 FR 42434, Nov. 5, 1987; 65 FR 61759, Oct. 17, 2000]

## § 60.331 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in subpart A of this part.

(a) Stationary gas turbine means any simple cycle gas turbine, regenerative cycle gas turbine or any gas turbine portion of a combined cycle steam/electric generating system that is not self propelled. It may, however, be mounted on a vehicle for portability.

(b) Simple cycle gas turbine means any stationary gas turbine which does not recover heat from the gas turbine exhaust gases to preheat the inlet combustion air to the gas turbine, or which does not recover heat from the gas turbine exhaust gases to heat water or generate steam.

(c) *Regenerative cycle gas turbine* means any stationary gas turbine which recovers heat from the gas turbine exhaust gases to preheat the inlet combustion air to the gas turbine.

(d) Combined cycle gas turbine means any stationary gas turbine which recovers heat from the gas turbine exhaust gases to heat water or generate steam.

(e) *Emergency gas turbine* means any stationary gas turbine which operates as a mechanical or electrical power source only when the primary power source for a facility has been rendered inoperable by an emergency situation.

(f) Ice fog means an atmospheric suspension of highly reflective ice crystals.

(g) ISO standard day conditions means 288 degrees Kelvin, 60 percent relative humidity and 101.3 kilopascals pressure.

(h) *Efficiency* means the gas turbine manufacturer's rated heat rate at peak load in terms of heat input per unit of power output based on the lower heating value of the fuel.

(i) Peak load means 100 percent of the manufacturer's design capacity of the gas turbine at ISO standard day conditions.

(j) Base load means the load level at which a gas turbine is normally operated.

(k) Fire-fighting turbine means any stationary gas turbine that is used solely to pump water for extinguishing fires.

(I) *Turbines employed in oil/gas production or oil/gas transportation* means any stationary gas turbine used to provide power to extract crude oil/natural gas from the earth or to move crude oil/natural gas, or products refined from these substances through pipelines.

(m) A Metropolitan Statistical Area or MSA as defined by the Department of Commerce.

(n) Offshore platform gas turbines means any stationary gas turbine located on a platform in an ocean.

(o) Garrison facility means any permanent military installation.

(p) Gas turbine model means a group of gas turbines having the same nominal air flow, combuster inlet pressure, combuster inlet temperature, firing temperature, turbine inlet temperature and turbine inlet pressure.

(q) *Electric utility stationary gas turbine* means any stationary gas turbine constructed for the purpose of supplying more than one-third of its potential electric output capacity to any utility power distribution system for sale.

(r) *Emergency fuel* is a fuel fired by a gas turbine only during circumstances, such as natural gas supply curtailment or breakdown of delivery system, that make it impossible to fire natural gas in the gas turbine.

(s) Unit operating hour means a clock hour during which any fuel is combusted in the affected unit. If the unit combusts fuel for the entire clock hour, it is considered to be a full unit operating hour. If the unit combusts fuel for only part of the clock hour, it is considered to be a partial unit operating hour.

(t) Excess emissions means a specified averaging period over which either:

(1) The NO<sub>x</sub>emissions are higher than the applicable emission limit in §60.332;

(2) The total sulfur content of the fuel being combusted in the affected facility exceeds the limit specified in §60.333; or

(3) The recorded value of a particular monitored parameter is outside the acceptable range specified in the parameter monitoring plan for the affected unit.

(u) *Natural gas* means a naturally occurring fluid mixture of hydrocarbons (e.g., methane, ethane, or propane) produced in geological formations beneath the Earth's surface that maintains a gaseous state at standard atmospheric temperature and pressure under ordinary conditions. Natural gas contains 20.0 grains or less of total sulfur per 100 standard cubic feet. Equivalents of this in other units are as follows: 0.068 weight percent total sulfur, 680 parts per million by weight (ppmw) total sulfur, and 338 parts per million by volume (ppmv) at 20 degrees Celsius total sulfur. Additionally, natural gas must either be composed of at least 70 percent methane by volume or have a gross calorific value between 950 and 1100 British thermal units (Btu) per standard cubic foot. Natural gas does not include the following gaseous fuels: landfill gas, digester gas, refinery gas, sour gas, blast furnace gas, coal-derived gas, producer gas, coke oven gas, or any gaseous fuel produced in a process which might result in highly variable sulfur content or heating value.

(v) Duct burner means a device that combusts fuel and that is placed in the exhaust duct from another source, such as a stationary gas turbine, internal combustion engine, kiln, etc., to allow the firing of additional fuel to heat the exhaust gases before the exhaust gases enter a heat recovery steam generating unit.

(w) Lean premix stationary combustion turbine means any stationary combustion turbine where the air and fuel are thoroughly mixed to form a lean mixture for combustion in the combustor. Mixing may occur before or in the combustion chamber. A unit which is capable of operating in both lean premix and diffusion flame modes is considered a lean premix stationary combustion turbine when it is in the lean premix mode, and it is considered a diffusion flame stationary combustion turbine when it is in the diffusion flame mode.

(x) Diffusion flame stationary combustion turbine means any stationary combustion turbine where fuel and air are injected at the combustor and are mixed only by diffusion prior to ignition. A unit which is capable of operating in both lean premix and diffusion flame modes is considered a lean premix stationary combustion turbine when it is in the lean premix mode, and it is considered a diffusion flame stationary combustion turbine when it is in the diffusion flame mode.

(y) Unit operating day means a 24-hour period between 12:00 midnight and the following midnight during which any fuel is combusted at any time in the unit. It is not necessary for fuel to be combusted continuously for the entire 24-hour period.

[44 FR 52798, Sept. 10, 1979, as amended at 47 FR 3770, Jan. 27, 1982; 65 FR 61759, Oct. 17, 2000; 69 FR 41359, July 8, 2004]

### § 60.332 Standard for nitrogen oxides.

(a) On and after the date on which the performance test required by 60.8 is completed, every owner or operator subject to the provisions of this subpart as specified in paragraphs (b), (c), and (d) of this section shall comply with one of the following, except as provided in paragraphs (e), (f), (g), (h), (i), (j), (k), and (l) of this section.

(1) No owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any stationary gas turbine, any gases which contain nitrogen oxides in excess of:

$$STD = 0.0075 \frac{(14.4)}{Y} + F$$

where:

STD = allowable ISO corrected (if required as given in 60.335(b)(1)) NO<sub>x</sub>emission concentration (percent by volume at 15 percent oxygen and on a dry basis),

Y = manufacturer's rated heat rate at manufacturer's rated load (kilojoules per watt hour) or, actual measured heat rate based on lower heating value of fuel as measured at actual peak load for the facility. The value of Y shall not exceed 14.4 kilojoules per watt hour, and

 $F = NO_x$  emission allowance for fuel-bound nitrogen as defined in paragraph (a)(4) of this section.

(2) No owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any stationary gas turbine, any gases which contain nitrogen oxides in excess of:

$$STD = 0.0150 \frac{(14.4)}{Y} + F$$

where:

STD = allowable ISO corrected (if required as given in 60.335(b)(1)) NO<sub>x</sub>emission concentration (percent by volume at 15 percent oxygen and on a dry basis),

Y = manufacturer's rated heat rate at manufacturer's rated peak load (kilojoules per watt hour), or actual measured heat rate based on lower heating value of fuel as measured at actual peak load for the facility. The value of Y shall not exceed 14.4 kilojoules per watt hour, and

 $F = NO_x$  emission allowance for fuel-bound nitrogen as defined in paragraph (a)(4) of this section.

(3) The use of F in paragraphs (a)(1) and (2) of this section is optional. That is, the owner or operator may choose to apply a NO<sub>x</sub>allowance for fuel-bound nitrogen and determine the appropriate F-value in accordance with paragraph (a)(4) of this section or may accept an F-value of zero.

(4) If the owner or operator elects to apply a NO<sub>x</sub>emission allowance for fuel-bound nitrogen, F shall be defined according to the nitrogen content of the fuel during the most recent performance test required under §60.8 as follows:

Fuel-bound nitrogen (percent by weight)	F (NO <sub>x</sub> percent by volume)
$N \leq .015$	0

$0.015 < N \le 0.1$	0.04 (N)
$0.1 < N \le 0.25$	0.004+0.0067(N-0.1)
N > 0.25	0.005

Where:

N = the nitrogen content of the fuel (percent by weight).

or:

Manufacturers may develop and submit to EPA custom fuel-bound nitrogen allowances for each gas turbine model they manufacture. These fuel-bound nitrogen allowances shall be substantiated with data and must be approved for use by the Administrator before the initial performance test required by §60.8. Notices of approval of custom fuel-bound nitrogen allowances will be published in theFederal Register.

(b) Electric utility stationary gas turbines with a heat input at peak load greater than 107.2 gigajoules per hour (100 million Btu/hour) based on the lower heating value of the fuel fired shall comply with the provisions of paragraph (a)(1) of this section.

(c) Stationary gas turbines with a heat input at peak load equal to or greater than 10.7 gigajoules per hour (10 million Btu/hour) but less than or equal to 107.2 gigajoules per hour (100 million Btu/hour) based on the lower heating value of the fuel fired, shall comply with the provisions of paragraph (a)(2) of this section.

(d) Stationary gas turbines with a manufacturer's rated base load at ISO conditions of 30 megawatts or less except as provided in §60.332(b) shall comply with paragraph (a)(2) of this section.

(e) Stationary gas turbines with a heat input at peak load equal to or greater than 10.7 gigajoules per hour (10 million Btu/hour) but less than or equal to 107.2 gigajoules per hour (100 million Btu/hour) based on the lower heating value of the fuel fired and that have commenced construction prior to October 3, 1982 are exempt from paragraph (a) of this section.

(f) Stationary gas turbines using water or steam injection for control of NO<sub>x</sub>emissions are exempt from paragraph (a) when ice fog is deemed a traffic hazard by the owner or operator of the gas turbine.

(g) Emergency gas turbines, military gas turbines for use in other than a garrison facility, military gas turbines installed for use as military training facilities, and fire fighting gas turbines are exempt from paragraph (a) of this section.

(h) Stationary gas turbines engaged by manufacturers in research and development of equipment for both gas turbine emission control techniques and gas turbine efficiency improvements are exempt from paragraph (a) on a case-by-case basis as determined by the Administrator.

(i) Exemptions from the requirements of paragraph (a) of this section will be granted on a case-by-case basis as determined by the Administrator in specific geographical areas where mandatory water restrictions are required by governmental agencies because of drought conditions. These exemptions will be allowed only while the mandatory water restrictions are in effect.

(j) Stationary gas turbines with a heat input at peak load greater than 107.2 gigajoules per hour that commenced construction, modification, or reconstruction between the dates of October 3, 1977, and January 27, 1982, and were required in the September 10, 1979, Federal Register(44 FR 52792) to comply with paragraph (a)(1) of this section, except electric utility stationary gas turbines, are exempt from paragraph (a) of this section.

(k) Stationary gas turbines with a heat input greater than or equal to 10.7 gigajoules per hour (10 million Btu/hour) when fired with natural gas are exempt from paragraph (a)(2) of this section when being fired with an emergency fuel.

(I) Regenerative cycle gas turbines with a heat input less than or equal to 107.2 gigajoules per hour (100 million Btu/hour) are exempt from paragraph (a) of this section.

[44 FR 52798, Sept. 10, 1979, as amended at 47 FR 3770, Jan. 27, 1982; 65 FR 61759, Oct. 17, 2000; 69 FR 41359, July 8, 2004]

### § 60.333 Standard for sulfur dioxide.

On and after the date on which the performance test required to be conducted by §60.8 is completed, every owner or operator subject to the provision of this subpart shall comply with one or the other of the following conditions:

(a) No owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any stationary gas turbine any gases which contain sulfur dioxide in excess of 0.015 percent by volume at 15 percent oxygen and on a dry basis.

(b) No owner or operator subject to the provisions of this subpart shall burn in any stationary gas turbine any fuel which contains total sulfur in excess of 0.8 percent by weight (8000 ppmw).

[44 FR 52798, Sept. 10, 1979, as amended at 69 FR 41360, July 8, 2004]

### § 60.334 Monitoring of operations.

(a) Except as provided in paragraph (b) of this section, the owner or operator of any stationary gas turbine subject to the provisions of this subpart and using water or steam injection to control NO<sub>x</sub>emissions shall install, calibrate, maintain and operate a continuous monitoring system to monitor and record the fuel consumption and the ratio of water or steam to fuel being fired in the turbine.

(b) The owner or operator of any stationary gas turbine that commenced construction, reconstruction or modification after October 3, 1977, but before July 8, 2004, and which uses water or steam injection to control NO<sub>x</sub>emissions may, as an alternative to operating the continuous monitoring system described in paragraph (a) of this section, install, certify, maintain, operate, and quality-assure a continuous emission monitoring system (CEMS) consisting of NO<sub>x</sub>and  $O_2$ monitors. As an alternative, a CO<sub>2</sub>monitor may be used to adjust the measured NO<sub>x</sub>concentrations to 15 percent  $O_2$ by either converting the CO<sub>2</sub>hourly averages to equivalent  $O_2$ concentrations using Equation F–14a or F–14b in appendix F to part 75 of this chapter and making the adjustments to 15 percent  $O_2$ , or by using the CO<sub>2</sub>readings directly to make the adjustments, as described in Method 20. If the option to use a CEMS is chosen, the CEMS shall be installed, certified, maintained and operated as follows:

(1) Each CEMS must be installed and certified according to PS 2 and 3 (for diluent) of 40 CFR part 60, appendix B, except the 7-day calibration drift is based on unit operating days, not calendar days. Appendix F, Procedure 1 is not required. The relative accuracy test audit (RATA) of the NO<sub>x</sub> and diluent monitors may be performed individually or on a combined basis, *i.e.*, the relative accuracy tests of the CEMS may be performed either:

(i) On a ppm basis (for NO<sub>x</sub>) and a percent O<sub>2</sub>basis for oxygen; or

(ii) On a ppm at 15 percent O<sub>2</sub>basis; or

(iii) On a ppm basis (for  $NO_x$ ) and a percent  $CO_2$  basis (for a  $CO_2$  monitor that uses the procedures in Method 20 to correct the  $NO_x$  data to 15 percent  $O_2$ ).

(2) As specified in §60.13(e)(2), during each full unit operating hour, each monitor must complete a minimum of one cycle of operation (sampling, analyzing, and data recording) for each 15-minute quadrant of the hour, to validate the hour. For partial unit operating hours, at least one valid data point must be obtained for each quadrant of the hour in which the unit operates. For unit operating hours in which required quality assurance and maintenance activities are performed on the CEMS, a minimum of two valid data points (one in each of two quadrants) are required to validate the hour.

(3) For purposes of identifying excess emissions, CEMS data must be reduced to hourly averages as specified in §60.13(h).

(i) For each unit operating hour in which a valid hourly average, as described in paragraph (b)(2) of this section, is obtained for both NO<sub>x</sub>and diluent, the data acquisition and handling system must calculate and record the hourly NO<sub>x</sub>emissions in the units of the applicable NO<sub>x</sub>emission standard under §60.332(a), *i.e.*, percent NO<sub>x</sub>by volume, dry basis, corrected to 15 percent O<sub>2</sub>and International Organization for Standardization (ISO) standard conditions (if required as given in §60.335(b)(1)). For any hour in which the hourly average O<sub>2</sub>concentration exceeds 19.0 percent O<sub>2</sub>, a diluent cap value of 19.0 percent O<sub>2</sub>may be used in the emission calculations.

(ii) A worst case ISO correction factor may be calculated and applied using historical ambient data. For the purpose of this calculation, substitute the maximum humidity of ambient air (Ho), minimum ambient temperature ( $T_a$ ), and minimum combustor inlet absolute pressure ( $P_o$ ) into the ISO correction equation.

(iii) If the owner or operator has installed a NO<sub>x</sub>CEMS to meet the requirements of part 75 of this chapter, and is continuing to meet the ongoing requirements of part 75 of this chapter, the CEMS may be used to meet the requirements of this section, except that the missing data substitution methodology provided for at 40 CFR part 75, subpart D, is not required for purposes of identifying excess emissions. Instead, periods of missing CEMS data are to be reported as monitor downtime in the excess emissions and monitoring performance report required in §60.7(c).

(c) For any turbine that commenced construction, reconstruction or modification after October 3, 1977, but before July 8, 2004, and which does not use steam or water injection to control NO<sub>x</sub>emissions, the owner or operator may, but is not required to, for purposes of determining excess emissions, use a CEMS that meets the requirements of paragraph (b) of this section. Also, if the owner or operator has previously submitted and received EPA, State, or local permitting authority approval of a procedure for monitoring compliance with the applicable NO<sub>x</sub>emission limit under §60.332, that approved procedure may continue to be used.

(d) The owner or operator of any new turbine constructed after July 8, 2004, and which uses water or steam injection to control NO<sub>x</sub>emissions may elect to use either the requirements in paragraph (a) of this section for continuous water or steam to fuel ratio monitoring or may use a NO<sub>x</sub>CEMS installed, certified, operated, maintained, and quality-assured as described in paragraph (b) of this section.

(e) The owner or operator of any new turbine that commences construction after July 8, 2004, and which does not use water or steam injection to control NO<sub>x</sub>emissions, may, but is not required to, elect to use a NO<sub>x</sub>CEMS installed, certified, operated, maintained, and quality-assured as described in paragraph (b) of this section. Other acceptable monitoring approaches include periodic testing approved by EPA or the State or local permitting authority or continuous parameter monitoring as described in paragraph (f) of this section.

(f) The owner or operator of a new turbine that commences construction after July 8, 2004, which does not use water or steam injection to control NO<sub>x</sub>emissions may, but is not required to, perform continuous parameter monitoring as follows:

(1) For a diffusion flame turbine without add-on selective catalytic reduction controls (SCR), the owner or operator shall define at least four parameters indicative of the unit's NO<sub>x</sub> formation characteristics and shall monitor these parameters continuously.

(2) For any lean premix stationary combustion turbine, the owner or operator shall continuously monitor the appropriate parameters to determine whether the unit is operating in low-NO<sub>x</sub>mode.

(3) For any turbine that uses SCR to reduce NO<sub>x</sub>emissions, the owner or operator shall continuously monitor appropriate parameters to verify the proper operation of the emission controls.

(4) For affected units that are also regulated under part 75 of this chapter, if the owner or operator elects to monitor NO<sub>x</sub>emission rate using the methodology in appendix E to part 75 of this chapter, or the low mass emissions methodology in 575.19 of this chapter, the requirements of this paragraph (f) may be met by performing the parametric monitoring described in section 2.3 of appendix E or in 575.19(c)(1)(iv)(H) of this chapter.

(g) The steam or water to fuel ratio or other parameters that are continuously monitored as described in paragraphs (a), (d) or (f) of this section shall be monitored during the performance test required under §60.8, to establish acceptable values and ranges. The owner or operator may supplement the performance test data with engineering analyses, design specifications, manufacturer's recommendations and other relevant information to define the acceptable parametric ranges more precisely. The owner or operator shall develop and keep on-site a parameter monitoring plan which explains the procedures used to document proper operation of the NO<sub>x</sub>emission controls. The plan shall include the parameter(s) monitored and the acceptable range(s) of the parameter(s) as well as the basis for designating the parameter(s) and acceptable range(s). Any supplemental data such as engineering analyses, design specifications, manufacturer's recommendations and other relevant information shall be included in the monitoring plan. For affected units that are also subject to part 75 of this chapter and that use the low mass emissions methodology in §75.19 of this chapter or the NO<sub>x</sub>emission measurement methodology in appendix E to part 75, the owner or operator may meet the requirements of this paragraph by developing and keeping on-site (or at a central location for unmanned facilities) a quality-assurance plan, as described in §75.19 (e)(5) or in section 2.3 of appendix E and section 1.3.6 of appendix B to part 75 of this chapter.

(h) The owner or operator of any stationary gas turbine subject to the provisions of this subpart:

(1) Shall monitor the total sulfur content of the fuel being fired in the turbine, except as provided in paragraph (h)(3) of this section. The sulfur content of the fuel must be determined using total sulfur methods described in §60.335(b)(10). Alternatively, if the total sulfur content of the gaseous fuel during the most recent performance test was less than 0.4 weight percent (4000 ppmw), ASTM D4084–82, 94, D5504–01, D6228–98, or Gas Processors Association Standard 2377–86 (all of which are incorporated by reference-see §60.17), which measure the major sulfur compounds may be used; and

(2) Shall monitor the nitrogen content of the fuel combusted in the turbine, if the owner or operator claims an allowance for fuel bound nitrogen (*i.e.*, if an F-value greater than zero is being or will be used by the owner or operator to calculate STD in §60.332). The nitrogen content of the fuel shall be determined using methods described in §60.335(b)(9) or an approved alternative.

(3) Notwithstanding the provisions of paragraph (h)(1) of this section, the owner or operator may elect not to monitor the total sulfur content of the gaseous fuel combusted in the turbine, if the gaseous fuel is demonstrated to meet the definition of natural gas in §60.331(u), regardless of whether an existing custom schedule approved by the administrator for subpart GG requires such monitoring. The owner or operator shall use one of the following sources of information to make the required demonstration:

(i) The gas quality characteristics in a current, valid purchase contract, tariff sheet or transportation contract for the gaseous fuel, specifying that the maximum total sulfur content of the fuel is 20.0 grains/100 scf or less; or

(ii) Representative fuel sampling data which show that the sulfur content of the gaseous fuel does not exceed 20 grains/100 scf. At a minimum, the amount of fuel sampling data specified in section 2.3.1.4 or 2.3.2.4 of appendix D to part 75 of this chapter is required.

(4) For any turbine that commenced construction, reconstruction or modification after October 3, 1977, but before July 8, 2004, and for which a custom fuel monitoring schedule has previously been approved, the owner or operator may, without submitting a special petition to the Administrator, continue monitoring on this schedule.

(i) The frequency of determining the sulfur and nitrogen content of the fuel shall be as follows:

(1) *Fuel oil.* For fuel oil, use one of the total sulfur sampling options and the associated sampling frequency described in sections 2.2.3, 2.2.4.1, 2.2.4.2, and 2.2.4.3 of appendix D to part 75 of this chapter (*i.e.*, flow proportional sampling, daily sampling, sampling from the unit's storage tank after each addition of fuel to the tank, or sampling each delivery prior to combining it with fuel oil already in the intended storage tank). If an emission allowance is being claimed for fuel-bound nitrogen, the nitrogen content of the oil shall be determined and recorded once per unit operating day.

(2) Gaseous fuel. Any applicable nitrogen content value of the gaseous fuel shall be determined and recorded once per unit operating day. For owners and operators that elect not to demonstrate sulfur content using options in paragraph (h)(3) of this section, and for which the fuel is supplied without intermediate bulk storage, the sulfur content value of the gaseous fuel shall be determined and recorded once per unit operating day.

(3) Custom schedules. Notwithstanding the requirements of paragraph (i)(2) of this section, operators or fuel vendors may develop custom schedules for determination of the total sulfur content of gaseous fuels, based on the design and operation of the affected facility and the characteristics of the fuel supply. Except as provided in paragraphs (i)(3)(i) and (i)(3)(ii) of this section, custom schedules shall be substantiated with data and shall be approved by the Administrator before they can be used to comply with the standard in §60.333.

(i) The two custom sulfur monitoring schedules set forth in paragraphs (i)(3)(i)(A) through (D) and in paragraph (i)(3)(ii) of this section are acceptable, without prior Administrative approval:

(A) The owner or operator shall obtain daily total sulfur content measurements for 30 consecutive unit operating days, using the applicable methods specified in this subpart. Based on the results of the 30 daily samples, the required frequency for subsequent monitoring of the fuel's total sulfur content shall be as specified in paragraph (i)(3)(i)(B), (C), or (D) of this section, as applicable.

(B) If none of the 30 daily measurements of the fuel's total sulfur content exceeds 0.4 weight percent (4000 ppmw), subsequent sulfur content monitoring may be performed at 12 month intervals. If any of the samples taken at 12-month intervals has a total sulfur content between 0.4 and 0.8 weight percent (4000 and 8000 ppmw), follow the procedures in paragraph (i)(3)(i)(C) of this section. If any measurement exceeds 0.8 weight percent (8000 ppmw), follow the procedures in paragraph (i)(3)(i)(D) of this section.

(C) If at least one of the 30 daily measurements of the fuel's total sulfur content is between 0.4 and 0.8 weight percent (4000 and 8000 ppmw), but none exceeds 0.8 weight percent (8000 ppmw), then:

(1) Collect and analyze a sample every 30 days for three months. If any sulfur content measurement exceeds 0.8 weight percent (8000 ppmw), follow the procedures in paragraph (i)(3)(i)(D) of this section. Otherwise, follow the procedures in paragraph (i)(3)(i)(C)(2) of this section.

(2) Begin monitoring at 6-month intervals for 12 months. If any sulfur content measurement exceeds 0.8 weight percent (8000 ppmw), follow the procedures in paragraph (i)(3)(i)(D) of this section. Otherwise, follow the procedures in paragraph (i)(3)(i)(C)(3) of this section.

(3) Begin monitoring at 12-month intervals. If any sulfur content measurement exceeds 0.8 weight percent (8000 ppmw), follow the procedures in paragraph (i)(3)(i)(D) of this section. Otherwise, continue to monitor at this frequency.

(D) If a sulfur content measurement exceeds 0.8 weight percent (8000 ppmw), immediately begin daily monitoring according to paragraph (i)(3)(i)(A) of this section. Daily monitoring shall continue until 30 consecutive daily samples, each having a sulfur content no greater than 0.8 weight percent (8000 ppmw), are obtained. At that point, the applicable procedures of paragraph (i)(3)(i)(B) or (C) of this section shall be followed.

(ii) The owner or operator may use the data collected from the 720-hour sulfur sampling demonstration described in section 2.3.6 of appendix D to part 75 of this chapter to determine a custom sulfur sampling schedule, as follows:

(A) If the maximum fuel sulfur content obtained from the 720 hourly samples does not exceed 20 grains/100 scf (*i.e.*, the maximum total sulfur content of natural gas as defined in §60.331(u)), no additional monitoring of the sulfur content of the gas is required, for the purposes of this subpart.

(B) If the maximum fuel sulfur content obtained from any of the 720 hourly samples exceeds 20 grains/100 scf, but none of the sulfur content values (when converted to weight percent sulfur) exceeds 0.4 weight percent (4000 ppmw), then the minimum required sampling frequency shall be one sample at 12 month intervals.

(C) If any sample result exceeds 0.4 weight percent sulfur (4000 ppmw), but none exceeds 0.8 weight percent sulfur (8000 ppmw), follow the provisions of paragraph (i)(3)(i)(C) of this section.

(D) If the sulfur content of any of the 720 hourly samples exceeds 0.8 weight percent (8000 ppmw), follow the provisions of paragraph (i)(3)(i)(D) of this section.

(j) For each affected unit that elects to continuously monitor parameters or emissions, or to periodically determine the fuel sulfur content or fuel nitrogen content under this subpart, the owner or operator shall submit reports of excess emissions and monitor downtime, in accordance with §60.7(c). Excess emissions shall be reported for all periods of unit operation, including startup, shutdown and malfunction. For the purpose of reports required under §60.7(c), periods of excess emissions and monitor downtime that shall be reported are defined as follows:

(1) Nitrogen oxides.

(i) For turbines using water or steam to fuel ratio monitoring:

(A) An excess emission shall be any unit operating hour for which the average steam or water to fuel ratio, as measured by the continuous monitoring system, falls below the acceptable steam or water to fuel ratio needed to demonstrate compliance with §60.332, as established during the performance test required in §60.8. Any unit operating hour in which no water or steam is injected into the turbine shall also be considered an excess emission.

(B) A period of monitor downtime shall be any unit operating hour in which water or steam is injected into the turbine, but the essential parametric data needed to determine the steam or water to fuel ratio are unavailable or invalid.

(C) Each report shall include the average steam or water to fuel ratio, average fuel consumption, ambient conditions (temperature, pressure, and humidity), gas turbine load, and (if applicable) the nitrogen content of the fuel during each excess emission. You do not have to report ambient conditions if you opt to use the worst case ISO correction factor as specified in §60.334(b)(3)(ii), or if you are not using the ISO correction equation under the provisions of §60.335(b)(1).

(ii) If the owner or operator elects to take an emission allowance for fuel bound nitrogen, then excess emissions and periods of monitor downtime are as described in paragraphs (j)(1)(ii)(A) and (B) of this section.

(A) An excess emission shall be the period of time during which the fuel-bound nitrogen (N) is greater than the value measured during the performance test required in §60.8 and used to determine the allowance. The excess emission begins on the date and hour of the sample which shows that N is greater than the performance test value, and ends with the date and hour of a subsequent sample which shows a fuel nitrogen content less than or equal to the performance test value.

(B) A period of monitor downtime begins when a required sample is not taken by its due date. A period of monitor downtime also begins on the date and hour that a required sample is taken, if invalid results are obtained. The period of monitor downtime ends on the date and hour of the next valid sample.

(iii) For turbines using NO<sub>x</sub>and diluent CEMS:

(A) An hour of excess emissions shall be any unit operating hour in which the 4-hour rolling average NO<sub>x</sub>concentration exceeds the applicable emission limit in §60.332(a)(1) or (2). For the purposes of this subpart, a "4-hour rolling average NO<sub>x</sub>concentration" is the arithmetic average of the average NO<sub>x</sub>concentration measured by the CEMS for a given hour (corrected to 15 percent O<sub>2</sub>and, if required under §60.335(b)(1), to ISO standard conditions) and the three unit operating hour average NO<sub>x</sub>concentrations immediately preceding that unit operating hour.

(B) A period of monitor downtime shall be any unit operating hour in which sufficient data are not obtained to validate the hour, for either NO<sub>x</sub>concentration or diluent (or both).

(C) Each report shall include the ambient conditions (temperature, pressure, and humidity) at the time of the excess emission period and (if the owner or operator has claimed an emission allowance for fuel bound nitrogen) the nitrogen content of the fuel during the period of excess emissions. You do not have to report ambient conditions if you opt to use the worst case ISO correction factor as specified in §60.334(b)(3)(ii), or if you are not using the ISO correction equation under the provisions of §60.335(b)(1).

(iv) For owners or operators that elect, under paragraph (f) of this section, to monitor combustion parameters or parameters that document proper operation of the NO<sub>x</sub>emission controls:

(A) An excess emission shall be a 4-hour rolling unit operating hour average in which any monitored parameter does not achieve the target value or is outside the acceptable range defined in the parameter monitoring plan for the unit.

(B) A period of monitor downtime shall be a unit operating hour in which any of the required parametric data are either not recorded or are invalid.

(2) Sulfur dioxide. If the owner or operator is required to monitor the sulfur content of the fuel under paragraph (h) of this section:

(i) For samples of gaseous fuel and for oil samples obtained using daily sampling, flow proportional sampling, or sampling from the unit's storage tank, an excess emission occurs each unit operating hour included in the period beginning on the date and hour of any sample for which the sulfur content of the fuel being fired in the gas turbine exceeds 0.8 weight percent and ending on the date and hour that a subsequent sample is taken that demonstrates compliance with the sulfur limit.

(ii) If the option to sample each delivery of fuel oil has been selected, the owner or operator shall immediately switch to one of the other oil sampling options (*i.e.*, daily sampling, flow proportional sampling, or sampling from the unit's storage tank) if the sulfur content of a delivery exceeds 0.8 weight percent. The owner or operator shall continue to use one of the other sampling options until all of the oil from the delivery has been combusted, and shall evaluate excess emissions according to paragraph (j)(2)(i) of this section. When all of the fuel from the delivery has been burned, the owner or operator may resume using the as-delivered sampling option.

(iii) A period of monitor downtime begins when a required sample is not taken by its due date. A period of monitor downtime also begins on the date and hour of a required sample, if invalid results are obtained. The period of monitor downtime shall include only unit operating hours, and ends on the date and hour of the next valid sample.

(3) *Ice fog.* Each period during which an exemption provided in §60.332(f) is in effect shall be reported in writing to the Administrator quarterly. For each period the ambient conditions existing during the period, the date and time the air pollution control system was deactivated, and the date and time the air pollution control system was reactivated shall be reported. All quarterly reports shall be postmarked by the 30th day following the end of each calendar quarter.

(4) *Emergency fuel.* Each period during which an exemption provided in §60.332(k) is in effect shall be included in the report required in §60.7(c). For each period, the type, reasons, and duration of the firing of the emergency fuel shall be reported.

(5) All reports required under §60.7(c) shall be postmarked by the 30th day following the end of each 6-month period.

[44 FR 52798, Sept. 10, 1979, as amended at 47 FR 3770, Jan. 27, 1982; 65 FR 61759, Oct. 17, 2000; 69 FR 41360, July 8, 2004; 71 FR 9457, Feb. 24, 2006]

### § 60.335 Test methods and procedures.

(a) The owner or operator shall conduct the performance tests required in §60.8, using either

(1) EPA Method 20,

(2) ASTM D6522-00 (incorporated by reference, see §60.17), or

(3) EPA Method 7E and either EPA Method 3 or 3A in appendix A to this part, to determine NO<sub>x</sub>and diluent concentration.

(4) Sampling traverse points are to be selected following Method 20 or Method 1, (non-particulate procedures) and sampled for equal time intervals. The sampling shall be performed with a traversing single-hole probe or, if feasible, with a stationary multi-hole probe that samples each of the points sequentially. Alternatively, a multi-hole probe designed and documented to sample equal volumes from each hole may be used to sample simultaneously at the required points.

(5) Notwithstanding paragraph (a)(4) of this section, the owner or operator may test at few points than are specified in Method 1 or Method 20 if the following conditions are met:

(i) You may perform a stratification test for NO<sub>x</sub>and diluent pursuant to

(A) [Reserved]

(B) The procedures specified in section 6.5.6.1(a) through (e) appendix A to part 75 of this chapter.

(ii) Once the stratification sampling is completed, the owner or operator may use the following alternative sample point selection criteria for the performance test:

(A) If each of the individual traverse point NO<sub>x</sub>concentrations, normalized to 15 percent  $O_2$ , is within ±10 percent of the mean normalized concentration for all traverse points, then you may use 3 points (located either 16.7, 50.0, and 83.3 percent of the way across the stack or duct, or, for circular stacks or ducts greater than 2.4 meters (7.8 feet) in diameter, at 0.4, 1.2, and 2.0 meters from the wall). The 3 points shall be located along the measurement line that exhibited the highest average normalized NO<sub>x</sub>concentration during the stratification test; or

(B) If each of the individual traverse point NO<sub>x</sub>concentrations, normalized to 15 percent  $O_2$ , is within ±5 percent of the mean normalized concentration for all traverse points, then you may sample at a single point, located at least 1 meter from the stack wall or at the stack centroid.

(6) Other acceptable alternative reference methods and procedures are given in paragraph (c) of this section.

(b) The owner or operator shall determine compliance with the applicable nitrogen oxides emission limitation in §60.332 and shall meet the performance test requirements of §60.8 as follows:

(1) For each run of the performance test, the mean nitrogen oxides emission concentration ( $NO_{xo}$ ) corrected to 15 percent  $O_2$ shall be corrected to ISO standard conditions using the following equation. Notwithstanding this requirement, use of the ISO correction equation is optional for: Lean premix stationary combustion turbines; units used in association with heat recovery steam generators (HRSG) equipped with duct burners; and units equipped with add-on emission control devices:

NO<sub>x</sub>=(NO<sub>x0</sub>)(P<sub>r</sub>/P<sub>0</sub>)<sup>0.5</sup> e19 (Ho-0.00633)(288°K/T<sub>a</sub>)<sup>1.53</sup>

#### Where:

Page 10 of 12 (NSPS Subpart GG)

 $NO_X$ = emission concentration of  $NO_X$ at 15 percent  $O_2$ and ISO standard ambient conditions, ppm by volume, dry basis,

 $NO_{Xo}$  = mean observed  $NO_X$  concentration, ppm by volume, dry basis, at 15 percent  $O_2$ ,

 $P_r$ = reference combustor inlet absolute pressure at 101.3 kilopascals ambient pressure, mm Hg,

P<sub>o</sub>= observed combustor inlet absolute pressure at test, mm Hg,

H<sub>o</sub>= observed humidity of ambient air, g H<sub>2</sub>O/g air,

e = transcendental constant, 2.718, and

 $T_a$ = ambient temperature, °K.

(2) The 3-run performance test required by §60.8 must be performed within ±5 percent at 30, 50, 75, and 90-to-100 percent of peak load or at four evenly-spaced load points in the normal operating range of the gas turbine, including the minimum point in the operating range and 90-to-100 percent of peak load, or at the highest achievable load point if 90-to-100 percent of peak load cannot be physically achieved in practice. If the turbine combusts both oil and gas as primary or backup fuels, separate performance testing is required for each fuel. Notwithstanding these requirements, performance testing is not required for any emergency fuel (as defined in §60.331).

(3) For a combined cycle turbine system with supplemental heat (duct burner), the owner or operator may elect to measure the turbine  $NO_x$  emissions after the duct burner rather than directly after the turbine. If the owner or operator elects to use this alternative sampling location, the applicable  $NO_x$  emission limit in §60.332 for the combustion turbine must still be met.

(4) If water or steam injection is used to control NO<sub>x</sub>with no additional post-combustion NO<sub>x</sub>control and the owner or operator chooses to monitor the steam or water to fuel ratio in accordance with §60.334(a), then that monitoring system must be operated concurrently with each EPA Method 20, ASTM D6522–00 (incorporated by reference, see §60.17), or EPA Method 7E run and shall be used to determine the fuel consumption and the steam or water to fuel ratio necessary to comply with the applicable §60.332 NO<sub>x</sub>emission limit.

(5) If the owner operator elects to claim an emission allowance for fuel bound nitrogen as described in §60.332, then concurrently with each reference method run, a representative sample of the fuel used shall be collected and analyzed, following the applicable procedures described in §60.335(b)(9). These data shall be used to determine the maximum fuel nitrogen content for which the established water (or steam) to fuel ratio will be valid.

(6) If the owner or operator elects to install a CEMS, the performance evaluation of the CEMS may either be conducted separately (as described in paragraph (b)(7) of this section) or as part of the initial performance test of the affected unit.

(7) If the owner or operator elects to install and certify a NO<sub>x</sub>CEMS under §60.334(e), then the initial performance test required under §60.8 may be done in the following alternative manner:

(i) Perform a minimum of 9 reference method runs, with a minimum time per run of 21 minutes, at a single load level, between 90 and 100 percent of peak (or the highest physically achievable) load.

(ii) Use the test data both to demonstrate compliance with the applicable NO<sub>x</sub>emission limit under §60.332 and to provide the required reference method data for the RATA of the CEMS described under §60.334(b).

(iii) The requirement to test at three additional load levels is waived.

(8) If the owner or operator elects under §60.334(f) to monitor combustion parameters or parameters indicative of proper operation of NO<sub>x</sub>emission controls, the appropriate parameters shall be continuously monitored and recorded during each run of the initial performance test, to establish acceptable operating ranges, for purposes of the parameter monitoring plan for the affected unit, as specified in §60.334(g).

(9) To determine the fuel bound nitrogen content of fuel being fired (if an emission allowance is claimed for fuel bound nitrogen), the owner or operator may use equipment and procedures meeting the requirements of:

(i) For liquid fuels, ASTM D2597-94 (Reapproved 1999), D6366-99, D4629-02, D5762-02 (all of which are incorporated by reference, see §60.17); or

(ii) For gaseous fuels, shall use analytical methods and procedures that are accurate to within 5 percent of the instrument range and are approved by the Administrator.

(10) If the owner or operator is required under §60.334(i)(1) or (3) to periodically determine the sulfur content of the fuel combusted in the turbine, a minimum of three fuel samples shall be collected during the performance test. Analyze the samples for the total sulfur content of the fuel using:

(i) For liquid fuels, ASTM D129–00, D2622–98, D4294–02, D1266–98, D5453–00 or D1552–01 (all of which are incorporated by reference, see §60.17); or

(ii) For gaseous fuels, ASTM D1072–80, 90 (Reapproved 1994); D3246–81, 92, 96; D4468–85 (Reapproved 2000); or D6667–01 (all of which are incorporated by reference, see §60.17). The applicable ranges of some ASTM methods mentioned above are not adequate to measure the levels of sulfur in some fuel gases. Dilution of samples before analysis (with verification of the dilution ratio) may be used, subject to the prior approval of the Administrator.

(11) The fuel analyses required under paragraphs (b)(9) and (b)(10) of this section may be performed by the owner or operator, a service contractor retained by the owner or operator, the fuel vendor, or any other qualified agency.

(c) The owner or operator may use the following as alternatives to the reference methods and procedures specified in this section:

(1) Instead of using the equation in paragraph (b)(1) of this section, manufacturers may develop ambient condition correction factors to adjust the nitrogen oxides emission level measured by the performance test as provided in §60.8 to ISO standard day conditions.

[69 FR 41363, July 8, 2004, as amended at 71 FR 9458, Feb. 24, 2006]

Browse Previous | Browse Next

# **APPENDIX C**

.

40 CFR Part 61, Subpart FF National Emission Standards for Hazardous Air Pollutants, Benzene Waste Operations

# e-CFR Data is current as of October 6, 2009

### **Title 40: Protection of Environment**

PART 61-NATIONAL EMISSION STANDARDS FOR HAZARDOUS AIR POLLUTANTS

Browse Previous

### Subpart FF—National Emission Standard for Benzene Waste Operations

Source: 55 FR 8346, Mar. 7, 1990, unless otherwise noted.

## § 61.340 Applicability.

(a) The provisions of this subpart apply to owners and operators of chemical manufacturing plants, coke by-product recovery plants, and petroleum refineries.

(b) The provisions of this subpart apply to owners and operators of hazardous waste treatment, storage, and disposal facilities that treat, store, or dispose of hazardous waste generated by any facility listed in paragraph (a) of this section. The waste streams at hazardous waste treatment, storage, and disposal facilities subject to the provisions of this subpart are the benzene-containing hazardous waste from any facility listed in paragraph (a) of this section. A hazardous waste treatment, storage, and disposal facility is a facility that must obtain a hazardous waste management permit under subtitle C of the Solid Waste Disposal Act.

(c) At each facility identified in paragraph (a) or (b) of this section, the following waste is exempt from the requirements of this subpart:

(1) Waste in the form of gases or vapors that is emitted from process fluids:

(2) Waste that is contained in a segregated stormwater sewer system.

(d) At each facility identified in paragraph (a) or (b) of this section, any gaseous stream from a waste management unit, treatment process, or wastewater treatment system routed to a fuel gas system, as defined in §61.341, is exempt from this subpart. No testing, monitoring, recordkeeping, or reporting is required under this subpart for any gaseous stream from a waste management unit, treatment process, or wastewater treatment unit routed to a fuel gas system.

[55 FR 8346, Mar. 7, 1990, as amended at 55 FR 37231, Sept. 10, 1990; 58 FR 3095, Jan. 7, 1993; 67 FR 68531, Nov. 12, 2002]

## § 61.341 Definitions.

Benzene concentration means the fraction by weight of benzene in a waste as determined in accordance with the procedures specified in §61.355 of this subpart.

Car-seal means a seal that is placed on a device that is used to change the position of a valve (e.g., from opened to closed) in such a way that the position of the valve cannot be changed without breaking the seal.

*Chemical manufacturing plant* means any facility engaged in the production of chemicals by chemical, thermal, physical, or biological processes for use as a product, co-product, by-product, or intermediate including but not limited to industrial organic chemicals, organic pesticide products, pharmaceutical preparations, paint and allied products, fertilizers, and agricultural chemicals. Examples of chemical manufacturing plants include facilities at which process units are operated to produce one or more of the following chemicals: benzenesulfonic acid, benzene, chlorobenzene, cumene, cyclohexane, ethylene, ethylbenzene, hydroquinone, linear alklylbenzene, nitrobenzene, resorcinol, sulfolane, or styrene.

*Closed-vent system* means a system that is not open to the atmosphere and is composed of piping, ductwork, connections, and, if necessary, flow inducing devices that transport gas or vapor from an emission source to a control device.

Coke by-product recovery plant means any facility designed and operated for the separation and recovery of coal tar derivatives (by-products) evolved from coal during the coking process of a coke oven battery.

*Container* means any portable waste management unit in which a material is stored, transported, treated, or otherwise handled. Examples of containers are drums, barrels, tank trucks, barges, dumpsters, tank cars, dump trucks, and ships.

Control device means an enclosed combustion device, vapor recovery system, or flare.

Cover means a device or system which is placed on or over a waste placed in a waste management unit so that the entire waste surface area is enclosed and sealed to minimize air emissions. A cover may have openings necessary for operation, inspection, and maintenance of the waste management unit such as access hatches, sampling ports, and gauge wells provided that each opening is closed and sealed when not in use. Example of covers include a fixed roof installed on a tank, a lid installed on a container, and an air-supported enclosure installed over a waste management unit.

External floating roof means a pontoon-type or double-deck type cover with certain rim sealing mechanisms that rests on the liquid surface in a waste management unit with no fixed roof.

Facility means all process units and product tanks that generate waste within a stationary source, and all waste management units that are used for waste treatment, storage, or disposal within a stationary source.

Fixed roof means a cover that is mounted on a waste management unit in a stationary manner and that does not move with fluctuations in liquid level.

Floating roof means a cover with certain rim sealing mechanisms consisting of a double deck, pontoon single deck, internal floating cover or covered floating roof, which rests upon and is supported by the liquid being contained, and is equipped with a closure seal or seals to close the space between the roof edge and unit wall.

Flow indicator means a device which indicates whether gas flow is present in a line or vent system.

*Fuel gas system* means the offsite and onsite piping and control system that gathers gaseous streams generated by facility operations, may blend them with sources of gas, if available, and transports the blended gaseous fuel at suitable pressures for use as fuel in heaters, furnaces, boilers, incinerators, gas turbines, and other combustion devices located within or outside the facility. The fuel is piped directly to each individual combustion device, and the system typically operates at pressures over atmospheric.

Individual drain system means the system used to convey waste from a process unit, product storage tank, or waste management unit to a waste management unit. The term includes all process drains and common junction boxes, together with their associated sewer lines and other junction boxes, down to the receiving waste management unit.

Internal floating roof means a cover that rests or floats on the liquid surface inside a waste management unit that has a fixed roof.

Liquid-mounted seal means a foam or liquid-filled primary seal mounted in contact with the liquid between the waste management unit wall and the floating roof continuously around the circumference.

Loading means the introduction of waste into a waste management unit but not necessarily to complete capacity (also referred to as filling).

Maximum organic vapor pressure means the equilibrium partial pressure exerted by the waste at the temperature equal to the highest calendar-month average of the waste storage temperature for waste stored above or below the ambient temperature or at the local maximum monthly average temperature as reported by the National Weather Service for waste stored at the ambient temperature, as determined:

- (1) In accordance with §60.17(c); or
- (2) As obtained from standard reference texts; or
- (3) In accordance with §60.17(a)(37); or
- (4) Any other method approved by the Administrator.

No detectable emissions means less than 500 parts per million by volume (ppmv) above background levels, as measured by a detection instrument reading in accordance with the procedures specified in §61.355(h) of this subpart.

*Oil-water separator* means a waste management unit, generally a tank or surface impoundment, used to separate oil from water. An oil-water separator consists of not only the separation unit but also the forebay and other separator basins, skimmers, weirs, grit chambers, sludge hoppers, and bar screens that are located directly after the individual drain system and prior to additional treatment units such as an air flotation unit, clarifier, or biological treatment unit. Examples of an oil-water separator incude an API separator, parallel-plate interceptor, and corrugated-plate interceptor with the associated ancillary equipment.

Petroleum refinery means any facility engaged in producing gasoline, kerosene, distillate fuel oils, residual fuel oils, lubricants, or other products through the distillation of petroleum, or through the redistillation, cracking, or reforming of unfinished petroleum derivatives.

Petroleum means the crude oil removed from the earth and the oils derived from tar sands, shale, and coal.

Point of waste generation means the location where the waste stream exits the process unit component or storage tank prior to handling or treatment in an operation that is not an integral part of the production process, or in the case of waste management units that generate new wastes after treatment, the location where the waste stream exits the waste management unit component.

Process unit means equipment assembled and connected by pipes or ducts to produce intermediate or final products. A process unit can be operated independently if supplied with sufficient fuel or raw materials and sufficient product storage facilities.

Process unit turnaround means the shutting down of the operations of a process unit, the purging of the contents of the process unit, the maintenance or repair work, followed by restarting of the process.

Process unit turnaround waste means a waste that is generated as a result of a process unit turnaround.

Process wastewater means water which comes in contact with benzene during manufacturing or processing operations conducted within a process unit. Process wastewater is not organic wastes, process fluids, product tank drawdown, cooling tower blowdown, steam trap condensate, or landfill leachate.

Process wastewater stream means a waste stream that contains only process wastewater.

*Product tank* means a stationary unit that is designed to contain an accumulation of materials that are fed to or produced by a process unit, and is constructed primarily of non-earthen materials (e.g., wood, concrete, steel, plastic) which provide structural support.

Product tank drawdown means any material or mixture of materials discharged from a product tank for the purpose of removing water or other contaminants from the product tank.

Safety device means a closure device such as a pressure relief valve, frangible disc, fusible plug, or any other type of device which functions exclusively to prevent physical damage or permanent deformation to a unit or its air emission control equipment by venting gases or vapors directly to the atmosphere during unsafe conditions resulting from an unplanned, accidental, or emergency event. For the purpose of this subpart, a safety device is not used for routine venting of gases or vapors from the vapor headspace underneath a cover such as during filling of the unit or to adjust the pressure in this vapor headspace in response to normal daily diurnal ambient temperature fluctuations. A safety device is designed to remain in a closed position during normal operations and open only when the internal pressure, or another relevant parameter, exceeds the device threshold setting applicable to the air emission control equipment as determined by the owner or operator based on manufacturer recommendations, applicable regulations, fire protection and prevention codes, standard engineering codes and practices, or other requirements for the safe handling of flammable, ignitable, explosive, reactive, or hazardous materials.

Segregated stormwater sewer system means a drain and collection system designed and operated for the sole purpose of collecting rainfall runoff at a facility, and which is segregated from all other individual drain systems.

Sewer line means a lateral, trunk line, branch line, or other enclosed conduit used to convey waste to a downstream waste management unit.

Slop oil means the floating oil and solids that accumulate on the surface of an oil-water separator.

Sour water stream means a stream that:

(1) Contains ammonia or sulfur compounds (usually hydrogen sulfide) at concentrations of 10 ppm by weight or more;

(2) Is generated from separation of water from a feed stock, intermediate, or product that contained ammonia or sulfur compounds; and

(3) Requires treatment to remove the ammonia or sulfur compounds.

Sour water stripper means a unit that:

(1) Is designed and operated to remove ammonia or sulfur compounds (usually hydrogen sulfide) from sour water streams;

(2) Has the sour water streams transferred to the stripper through hard piping or other enclosed system; and

(3) Is operated in such a manner that the offgases are sent to a sulfur recovery unit, processing unit, incinerator, flare, or other combustion device.

Surface impoundment means a waste management unit which is a natural topographic depression, man-made excavation, or diked area formed primarily of earthen materials (although it may be lined with man-made materials), which is designed to hold an accumulation of liquid wastes or waste containing free liquids, and which is not an injection well. Examples of surface impoundments are holding, storage, settling, and aeration pits, ponds, and lagoons.

Tank means a stationary waste management unit that is designed to contain an accumulation of waste and is constructed primarily of nonearthen materials (e.g., wood, concrete, steel, plastic) which provide structural support.

Treatment process means a stream stripping unit, thin-film evaporation unit, waste incinerator, or any other process used to comply with §61.348 of this subpart.

Vapor-mounted seal means a foam-filled primary seal mounted continuously around the perimeter of a waste management unit so there is an annular vapor space underneath the seal. The annular vapor space is bounded by the bottom of the primary seal, the unit wall, the liquid surface, and the floating roof.

Waste means any material resulting from industrial, commercial, mining or agricultural operations, or from community activities that is discarded or is being accumulated, stored, or physically, chemically, thermally, or biologically treated prior to being discarded, recycled, or discharged.

Waste management unit means a piece of equipment, structure, or transport mechanism used in handling, storage, treatment, or disposal of waste. Examples of a waste management unit include a tank, surface impoundment, container, oil-water separator, individual drain system, steam stripping unit, thin-film evaporation unit, waste incinerator, and landfill.

Waste stream means the waste generated by a particular process unit, product tank, or waste management unit. The characteristics of the waste stream (e.g., flow rate, benzene concentration, water content) are determined at the point of waste generation. Examples of a waste stream include process wastewater, product tank drawdown, sludge and slop oil removed from waste management units, and landfill leachate.

Wastewater treatment system means any component, piece of equipment, or installation that receives, manages, or treats process wastewater, product tank drawdown, or landfill leachate prior to direct or indirect discharge in accordance with the National Pollutant Discharge Elimination System permit regulations under 40 CFR part 122. These systems typically include individual drain systems, oil-water separators, air flotation units, equalization tanks, and biological treatment units.

Water seal controls means a seal pot, p-leg trap, or other type of trap filled with water (e.g., flooded sewers that maintain water levels adequate to prevent air flow through the system) that creates a water barrier between the sewer line and the atmosphere. The water level of the seal must be maintained in the vertical leg of a drain in order to be considered a water seal.

[55 FR 8346, Mar. 7, 1990; 55 FR 12444, Apr. 3, 1990, as amended at 58 FR 3095, Jan. 7, 1993; 67 FR 68531, Nov. 12, 2002]

# § 61.342 Standards: General.

(a) An owner or operator of a facility at which the total annual benzene quantity from facility waste is less than 10 megagrams per year (Mg/yr) (11 ton/yr) shall be exempt from the requirements of paragraphs (b) and (c) of this section. The total annual benzene quantity from facility waste is the sum of the annual benzene quantity for each waste stream at the facility that has a flow-weighted annual average water content greater than 10 percent or that is mixed with water, or other wastes, at any time and the mixture has an annual average water content greater than 10 percent. The benzene quantity in a waste stream is to be counted only once without

multiple counting if other waste streams are mixed with or generated from the original waste stream. Other specific requirements for calculating the total annual benzene waste quantity are as follows:

(1) Wastes that are exempted from control under §§61.342(c)(2) and 61.342(c)(3) are included in the calculation of the total annual benzene quantity if they have an annual average water content greater than 10 percent, or if they are mixed with water or other wastes at any time and the mixture has an annual average water content greater than 10 percent.

(2) The benzene in a material subject to this subpart that is sold is included in the calculation of the total annual benzene quantity if the material has an annual average water content greater than 10 percent.

(3) Benzene in wastes generated by remediation activities conducted at the facility, such as the excavation of contaminated soil, pumping and treatment of groundwater, and the recovery of product from soil or groundwater, are not included in the calculation of total annual benzene quantity for that facility. If the facility's total annual benzene quantity is 10 Mg/yr (11 ton/yr) or more, wastes generated by remediation activities are subject to the requirements of paragraphs (c) through (h) of this section. If the facility is managing remediation waste generated offsite, the benzene in this waste shall be included in the calculation of total annual benzene quantity in facility waste, if the waste streams have an annual average water content greater than 10 percent, or if they are mixed with water or other wastes at any time and the mixture has an annual average water content greater than 10 percent.

(4) The total annual benzene quantity is determined based upon the quantity of benzene in the waste before any waste treatment occurs to remove the benzene except as specified in  $\S61.355(c)(1)(i)$  (A) through (C).

(b) Each owner or operator of a facility at which the total annual benzene quantity from facility waste is equal to or greater than 10 Mg/yr (11 ton/yr) as determined in paragraph (a) of this section shall be in compliance with the requirements of paragraphs (c) through (h) of this section no later than 90 days following the effective date, unless a waiver of compliance has been obtained under §61.11, or by the initial startup for a new source with an initial startup after the effective date.

(1) The owner or operator of an existing source unable to comply with the rule within the required time may request a waiver of compliance under §61.10.

(2) As part of the waiver application, the owner or operator shall submit to the Administrator a plan under §61.10(b)(3) that is an enforceable commitment to obtain environmental benefits to mitigate the benzene emissions that result from extending the compliance date. The plan shall include the following information:

(i) A description of the method of compliance, including the control approach, schedule for installing controls, and quantity of the benzene emissions that result from extending the compliance date;

(ii) If the control approach involves a compliance strategy designed to obtain integrated compliance with multiple regulatory requirements, a description of the other regulations involved and their effective dates; and

(iii) A description of the actions to be taken at the facility to obtain mitigating environmental benefits, including how the benefits will be obtained, the schedule for these actions, and an estimate of the quantifiable benefits that directly result from these actions.

(c) Each owner or operator of a facility at which the total annual benzene quantity from facility waste is equal to or greater than 10 Mg/yr (11 ton/yr) as determined in paragraph (a) of this section shall manage and treat the facility waste as follows:

(1) For each waste stream that contains benzene, including (but not limited to) organic waste streams that contain less than 10 percent water and aqueous waste streams, even if the wastes are not discharged to an individual drain system, the owner or operator shall:

(i) Remove or destroy the benzene contained in the waste using a treatment process or wastewater treatment system that complies with the standards specified in §61.348 of this subpart.

(ii) Comply with the standards specified in §§61.343 through 61.347 of this subpart for each waste management unit that receives or manages the waste stream prior to and during treatment of the waste stream in accordance with paragraph (c)(1)(i) of this section.

(iii) Each waste management unit used to manage or treat waste streams that will be recycled to a process shall comply with the standards specified in §§61.343 through 61.347. Once the waste stream is recycled to a process, including to a tank used for the storage of production process feed, product, or product intermediates, unless this tank is used primarily for the storage of wastes, the material is no longer subject to paragraph (c) of this section.

(2) A waste stream is exempt from paragraph (c)(1) of this section provided that the owner or operator demonstrates initially and,

thereafter, at least once per year that the flow-weighted annual average benzene concentration for the waste stream is less than 10 ppmw as determined by the procedures specified in §61.355(c)(2) or §61.355(c)(3).

(3) A waste stream is exempt from paragraph (c)(1) of this section provided that the owner or operator demonstrates initially and, thereafter, at least once per year that the conditions specified in either paragraph (c)(3)(i) or (c)(3)(ii) of this section are met.

(i) The waste stream is process wastewater that has a flow rate less than 0.02 liters per minute (0.005 gallons per minute) or an annual wastewater quantity of less than 10 Mg/yr (11 ton/yr); or

(ii) All of the following conditions are met:

(A) The owner or operator does not choose to exempt process wastewater under paragraph (c)(3)(i) of this section,

(B) The total annual benzene quantity in all waste streams chosen for exemption in paragraph (c)(3)(ii) of this section does not exceed 2.0 Mg/yr (2.2 ton/yr) as determined in the procedures in §61.355(j), and

(C) The total annual benzene quantity in a waste stream chosen for exemption, including process unit turnaround waste, is determined for the year in which the waste is generated.

(d) As an alternative to the requirements specified in paragraphs (c) and (e) of this section, an owner or operator of a facility at which the total annual benzene quantity from facility waste is equal to or greater than 10 Mg/yr (11 ton/yr) as determined in paragraph (a) of this section may elect to manage and treat the facility waste as follows:

(1) The owner or operator shall manage and treat facility waste other than process wastewater in accordance with the requirements of paragraph (c)(1) of this section.

(2) The owner or operator shall manage and treat process wastewater in accordance with the following requirements:

(i) Process wastewater shall be treated to achieve a total annual benzene quantity from facility process wastewater less than 1 Mg/yr (1.1 ton/yr). Total annual benzene from facility process wastewater shall be determined by adding together the annual benzene quantity at the point of waste generation for each untreated process wastewater stream plus the annual benzene quantity exiting the treatment process for each process wastewater stream treated in accordance with the requirements of paragraph (c)(1)(i) of this section.

(ii) Each treated process wastewater stream identified in paragraph (d)(2)(i) of this section shall be managed and treated in accordance with paragraph (c)(1) of this section.

(iii) Each untreated process wastewater stream identified in paragraph (d)(2)(i) of this section is exempt from the requirements of paragraph (c)(1) of this section.

(e) As an alternative to the requirements specified in paragraphs (c) and (d) of this section, an owner or operator of a facility at which the total annual benzene quantity from facility waste is equal to or greater than 10 Mg/yr (11 ton/yr) as determined in paragraph (a) of this section may elect to manage and treat the facility waste as follows:

(1) The owner or operator shall manage and treat facility waste with a flow-weighted annual average water content of less than 10 percent in accordance with the requirements of paragraph (c)(1) of this section; and

(2) The owner or operator shall manage and treat facility waste (including remediation and process unit turnaround waste) with a flow-weighted annual average water content of 10 percent or greater, on a volume basis as total water, and each waste stream that is mixed with water or wastes at any time such that the resulting mixture has an annual water content greater than 10 percent, in accordance with the following:

(i) The benzene quantity for the wastes described in paragraph (e)(2) of this section must be equal to or less than 6.0 Mg/yr (6.6 ton/yr), as determined in §61.355(k). Wastes as described in paragraph (e)(2) of this section that are transferred offsite shall be included in the determination of benzene quantity as provided in §61.355(k). The provisions of paragraph (f) of this section shall not apply to any owner or operator who elects to comply with the provisions of paragraph (e) of this section.

(ii) The determination of benzene quantity for each waste stream defined in paragraph (e)(2) of this section shall be made in accordance with §61.355(k).

(f) Rather than treating the waste onsite, an owner or operator may elect to comply with paragraph (c)(1)(i) of this section by transferring the waste offsite to another facility where the waste is treated in accordance with the requirements of paragraph (c)(1)(i) of this section. The owner or operator transferring the waste shall:

(1) Comply with the standards specified in §§61.343 through 61.347 of this subpart for each waste management unit that receives or manages the waste prior to shipment of the waste offsite.

(2) Include with each offsite waste shipment a notice stating that the waste contains benzene which is required to be managed and treated in accordance with the provisions of this subpart.

(g) Compliance with this subpart will be determined by review of facility records and results from tests and inspections using methods and procedures specified in §61.355 of this subpart.

(h) Permission to use an alternative means of compliance to meet the requirements of §§61.342 through 61.352 of this subpart may be granted by the Administrator as provided in §61.353 of this subpart.

[55 FR 8346, Mar. 7, 1990, as amended at 58 FR 3095, Jan. 7, 1993; 65 FR 62159, 62160, Oct. 17, 2000]

## § 61.343 Standards: Tanks.

(a) Except as provided in paragraph (b) of this section and in §61.351, the owner or operator must meet the standards in paragraph (a)(1) or (2) of this section for each tank in which the waste stream is placed in accordance with §61.342 (c)(1)(ii). The standards in this section apply to the treatment and storage of the waste stream in a tank, including dewatering.

(1) The owner or operator shall install, operate, and maintain a fixed-roof and closed-vent system that routes all organic vapors vented from the tank to a control device.

(i) The fixed-roof shall meet the following requirements:

(A) The cover and all openings (e.g., access hatches, sampling ports, and gauge wells) shall be designed to operate with no detectable emissions as indicated by an instrument reading of less than 500 ppmv above background, as determined initially and thereafter at least once per year by the methods specified in §61.355(h) of this subpart.

(B) Each opening shall be maintained in a closed, sealed position (e.g., covered by a lid that is gasketed and latched) at all times that waste is in the tank except when it is necessary to use the opening for waste sampling or removal, or for equipment inspection, maintenance, or repair.

(C) If the cover and closed-vent system operate such that the tank is maintained at a pressure less than atmospheric pressure, then paragraph (a)(1)(i)(B) of this section does not apply to any opening that meets all of the following conditions:

(1) The purpose of the opening is to provide dilution air to reduce the explosion hazard;

(2) The opening is designed to operate with no detectable emissions as indicated by an instrument reading of less than 500 ppmv above background, as determined initially and thereafter at least once per year by the methods specified in §61.355(h); and

(3) The pressure is monitored continuously to ensure that the pressure in the tank remains below atmospheric pressure.

(ii) The closed-vent system and control device shall be designed and operated in accordance with the requirements of §61.349 of this subpart.

(2) The owner or operator must install, operate, and maintain an enclosure and closed-vent system that routes all organic vapors vented from the tank, located inside the enclosure, to a control device in accordance with the requirements specified in paragraph (e) of this section.

(b) For a tank that meets all the conditions specified in paragraph (b)(1) of this section, the owner or operator may elect to comply with paragraph (b)(2) of this section as an alternative to the requirements specified in paragraph (a)(1) of this section.

(1) The waste managed in the tank complying with paragraph (b)(2) of this section shall meet all of the following conditions:

(i) Each waste stream managed in the tank must have a flow-weighted annual average water content less than or equal to 10 percent water, on a volume basis as total water.

(ii) The waste managed in the tank either:

(A) Has a maximum organic vapor pressure less than 5.2 kilopascals (kPa) (0.75 pounds per square inch (psi));

(B) Has a maximum organic vapor pressure less than 27.6 kPa (4.0 psi) and is managed in a tank having design capacity less than 151 m<sup>3</sup> (40,000 gal); or

(C) Has a maximum organic vapor pressure less than 76.6 kPa (11.1 psi) and is managed in a tank having a design capacity less than 75 m<sup>3</sup> (20,000 gal).

(2) The owner or operator shall install, operate, and maintain a fixed roof as specified in paragraph (a)(1)(i).

(3) For each tank complying with paragraph (b) of this section, one or more devices which vent directly to the atmosphere may be used on the tank provided each device remains in a closed, sealed position during normal operations except when the device needs to open to prevent physical damage or permanent deformation of the tank or cover resulting from filling or emptying the tank, diurnal temperature changes, atmospheric pressure changes or malfunction of the unit in accordance with good engineering and safety practices for handling flammable, explosive, or other hazardous materials.

(c) Each fixed-roof, seal, access door, and all other openings shall be checked by visual inspection initially and quarterly thereafter to ensure that no cracks or gaps occur and that access doors and other openings are closed and gasketed properly.

(d) Except as provided in §61.350 of this subpart, when a broken seal or gasket or other problem is identified, or when detectable emissions are measured, first efforts at repair shall be made as soon as practicable, but not later than 45 calendar days after identification.

(e) Each owner or operator who controls air pollutant emissions by using an enclosure vented through a closed-vent system to a control device must meet the requirements specified in paragraphs (e)(1) through (4) of this section.

(1) The tank must be located inside a total enclosure. The enclosure must be designed and operated in accordance with the criteria for a permanent total enclosure as specified in "Procedure T----Criteria for and Verification of a Permanent or Temporary Total Enclosure" in 40 CFR 52.741, appendix B. The enclosure may have permanent or temporary openings to allow worker access; passage of material into or out of the enclosure by conveyor, vehicles, or other mechanical means; entry of permanent mechanical or electrical equipment; or direct airflow into the enclosure. The owner or operator must perform the verification procedure for the enclosure as specified in section 5.0 of Procedure T initially when the enclosure is first installed and, thereafter, annually. A facility that has conducted an initial compliance demonstration and that performs annual compliance demonstrations in accordance with the requirements for Tank Level 2 control requirements 40 CFR 264.1084(i) or 40 CFR 265(i) is not required to make repeat demonstrations of initial and continuous compliance for the purposes of this subpart.

(2) The enclosure must be vented through a closed-vent system to a control device that is designed and operated in accordance with the standards for control devices specified in §61.349.

(3) Safety devices, as defined in this subpart, may be installed and operated as necessary on any enclosure, closed-vent system, or control device used to comply with the requirements of paragraphs (e)(1) and (2) of this section.

(4) The closed-vent system must be designed and operated in accordance with the requirements of §61.349.

[55 FR 8346, Mar. 7, 1990, as amended at 55 FR 18331, May 2, 1990; 58 FR 3096, Jan. 7, 1993; 67 FR 68532, Nov. 12, 2002; 68 FR 6082, Feb. 6, 2003; 68 FR 67935, Dec. 4, 2003]

# § 61.344 Standards: Surface impoundments.

(a) The owner or operator shall meet the following standards for each surface impoundment in which waste is placed in accordance with §61.342(c)(1)(ii) of this subpart:

(1) The owner or operator shall install, operate, and maintain on each surface impoundment a cover (e.g., air-supported structure or rigid cover) and closed-vent system that routes all organic vapors vented from the surface impoundment to a control device.

(i) The cover shall meet the following requirements:

(A) The cover and all openings (e.g., access hatches, sampling ports, and gauge wells) shall be designed to operate with no detectable emissions as indicated by an instrument reading of less than 500 ppmv above background, initially and thereafter at least once per year by the methods specified in §61.355(h) of this subpart.

(B) Each opening shall be maintained in a closed, sealed position (e.g., covered by a lid that is gasketed and latched) at all times that waste is in the surface impoundment except when it is necessary to use the opening for waste sampling or removal, or for equipment inspection, maintenance, or repair.

(C) If the cover and closed-vent system operate such that the enclosure of the surface impoundment is maintained at a pressure less than atmospheric pressure, then paragraph (a)(1)(i)(B) of this section does not apply to any opening that meets all of the following conditions:

(1) The purpose of the opening is to provide dilution air to reduce the explosion hazard;

(2) The opening is designed to operate with no detectable emissions as indicated by an instrument reading of less than 500 ppmv above background, as determined initially and thereafter at least once per year by the methods specified in §61.355(h) of this subpart; and

(3) The pressure is monitored continuously to ensure that the pressure in the enclosure of the surface impoundment remains below atmospheric pressure.

(D) The cover shall be used at all times that waste is placed in the surface impoundment except during removal of treatment residuals in accordance with 40 CFR 268.4 or closure of the surface impoundment in accordance with 40 CFR 264.228. (Note: the treatment residuals generated by these activities may be subject to the requirements of this part.)

(ii) The closed-vent system and control device shall be designed and operated in accordance with §61.349 of this subpart.

(b) Each cover seal, access hatch, and all other openings shall be checked by visual inspection initially and quarterly thereafter to ensure that no cracks or gaps occur and that access hatches and other openings are closed and gasketed properly.

(c) Except as provided in §61.350 of this subpart, when a broken seal or gasket or other problem is identified, or when detectable emissions are measured, first efforts at repair shall be made as soon as practicable, but not later than 15 calendar days after identification.

[55 FR 8346, Mar. 7, 1990, as amended at 58 FR 3097, Jan. 7, 1993]

### § 61.345 Standards: Containers.

(a) The owner or operator shall meet the following standards for each container in which waste is placed in accordance with §61.342(c)(1)(ii) of this subpart:

(1) The owner or operator shall install, operate, and maintain a cover on each container used to handle, transfer, or store waste in accordance with the following requirements:

(i) The cover and all openings (e.g., bungs, hatches, and sampling ports) shall be designed to operate with no detectable emissions as indicated by an instrument reading of less than 500 ppmv above background, initially and thereafter at least once per year by the methods specified in §61.355(h) of this subpart.

(ii) Except as provided in paragraph (a)(4) of this section, each opening shall be maintained in a closed, sealed position (e.g., covered by a lid that is gasketed and latched) at all times that waste is in the container except when it is necessary to use the opening for waste loading, removal, inspection, or sampling.

(2) When a waste is transferred into a container by pumping, the owner or operator shall perform the transfer using a submerged fill pipe. The submerged fill pipe outlet shall extend to within two fill pipe diameters of the bottom of the container while the container is being loaded. During loading of the waste, the cover shall remain in place and all openings shall be maintained in a closed, sealed position except for those openings required for the submerged fill pipe, those openings required for venting of the container to prevent physical damage or permanent deformation of the container or cover, and any openings complying with paragraph (a)(4) of this section.

(3) Treatment of a waste in a container, including aeration, thermal or other treatment, must be performed by the owner or operator in a manner such that while the waste is being treated the container meets the standards specified in paragraphs (a)(3)(i) through (iii) of this section, except for covers and closed-vent systems that meet the requirements in paragraph (a)(4) of this section.

(i) The owner or operator must either:

(A) Vent the container inside a total enclosure which is exhausted through a closed-vent system to a control device in accordance with the requirements of paragraphs (a)(3)(ii)(A) and (B) of this section; or

(B) Vent the covered or closed container directly through a closed-vent system to a control device in accordance with the requirements of paragraphs (a)(3)(ii)(B) and (C) of this section.

(ii) The owner or operator must meet the following requirements, as applicable to the type of air emission control equipment selected by the owner or operator:

(A) The total enclosure must be designed and operated in accordance with the criteria for a permanent total enclosure as specified in section 5 of the "Procedure T—Criteria for and Verification of a Permanent or Temporary Total Enclosure" in 40 CFR 52.741, appendix B. The enclosure may have permanent or temporary openings to allow worker access; passage of containers through the enclosure by conveyor or other mechanical means; entry of permanent mechanical or electrical equipment; or direct airflow into the enclosure. The owner or operator must perform the verification procedure for the enclosure as specified in section 5.0 of "Procedure T—Criteria for and Verification of a Permanent or Temporary Total Enclosure" initially when the enclosure is first installed and, thereafter, annually. A facility that has conducted an initial compliance demonstration and that performs annual compliance demonstrations in accordance with the Container Level 3 control requirements in 40 CFR 264.1086(e)(2)(i) or 40 CFR 265.1086(e)(2)(i) is not required to make repeat demonstrations of initial and continuous compliance for the purposes of this subpart.

(B) The closed-vent system and control device must be designed and operated in accordance with the requirements of §61.349.

(C) For a container cover, the cover and all openings (e.g., doors, hatches) must be designed to operate with no detectable emissions as indicated by an instrument reading of less than 500 ppmv above background, initially and thereafter at least once per year by the methods specified in §61.355(h).

(iii) Safety devices, as defined in this subpart, may be installed and operated as necessary on any container, enclosure, closed-vent system, or control device used to comply with the requirements of paragraph (a)(3)(i) of this section.

(4) If the cover and closed-vent system operate such that the container is maintained at a pressure less than atmospheric pressure, the owner or operator may operate the system with an opening that is not sealed and kept closed at all times if the following conditions are met:

(i) The purpose of the opening is to provide dilution air to reduce the explosion hazard;

(ii) The opening is designed to operate with no detectable emissions as indicated by an instrument reading of less than 500 ppmv above background, as determined initially and thereafter at least once per year by methods specified in §61.355(h); and

(iii) The pressure is monitored continuously to ensure that the pressure in the container remains below atmospheric pressure.

(b) Each cover and all openings shall be visually inspected initially and quarterly thereafter to ensure that they are closed and gasketed properly.

(c) Except as provided in §61.350 of this subpart, when a broken seal or gasket or other problem is identified, first efforts at repair shall be made as soon as practicable, but not later than 15 calendar days after identification.

[55 FR 8346, Mar. 7, 1990, as amended at 58 FR 3097, Jan. 7, 1993; 67 FR 68532, Nov. 12, 2002; 68 FR 67936, Dec. 4, 2003]

### § 61.346 Standards: Individual drain systems.

(a) Except as provided in paragraph (b) of this section, the owner or operator shall meet the following standards for each individual drain system in which waste is placed in accordance with §61.342(c)(1)(ii) of this subpart:

(1) The owner or operator shall install, operate, and maintain on each drain system opening a cover and closed-vent system that

routes all organic vapors vented from the drain system to a control device.

(i) The cover shall meet the following requirements:

(A) The cover and all openings (e.g., access hatches, sampling ports) shall be designed to operate with no detactable emissions as indicated by an instrument reading of less than 500 ppmv above background, initially and thereafter at least once per year by the methods specified in §61.355(h) of this subpart.

(B) Each opening shall be maintained in a closed, sealed position (e.g., covered by a lid that is gasketed and latched) at all times that waste is in the drain system except when it is necessary to use the opening for waste sampling or removal, or for equipment inspection, maintenance, or repair.

(C) If the cover and closed-vent system operate such that the individual drain system is maintained at a pressure less than atmospheric pressure, then paragraph (a)(1)(i)(B) of this section does not apply to any opening that meets all of the following conditions:

(1) The purpose of the opening is to provide dilution air to reduce the explosion hazard;

(2) The opening is designed to operate with no detectable emissions as indicated by an instrument reading of less than 500 ppmv above background, as determined initially and thereafter at least once per year by the methods specified in §61.355(h); and

(3) The pressure is monitored continuously to ensure that the pressure in the individual drain system remains below atmospheric pressure.

(ii) The closed-vent system and control device shall be designed and operated in accordance with §61.349 of this subpart.

(2) Each cover seal, access hatch, and all other openings shall be checked by visual inspection initially and quarterly thereafter to ensure that no cracks or gaps occur and that access hatches and other openings are closed and gasketed properly.

(3) Except as provided in §61.350 of this subpart, when a broken seal or gasket or other problem is identified, or when detectable emissions are measured, first efforts at repair shall be made as soon as practicable, but not later than 15 calendar days after identification.

(b) As an alternative to complying with paragraph (a) of this section, an owner or operator may elect to comply with the following requirements:

(1) Each drain shall be equipped with water seal controls or a tightly sealed cap or plug.

(2) Each junction box shall be equipped with a cover and may have a vent pipe. The vent pipe shall be at least 90 cm (3 ft) in length and shall not exceed 10.2 cm (4 in) in diameter.

(i) Junction box covers shall have a tight seal around the edge and shall be kept in place at all times, except during inspection and maintenance.

(ii) One of the following methods shall be used to control emissions from the junction box vent pipe to the atmosphere:

(A) Equip the junction box with a system to prevent the flow of organic vapors from the junction box vent pipe to the atmosphere during normal operation. An example of such a system includes use of water seal controls on the junction box. A flow indicator shall be installed, operated, and maintained on each junction box vent pipe to ensure that organic vapors are not vented from the junction box to the atmosphere during normal operation.

(B) Connect the junction box vent pipe to a closed-vent system and control device in accordance with §61.349 of this subpart.

(3) Each sewer line shall not be open to the atmosphere and shall be covered or enclosed in a manner so as to have no visual gaps or cracks in joints, seals, or other emission interfaces.

(4) Equipment installed in accordance with paragraphs (b)(1), (b)(2), or (b)(3) of this section shall be inspected as follows:

(i) Each drain using water seal controls shall be checked by visual or physical inspection initially and thereafter quarterly for indications of low water levels or other conditions that would reduce the effectiveness of water seal controls.

(ii) Each drain using a tightly sealed cap or plug shall be visually inspected initially and thereafter quarterly to ensure caps or plugs are in place and properly installed.

(iii) Each junction box shall be visually inspected initially and thereafter quarterly to ensure that the cover is in place and to ensure that the cover has a tight seal around the edge.

(iv) The unburied portion of each sewer line shall be visually inspected initially and thereafter quarterly for indication of cracks, gaps, or other problems that could result in benzene emissions.

(5) Except as provided in §61.350 of this subpart, when a broken seal, gap, crack or other problem is identified, first efforts at repair shall be made as soon as practicable, but not later than 15 calendar days after identification.

[55 FR 8346, Mar. 7, 1990, as amended at 55 FR 37231, Sept. 10, 1990; 58 FR 3097, Jan. 7, 1993]

# § 61.347 Standards: Oil-water separators.

(a) Except as provided in §61.352 of this subpart, the owner or operator shall meet the following standards for each oil-water separator in which waste is placed in accordance with §61.342(c)(1)(ii) of this subpart:

(1) The owner or operator shall install, operate, and maintain a fixed-roof and closed-vent system that routes all organic vapors vented from the oil-water separator to a control device.

(i) The fixed-roof shall meet the following requirements:

(A) The cover and all openings (e.g., access hatches, sampling ports, and gauge wells) shall be designed to operate with no detectable emissions as indicated by an instrument reading of less than 500 ppmv above background, as determined initially and thereafter at least once per year by the methods specified in §61.355(h) of this subpart.

(B) Each opening shall be maintained in a closed, sealed position (e.g., covered by a lid that is gasketed and latched) at all times that waste is in the oil-water separator except when it is necessary to use the opening for waste sampling or removal, or for equipment inspection, maintenance, or repair.

(C) If the cover and closed-vent system operate such that the oil-water separator is maintained at a pressure less than atmospheric pressure, then paragraph (a)(1)(i)(B) of this section does not apply to any opening that meets all of the following conditions:

(1) The purpose of the opening is to provide dilution air to reduce the explosion hazard;

(2) The opening is designed to operate with no detectable emissions as indicated by an instrument reading of less than 500 ppmv above background, as determined initially and thereafter at least once per year by the methods specified in §61.355(h); and

(3) The pressure is monitored continuously to ensure that the pressure in the oil-water separator remains below atmospheric pressure.

(ii) The closed-vent system and control device shall be designed and operated in accordance with the requirements of §61.349 of this subpart.

(b) Each cover seal, access hatch, and all other openings shall be checked by visual inspection initially and quarterly thereafter to ensure that no cracks or gaps occur between the cover and oil-water separator wall and that access hatches and other openings are closed and gasketed properly.

(c) Except as provided in §61.350 of this subpart, when a broken seal or gasket or other problem is identified, or when detectable emissions are measured, first efforts at repair shall be made as soon as practicable, but not later than 15 calendar days after identification.

[55 FR 8346, Mar. 7, 1990, as amended at 58 FR 3098, Jan. 7, 1993]

# § 61.348 Standards: Treatment processes.

(a) Except as provided in paragraph (a)(5) of this section, the owner or operator shall treat the waste stream in accordance with the following requirements:

(1) The owner or operator shall design, install, operate, and maintain a treatment process that either:

(i) Removes benzene from the waste stream to a level less than 10 parts per million by weight (ppmw) on a flow-weighted annual average basis,

(ii) Removes benzene from the waste stream by 99 percent or more on a mass basis, or

(iii) Destroys benzene in the waste stream by incinerating the waste in a combustion unit that achieves a destruction efficiency of 99 percent or greater for benzene.

(2) Each treatment process complying with paragraphs (a)(1)(i) or (a)(1)(ii) of this section shall be designed and operated in accordance with the appropriate waste management unit standards specified in §§61.343 through 61.347 of this subpart. For example, if a treatment process is a tank, then the owner or operator shall comply with §61.343 of this subpart.

(3) For the purpose of complying with the requirements specified in paragraph (a)(1)(i) of this section, the intentional or unintentional reduction in the benzene concentration of a waste stream by dilution of the waste stream with other wastes or materials is not allowed.

(4) An owner or operator may aggregate or mix together individual waste streams to create a combined waste stream for the purpose of facilitating treatment of waste to comply with the requirements of paragraph (a)(1) of this section except as provided in paragraph (a)(5) of this section.

(5) If an owner or operator aggregates or mixes any combination of process wastewater, product tank drawdown, or landfill leachate subject to §61.342(c)(1) of this subpart together with other waste streams to create a combined waste stream for the purpose of facilitating management or treatment of waste in a wastewater treatment system, then the wastewater treatment system shall be operated in accordance with paragraph (b) of this section. These provisions apply to above-ground wastewater treatment systems as well as those that are at or below ground level.

(b) Except for facilities complying with §61.342(e), the owner or operator that aggregates or mixes individual waste streams as defined in paragraph (a)(5) of this section for management and treatment in a wastewater treatment system shall comply with the following requirements:

(1) The owner or operator shall design and operate each waste management unit that comprises the wastewater treatment system in accordance with the appropriate standards specified in §§61.343 through 61.347 of this subpart.

(2) The provisions of paragraph (b)(1) of this section do not apply to any waste management unit that the owner or operator demonstrates to meet the following conditions initially and, thereafter, at least once per year:

(i) The benzene content of each waste stream entering the waste management unit is less than 10 ppmw on a flow-weighted annual average basis as determined by the procedures specified in §61.355(c) of this subpart; and

(ii) The total annual benzene quantity contained in all waste streams managed or treated in exempt waste management units comprising the facility wastewater treatment systems is less than 1 Mg/yr (1.1 ton/yr). For this determination, total annual benzene quantity shall be calculated as follows:

(A) The total annual benzene quantity shall be calculated as the sum of the individual benzene quantities determined at each location where a waste stream first enters an exempt waste management unit. The benzene quantity discharged from an exempt waste management unit shall not be included in this calculation.

(B) The annual benzene quantity in a waste stream managed or treated in an enhanced biodegradation unit shall not be included in the calculation of the total annual benzene quantity, if the enhanced biodegradation unit is the first exempt unit in which the waste is managed or treated. A unit shall be considered enhanced biodegradation if it is a suspended-growth process that generates biomass, uses recycled biomass, and periodically removes biomass from the process. An enhanced biodegradation unit typically operates at a food-to-microorganism ratio in the range of 0.05 to 1.0 kg of biological oxygen demand per kg of biomass per day, a mixed liquor suspended solids ratio in the range of 1 to 8 grams per liter (0.008 to 0.7 pounds per liter), and a residence time in the range of 3 to

36 hours.

(c) The owner and operator shall demonstrate that each treatment process or wastewater treatment system unit, except as provided in paragraph (d) of this section, achieves the appropriate conditions specified in paragraphs (a) or (b) of this section in accordance with the following requirements:

(1) Engineering calculations in accordance with requirements specified in §61.356(e) of this subpart; or

(2) Performance tests conducted using the test methods and procedures that meet the requirements specified in §61.355 of this subpart.

(d) A treatment process or waste stream is in compliance with the requirements of this subpart and exempt from the requirements of paragraph (c) of this section provided that the owner or operator documents that the treatment process or waste stream is in compliance with other regulatory requirements as follows:

(1) The treatment process is a hazardous waste incinerator for which the owner or operator has been issued a final permit under 40 CFR part 270 and complies with the requirements of 40 CFR part 264, subpart O;

(2) The treatment process is an industrial furnace or boiler burning hazardous waste for energy recovery for which the owner or operator has been issued a final permit under 40 CFR part 270 and complies with the requirements of 40 CFR part 266, subpart D;

(3) The waste stream is treated by a means or to a level that meets benzene-specific treatment standards in accordance with the Land Disposal Restrictions under 40 CFR part 268, and the treatment process is designed and operated with a closed-vent system and control device meeting the requirements of §61.349 of this subpart;

(4) The waste stream is treated by a means or to a level that meets benzene-specific effluent limitations or performance standards in accordance with the Effluent Guidelines and Standards under 40 CFR parts 401–464, and the treatment process is designed and operated with a closed-vent system and control device meeting the requirements of §61.349 of this subpart; or

(5) The waste stream is discharged to an underground injection well for which the owner or operator has been issued a final permit under 40 CFR part 270 and complies with the requirements of 40 CFR part 122.

(e) Except as specified in paragraph (e)(3) of this section, if the treatment process or wastewater treatment system unit has any openings (e.g., access doors, hatches, etc.), all such openings shall be sealed (e.g., gasketed, latched, etc.) and kept closed at all times when waste is being treated, except during inspection and maintenance.

(1) Each seal, access door, and all other openings shall be checked by visual inspections initially and quarterly thereafter to ensure that no cracks or gaps occur and that openings are closed and gasketed properly.

(2) Except as provided in §61.350 of this subpart, when a broken seal or gasket or other problem is identified, first efforts at repair shall be made as soon as practicable, but not later than 15 calendar days after identification.

(3) If the cover and closed-vent system operate such that the treatment process and wastewater treatment system unit are maintained at a pressure less than atmospheric pressure, the owner or operator may operate the system with an opening that is not sealed and kept closed at all times if the following conditions are met:

(i) The purpose of the opening is to provide dilution air to reduce the explosion hazard;

(ii) The opening is designed to operate with no detectable emissions as indicated by an instrument reading of less than 500 ppmv above background, as determined initially and thereafter at least once per year by the methods specified in §61.355(h); and

(iii) The pressure is monitored continuously to ensure that the pressure in the treatment process and wastewater treatment system unit remain below atmospheric pressure.

(f) Except for treatment processes complying with paragraph (d) of this section, the Administrator may request at any time an owner or operator demonstrate that a treatment process or wastewater treatment system unit meets the applicable requirements specified in paragraphs (a) or (b) of this section by conducting a performance test using the test methods and procedures as required in §61.355 of this subpart.

(g) The owner or operator of a treatment process or wastewater treatment system unit that is used to comply with the provisions of this section shall monitor the unit in accordance with the applicable requirements in §61.354 of this subpart.

[55 FR 8346, Mar. 7, 1990, as amended at 55 FR 37231, Sept. 10, 1990; 58 FR 3098, Jan. 7, 1993; 65 FR 62160, Oct. 17, 2000]

### § 61.349 Standards: Closed-ventsystems and control devices.

(a) For each closed-vent system and control device used to comply with standards in accordance with §§61.343 through 61.348 of this subpart, the owner or operator shall properly design, install, operate, and maintain the closed-vent system and control device in accordance with the following requirements:

(1) The closed-vent system shall:

(i) Be designed to operate with no detectable emissions as indicated by an instrument reading of less than 500 ppmv above background, as determined initially and thereafter at least once per year by the methods specified in §61.355(h) of this subpart.

(ii) Vent systems that contain any bypass line that could divert the vent stream away from a control device used to comply with the provisions of this subpart shall install, maintain, and operate according to the manufacturer's specifications a flow indicator that provides a record of vent stream flow away from the control device at least once every 15 minutes, except as provided in paragraph (a)(1)(ii)(B) of this section.

(A) The flow indicator shall be installed at the entrance to any bypass line that could divert the vent stream away from the control device to the atmosphere.

(B) Where the bypass line valve is secured in the closed position with a car-seal or a lock-and-key type configuration, a flow indicator is not required.

(iii) All gauging and sampling devices shall be gas-tight except when gauging or sampling is taking place.

(iv) For each closed-vent system complying with paragraph (a) of this section, one or more devices which vent directly to the atmosphere may be used on the closed-vent system provided each device remains in a closed, sealed position during normal operations except when the device needs to open to prevent physical damage or permanent deformation of the closed-vent system resulting from malfunction of the unit in accordance with good engineering and safety practices for handling flammable, explosive, or other hazardous materials.

(2) The control device shall be designed and operated in accordance with the following conditions:

(i) An enclosed combustion device (e.g., a vapor incinerator, boiler, or process heater) shall meet one of the following conditions:

(A) Reduce the organic emissions vented to it by 95 weight percent or greater;

(B) Achieve a total organic compound concentration of 20 ppmv (as the sum of the concentrations for individual compounds using Method 18) on a dry basis corrected to 3 percent oxygen; or

(C) Provide a minimum residence time of 0.5 seconds at a minimum temperature of 760 °C (1,400 °F). If a boiler or process heater issued as the control device, then the vent stream shall be introduced into the flame zone of the boiler or process heater.

(ii) A vapor recovery system (e.g., a carbon adsorption system or a condenser) shall recover or control the organic emissions vented to it with an efficiency of 95 weight percent or greater, or shall recover or control the benzene emissions vented to it with an efficiency of 98 weight percent or greater.

(iii) A flare shall comply with the requirements of 40 CFR 60.18.

(iv) A control device other than those described in paragraphs (a)(2) (i) through (iii) of this section may be used provided that the following conditions are met:

(A) The device shall recover or control the organic emissions vented to it with an efficiency of 95 weight percent or greater, or shall recover or control the benzene emissions vented to it with an efficiency of 98 weight percent or greater.

(B) The owner or operator shall develop test data and design information that documents the control device will achieve an emission control efficiency of either 95 percent or greater for organic compounds or 98 percent or greater for benzene.

(C) The owner or operator shall identify:

(1) The critical operating parameters that affect the emission control performance of the device;

(2) The range of values of these operating parameters that ensure the emission control efficiency specified in paragraph (a)(2)(iv)(A) of this section is maintained during operation of the device; and

(3) How these operating parameters will be monitored to ensure the proper operation and maintenance of the device.

(D) The owner or operator shall submit the information and data specified in paragraphs (a)(2)(iv) (B) and (C) of this section to the Administrator prior to operation of the alternative control device.

(E) The Administrator will determine, based on the information submitted under paragraph (a)(2)(iv)(D) of this section, if the control device subject to paragraph (a)(2)(iv) of this section meets the requirements of §61.349. The control device subject to paragraph (a)(2)(iv) of this section meets the requirements of §61.349. The control device subject to paragraph (a)(2)(iv) of this section may be operated prior to receiving approval from the Administrator. However, if the Administrator determines that the control device does not meet the requirements of §61.349, the facility may be subject to enforcement action beginning from the time the control device began operation.

(b) Each closed-vent system and control device used to comply with this subpart shall be operated at all times when waste is placed in the waste management unit vented to the control device except when maintenance or repair of the waste management unit cannot be completed without a shutdown of the control device.

(c) An owner and operator shall demonstrate that each control device, except for a flare, achieves the appropriate conditions specified in paragraph (a)(2) of this section by using one of the following methods:

(1) Engineering calculations in accordance with requirements specified in §61.356(f) of this subpart; or

(2) Performance tests conducted using the test methods and procedures that meet the requirements specified in §61.355 of this subpart.

(d) An owner or operator shall demonstrate compliance of each flare in accordance with paragraph (a)(2)(iii) of this section.

(e) The Administrator may request at any time an owner or operator demonstrate that a control device meets the applicable conditions specified in paragraph (a)(2) of this section by conducting a performance test using the test methods and procedures as required in §61.355, and for control devices subject to paragraph (a)(2)(iv) of this section, the Administrator may specify alternative test methods and procedures, as appropriate.

(f) Each closed-vent system and control device shall be visually inspected initially and quarterly thereafter. The visual inspection shall include inspection of ductwork and piping and connections to covers and control devices for evidence of visable defects such as holes in ductwork or piping and loose connections.

(g) Except as provided in §61.350 of this subpart, if visible defects are observed during an inspection, or if other problems are identified, or if detectable emissions are measured, a first effort to repair the closed-vent system and control device shall be made as soon as practicable but no later than 5 calendar days after detection. Repair shall be completed no later than 15 calendar days after the emissions are detected or the visible defect is observed.

(h) The owner or operator of a control device that is used to comply with the provisions of this section shall monitor the control device in accordance with §61.354(c) of this subpart.

[55 FR 8346, Mar. 7, 1990; 55 FR 12444, Apr. 3, 1990, as amended at 55 FR 37231, Sept. 10, 1990; 58 FR 3098, Jan. 7, 1993; 65 FR 62160, Oct. 17, 2000]

## § 61.350 Standards: Delay of repair.

(a) Delay of repair of facilities or units that are subject to the provisions of this subpart will be allowed if the repair is technically impossible without a complete or partial facility or unit shutdown.

(b) Repair of such equipment shall occur before the end of the next facility or unit shutdown.

#### § 61.351 Alternative standards for tanks.

(a) As an alternative to the standards for tanks specified in §61.343 of this subpart, an owner or operator may elect to comply with one of the following:

(1) A fixed roof and internal floating roof meeting the requirements in 40 CFR 60.112b(a)(1);

(2) An external floating roof meeting the requirements of 40 CFR 60.112b (a)(2); or

(3) An alternative means of emission limitation as described in 40 CFR 60.114b.

(b) If an owner or operator elects to comply with the provisions of this section, then the owner or operator is exempt from the provisions of §61.343 of this subpart applicable to the same facilities.

[55 FR 8346, Mar. 7, 1990, as amended at 55 FR 37231, Sept. 10, 1990]

#### § 61.352 Alternative standards for oil-water separators.

(a) As an alternative to the standards for oil-water separators specified in §61.347 of this subpart, an owner or operator may elect to comply with one of the following:

(1) A floating roof meeting the requirements in 40 CFR 60.693-2(a); or

(2) An alternative means of emission limitation as described in 40 CFR 60.694.

(b) For portions of the oil-water separator where it is infeasible to construct and operate a floating roof, such as over the weir mechanism, a fixed roof vented to a vapor control device that meets the requirements in §§61.347 and 61.349 of this subpart shall be installed and operated.

(c) Except as provided in paragraph (b) of this section, if an owner or operator elects to comply with the provisions of this section, then the owner or operator is exempt from the provisions in §61.347 of this subpart applicable to the same facilities.

### § 61.353 Alternative means of emission limitation.

(a) If, in the Administrator's judgment, an alternative means of emission limitation will achieve a reduction in benzene emissions at least equivalent to the reduction in benzene emissions from the source achieved by the applicable design, equipment, work practice, or operational requirements in §§61.342 through 61.349, the Administrator will publish in theFederal Registera notice permitting the use of the alternative means for purposes of compliance with that requirement. The notice may condition the permission on requirements related to the operation and maintenance of the alternative means.

(b) Any notice under paragraph (a) of this section shall be published only after public notice and an opportunity for a hearing.

(c) Any person seeking permission under this section shall collect, verify, and submit to the Administrator information showing that the alternative means achieves equivalent emission reductions.

[55 FR 8346, Mar. 7, 1990, as amended at 58 FR 3099, Jan. 7, 1993]

### § 61.354 Monitoring of operations.

(a) Except for a treatment process or waste stream complying with §61.348(d), the owner or operator shall monitor each treatment process or wastewater treatment system unit to ensure the unit is properly operated and maintained by one of the following monitoring procedures:

(1) Measure the benzene concentration of the waste stream exiting the treatment process complying with §61.348(a)(1)(i) at least once per month by collecting and analyzing one or more samples using the procedures specified in §61.355(c)(3).
(2) Install, calibrate, operate, and maintain according to manufacturer's specifications equipment to continuously monitor and record a process parameter (or parameters) for the treatment process or wastewater treatment system unit that indicates proper system operation. The owner or operator shall inspect at least once each operating day the data recorded by the monitoring equipment (e.g., temperature monitor or flow indicator) to ensure that the unit is operating properly.

(b) If an owner or operator complies with the requirements of §61.348(b), then the owner or operator shall monitor each wastewater treatment system to ensure the unit is properly operated and maintained by the appropriate monitoring procedure as follows:

(1) For the first exempt waste management unit in each waste treatment train, other than an enhanced biodegradation unit, measure the flow rate, using the procedures of §61.355(b), and the benzene concentration of each waste stream entering the unit at least once per month by collecting and analyzing one or more samples using the procedures specified in §61.355(c)(3).

(2) For each enhanced biodegradation unit that is the first exempt waste management unit in a treatment train, measure the benzene concentration of each waste stream entering the unit at least once per month by collecting and analyzing one or more samples using the procedures specified in §61.355(c)(3).

(c) An owner or operator subject to the requirements in §61.349 of this subpart shall install, calibrate, maintain, and operate according to the manufacturer's specifications a device to continuously monitor the control device operation as specified in the following paragraphs, unless alternative monitoring procedures or requirements are approved for that facility by the Administrator. The owner or operator shall inspect at least once each operating day the data recorded by the monitoring equipment (e.g., temperature monitor or flow indicator) to ensure that the control device is operating properly.

(1) For a thermal vapor incinerator, a temperature monitoring device equipped with a continuous recorder. The device shall have an accuracy of  $\pm 1$  percent of the temperature being monitored in °C or  $\pm 0.5$  °C, whichever is greater. The temperature sensor shall be installed at a representative location in the combustion chamber.

(2) For a catalytic vapor incinerator, a temperature monitoring device equipped with a continuous recorder. The device shall be capable of monitoring temperature at two locations, and have an accuracy of  $\pm 1$  percent of the temperature being monitored in °C or  $\pm 0.5$  °C, whichever is greater. One temperature sensor shall be installed in the vent stream at the nearest feasible point to the catalyst bed inlet and a second temperature sensor shall be installed in the vent stream at the nearest feasible point to the catalyst bed outlet.

(3) For a flare, a monitoring device in accordance with 40 CFR 60.18(f)(2) equipped with a continuous recorder.

(4) For a boiler or process heater having a design heat input capacity less than 44 MW ( $150 \times 10^6$  BTU/hr), a temperature monitoring device equipped with a continuous recorder. The device shall have an accuracy of ±1 percent of the temperature being monitored in °C or ±0.5 °C, whichever is greater. The temperature sensor shall be installed at a representative location in the combustion chamber.

(5) For a boiler or process heater having a design heat input capacity greater than or equal to 44 MW ( $150 \times 10^6$  BTU/hr), a monitoring device equipped with a continuous recorder to measure a parameter(s) that indicates good combustion operating practices are being used.

(6) For a condenser, either:

(i) A monitoring device equipped with a continuous recorder to measure either the concentration level of the organic compounds or the concentration level of benzene in the exhaust vent stream from the condenser; or

(ii) A temperature monitoring device equipped with a continuous recorder. The device shall be capable of monitoring temperature at two locations, and have an accuracy of  $\pm 1$  percent of the temperature being monitored in °C or  $\pm 0.5$  °C, whichever is greater. One temperature sensor shall be installed at a location in the exhaust stream from the condenser, and a second temperature sensor shall be installed at a location in the condenser.

(7) For a carbon adsorption system that regenerates the carbon bed directly in the control device such as a fixed-bed carbon adsorber, either:

(i) A monitoring device equipped with a continuous recorder to measure either the concentration level of the organic compounds or the benzene concentration level in the exhaust vent stream from the carbon bed; or

(ii) A monitoring device equipped with a continuous recorder to measure a parameter that indicates the carbon bed is regenerated on a regular, predetermined time cycle.

(8) For a vapor recovery system other than a condenser or carbon adsorption system, a monitoring device equipped with a continuous recorder to measure either the concentration level of the organic compounds or the benzene concentration level in the exhaust vent stream from the control device.

(9) For a control device subject to the requirements of §61.349(a)(2)(iv), devices to monitor the parameters as specified in §61.349(a)(2)(iv)(C).

(d) For a carbon adsorption system that does not regenerate the carbon bed directly on site in the control device (e.g., a carbon canister), either the concentration level of the organic compounds or the concentration level of benzene in the exhaust vent stream from the carbon adsorption system shall be monitored on a regular schedule, and the existing carbon shall be replaced with fresh carbon immediately when carbon breakthrough is indicated. The device shall be monitored on a daily basis or at intervals no greater than 20 percent of the design carbon replacement interval, whichever is greater. As an alternative to conducting this monitoring, an owner or operator may replace the carbon in the carbon adsorption system with fresh carbon at a regular predetermined time interval that is less than the carbon replacement interval that is determined by the maximum design flow rate and either the organic concentration or the benzene concentration in the gas stream vented to the carbon adsorption system.

(e) An alternative operation or process parameter may be monitored if it can be demonstrated that another parameter will ensure that the control device is operated in conformance with these standards and the control device's design specifications.

(f) Owners or operators using a closed-vent system that contains any bypass line that could divert a vent stream from a control device used to comply with the provisions of this subpart shall do the following:

(1) Visually inspect the bypass line valve at least once every month, checking the position of the valve and the condition of the carseal or closure mechanism required under §61.349(a)(1)(ii) to ensure that the valve is maintained in the closed position and the vent stream is not diverted through the bypass line.

(2) Visually inspect the readings from each flow monitoring device required by §61.349(a)(1)(ii) at least once each operating day to check that vapors are being routed to the control device as required.

(g) Each owner or operator who uses a system for emission control that is maintained at a pressure less than atmospheric pressure with openings to provide dilution air shall install, calibrate, maintain, and operate according to the manufacturer's specifications a device equipped with a continuous recorder to monitor the pressure in the unit to ensure that it is less than atmospheric pressure.

[55 FR 8346, Mar. 7, 1990, as amended at 58 FR 3099, Jan. 7, 1993; 65 FR 62160, Oct. 17, 2000]

# § 61.355 Test methods, procedures, and compliance provisions.

(a) An owner or operator shall determine the total annual benzene quantity from facility waste by the following procedure:

(1) For each waste stream subject to this subpart having a flow-weighted annual average water content greater than 10 percent water, on a volume basis as total water, or is mixed with water or other wastes at any time and the resulting mixture has an annual average water content greater than 10 percent as specified in §61.342(a), the owner or operator shall:

(i) Determine the annual waste quantity for each waste stream using the procedures specified in paragraph (b) of this section.

(ii) Determine the flow-weighted annual average benzene concentration for each waste stream using the procedures specified in paragraph (c) of this section.

(iii) Calculate the annual benzene quantity for each waste stream by multiplying the annual waste quantity of the waste stream times the flow-weighted annual average benzene concentration.

(2) Total annual benzene quantity from facility waste is calculated by adding together the annual benzene quantity for each waste stream generated during the year and the annual benzene quantity for each process unit turnaround waste annualized according to paragraph (b)(4) of this section.

(3) If the total annual benzene quantity from facility waste is equal to or greater than 10 Mg/yr (11 ton/yr), then the owner or operator shall comply with the requirements of §61.342 (c), (d), or (e).

(4) If the total annual benzene quantity from facility waste is less than 10 Mg/yr (11 ton/yr) but is equal to or greater than 1 Mg/yr (1.1 ton/yr), then the owner or operator shall:

(i) Comply with the recordkeeping requirements of §61.356 and reporting requirements of §61.357 of this subpart; and

(ii) Repeat the determination of total annual benzene quantity from facility waste at least once per year and whenever there is a change in the process generating the waste that could cause the total annual benzene quantity from facility waste to increase to 10 Mg/yr (11 ton/yr) or more.

(5) If the total annual benzene quantity from facility waste is less than 1 Mg/yr (1.1 ton/yr), then the owner or operator shall:

(i) Comply with the recordkeeping requirements of §61.356 and reporting requirements of §61.357 of this subpart; and

(ii) Repeat the determination of total annual benzene quantity from facility waste whenever there is a change in the process generating the waste that could cause the total annual benzene quantity from facility waste to increase to 1 Mg/yr (1.1 ton/yr) or more.

(6) The benzene quantity in a waste stream that is generated less than one time per year, except as provided for process unit turnaround waste in paragraph (b)(4) of this section, shall be included in the determination of total annual benzene quantity from facility waste for the year in which the waste is generated unless the waste stream is otherwise excluded from the determination of total annual benzene quantity from facility waste in accordance with paragraphs (a) through (c) of this section. The benzene quantity in this waste stream shall not be annualized or averaged over the time interval between the activities that resulted in generation of the waste, for purposes of determining the total annual benzene quantity from facility waste.

(b) For purposes of the calculation required by paragraph (a) of this section, an owner or operator shall determine the annual waste quantity at the point of waste generation, unless otherwise provided in paragraphs (b) (1), (2), (3), and (4) of this section, by one of the methods given in paragraphs (b) (5) through (7) of this section.

(1) The determination of annual waste quantity for sour water streams that are processed in sour water strippers shall be made at the point that the water exits the sour water stripper.

(2) The determination of annual waste quantity for wastes at coke by-product plants subject to and complying with the control requirements of §61.132, 61.133, 61.134, or 61.139 of subpart L of this part shall be made at the location that the waste stream exits the process unit component or waste management unit controlled by that subpart or at the exit of the ammonia still, provided that the following conditions are met:

(i) The transfer of wastes between units complying with the control requirements of subpart L of this part, process units, and the ammonia still is made through hard piping or other enclosed system.

(ii) The ammonia still meets the definition of a sour water stripper in §61.341.

(3) The determination of annual waste quantity for wastes that are received at hazardous waste treatment, storage, or disposal facilities from offsite shall be made at the point where the waste enters the hazardous waste treatment, storage, or disposal facility.

(4) The determination of annual waste quantity for each process unit turnaround waste generated only at 2 year or greater intervals, may be made by dividing the total quantity of waste generated during the most recent process unit turnaround by the time period (in the nearest tenth of a year) between the turnaround resulting in generation of the waste and the most recent preceding process turnaround for the unit. The resulting annual waste quantity shall be included in the calculation of the annual benzene quantity as provided in paragraph (a)(1)(iii) of this section for the year in which the turnaround occurs and for each subsequent year until the unit undergoes the next process turnaround. For estimates of total annual benzene quantity as specified in the 90-day report, required under §61.357(a)(1), the owner or operator shall estimate the waste quantity generated during the most recent turnaround, and the time period between turnaround waste, as specified in this paragraph, then the process unit turnaround waste quantity shall be included in the calculation of the annual benzene quantity for the year in which the turnaround occurs.

(5) Select the highest annual quantity of waste managed from historical records representing the most recent 5 years of operation or, if the facility has been in service for less than 5 years but at least 1 year, from historical records representing the total operating life of the facility;

(6) Use the maximum design capacity of the waste management unit; or

(7) Use measurements that are representative of maximum waste generation rates.

(c) For the purposes of the calculation required by §§61.355(a) of this subpart, an owner or operator shall determine the flow-

weighted annual average ben- zene concentration in a manner that meets the requirements given in paragraph (c)(1) of this section using either of the methods given in paragraphs (c)(2) and (c)(3) of this section.

(1) The determination of flow-weighted annual average benzene concentration shall meet all of the following criteria:

(i) The determination shall be made at the point of waste generation except for the specific cases given in paragraphs (c)(1)(i)(A) through (D) of this section.

(A) The determination for sour water streams that are processed in sour water strippers shall be made at the point that the water exits the sour water stripper.

(B) The determination for wastes at coke by-product plants subject to and complying with the control requirements of §61.132, 61.133, 61.134, or 61.139 of subpart L of this part shall be made at the location that the waste stream exits the process unit component or waste management unit controlled by that subpart or at the exit of the ammonia still, provided that the following conditions are met:

(1) The transfer of wastes between units complying with the control requirements of subpart L of this part, process units, and the ammonia still is made through hard piping or other enclosed system.

(2) The ammonia still meets the definition of a sour water stripper in §61.341.

(C) The determination for wastes that are received from offsite shall be made at the point where the waste enters the hazardous waste treatment, storage, or disposal facility.

(D) The determination of flow-weighted annual average benzene concentration for process unit turnaround waste shall be made using either of the methods given in paragraph (c)(2) or (c)(3) of this section. The resulting flow-weighted annual average benzene concentration shall be included in the calculation of annual benzene quantity as provided in paragraph (a)(1)(iii) of this section for the year in which the turnaround occurs and for each subsequent year until the unit undergoes the next process unit turnaround.

(ii) Volatilization of the benzene by exposure to air shall not be used in the determination to reduce the benzene concentration.

(iii) Mixing or diluting the waste stream with other wastes or other materials shall not be used in the determination—to reduce the benzene concentration.

(iv) The determination shall be made prior to any treatment of the waste that removes benzene, except as specified in paragraphs (c)(1)(i)(A) through (D) of this section.

(v) For wastes with multiple phases, the determination shall provide the weighted-average benzene concentration based on the benzene concentration in each phase of the waste and the relative proportion of the phases.

(2) Knowledge of the waste. The owner or operator shall provide sufficient information to document the flow-weighted annual average benzene concentration of each waste stream. Examples of information that could constitute knowledge include material balances, records of chemicals purchases, or previous test results provided the results are still relevant to the current waste stream conditions. If test data are used, then the owner or operator shall provide documentation describing the testing protocol and the means by which sampling variability and analytical variability were accounted for in the determination of the flow-weighted annual average benzene concentration for the waste stream. When an owner or operator and the Administrator do not agree on determinations of the flow-weighted annual average benzene concentration based on knowledge of the waste, the procedures under paragraph (c)(3) of this section shall be used to resolve the disagreement.

(3) Measurements of the benzene concentration in the waste stream in accordance with the following procedures:

(i) Collect a minimum of three representative samples from each waste stream. Where feasible, samples shall be taken from an enclosed pipe prior to the waste being exposed to the atmosphere.

(ii) For waste in enclosed pipes, the following procedures shall be used:

(A) Samples shall be collected prior to the waste being exposed to the atmosphere in order to minimize the loss of benzene prior to sampling.

(B) A static mixer shall be installed in the process line or in a by-pass line unless the owner or operator demonstrates that installation of a static mixer in the line is not necessary to accurately determine the benzene concentration of the waste stream.

(C) The sampling tap shall be located within two pipe diameters of the static mixer outlet.

(D) Prior to the initiation of sampling, sample lines and cooling coil shall be purged with at least four volumes of waste.

(E) After purging, the sample flow shall be directed to a sample container and the tip of the sampling tube shall be kept below the surface of the waste during sampling to minimize contact with the atmosphere.

(F) Samples shall be collected at a flow rate such that the cooling coil is able to maintain a waste temperature less than 10 °C (50 °F).

(G) After filling, the sample container shall be capped immediately (within 5 seconds) to leave a minimum headspace in the container.

(H) The sample containers shall immediately be cooled and maintained at a temperature below 10 °C (50 °F) for transfer to the laboratory.

(iii) When sampling from an enclosed pipe is not feasible, a minimum of three representative samples shall be collected in a manner to minimize exposure of the sample to the atmosphere and loss of benzene prior to sampling.

(iv) Each waste sample shall be analyzed using one of the following test methods for determining the benzene concentration in a waste stream:

(A) Method 8020, Aromatic Volatile Organics, in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA Publication No. SW-846 (incorporation by reference as specified in §61.18 of this part);

(B) Method 8021, Volatile Organic Compounds in Water by Purge and Trap Capillary Column Gas Chromatography with Photoionization and Electrolytic Conductivity Detectors in Series in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA Publication No. SW–846 (incorporation by reference as specified in §61.18 of this part);

(C) Method 8240, Gas Chromatography/Mass Spectrometry for Volatile Organics in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA Publication No. SW–846 (incorporation by reference as specified in §61.18 of this part);

(D) Method 8260, Gas Chromatography/Mass Spectrometry for Volatile Organics: Capillary Column Technique in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA Publication No. SW-846 (incorporation by reference as specified in §61.18 of this part);

(E) Method 602, Purgeable Aromatics, as described in 40 CFR part 136, appendix A, Test Procedures for Analysis of Organic Pollutants, for wastewaters for which this is an approved EPA methods; or

(F) Method 624, Purgeables, as described in 40 CFR part 136, appendix A, Test Procedures for Analysis of Organic Pollutants, for wastewaters for which this is an approved EPA method.

(v) The flow-weighted annual average benzene concentration shall be calculated by averaging the results of the sample analyses as follows:

$$\overline{C} = \frac{1}{Q_i} \times \sum_{i=1}^{n} (Q_i) (C_i)$$

Where:

C=Flow-weighted annual average benzene concentration for waste stream, ppmw.

Qt=Total annual waste quantity for waste stream, kg/yr (lb/yr).

n=Number of waste samples (at least 3).

Q<sub>i</sub>=Annual waste quantity for waste stream represented by C<sub>i</sub>, kg/yr (lb/yr).

C<sub>i</sub>=Measured concentration of benzene in waste sample i, ppmw.

(d) An owner or operator using performance tests to demonstrate compliance of a treatment process with §61.348 (a)(1)(i) shall measure the flow-weighted annual average benzene concentration of the waste stream exiting the treatment process by collecting and analyzing a minimum of three representative samples of the waste stream using the procedures in paragraph (c)(3) of this section. The test shall be conducted under conditions that exist when the treatment process is operating at the highest inlet waste stream flow rate and benzene content expected to occur. Operations during periods of startup, shutdown, and malfunction shall not constitute representative conditions for the purpose of a test. The owner or operator shall record all process information as is necessary to document the operating conditions during the test.

(e) An owner or operator using performance tests to demonstrate compliance of a treatment process with §61.348(a)(1)(ii) of this subpart shall determine the percent reduction of benzene in the waste stream on a mass basis by the following procedure:

(1) The test shall be conducted under conditions that exist when the treatment process is operating at the highest inlet waste stream flow rate and benzene content expected to occur. Operations during periods of startup, shutdown, and malfunction shall not constitute representative conditions for the purpose of a test. The owner or operator shall record all process information as is necessary to document the operating conditions during the test.

(2) All testing equipment shall be prepared and installed as specified in the appropriate test methods.

(3) The mass flow rate of benzene entering the treatment process ( $E_b$ ) shall be determined by computing the product of the flow rate of the waste stream entering the treatment process, as determined by the inlet flow meter, and the benzene concentration of the waste stream, as determined using the sampling and analytical procedures specified in paragraph (c)(2) or (c)(3) of this section. Three grab samples of the waste shall be taken at equally spaced time intervals over a 1-hour period. Each 1-hour period constitutes a run, and the performance test shall consist of a minimum of 3 runs conducted over a 3-hour period. The mass flow rate of benzene entering the treatment process is calculated as follows:

$$E_{b} = \frac{K}{n \times 10^{6}} \left[ \sum_{i=1}^{n} V_{i} C_{i} \right]$$

Where:

E<sub>b</sub>= Mass flow rate of benzene entering the treatment process, kg/hr (lb/hr).

K = Density of the waste stream,  $kg/m^3$  (lb/ft<sup>3</sup>).

V<sub>i</sub>= Average volume flow rate of waste entering the treatment process during each run i, m<sup>3</sup> /hr (ft<sup>3</sup> /hr).

C<sub>i</sub>= Average concentration of benzene in the waste stream entering the treatment process during each run i, ppmw.

n = Number of runs.

 $10^6$  = Conversion factor for ppmw.

<sup>(4)</sup> The mass flow rate of benzene exiting the treatment process (E<sub>a</sub>) shall be determined by computing the product of the flow rate of the waste stream exiting the treatment process, as determined by the outlet flow meter or the inlet flow meter, and the benzene concentration of the waste stream, as determined using the sampling and analytical procedures specified in paragraph (c)(2) or (c)(3) of this section. Three grab samples of the waste shall be taken at equally spaced time intervals over a 1-hour period. Each 1-hour period constitutes a run, and the performance test shall consist of a minimum of 3 runs conducted over the same 3-hour period at which the mass flow rate of benzene entering the treatment process is determined. The mass flow rate of benzene exiting the treatment process is calculated as follows:

$$\mathbf{E}_{\mathbf{a}} = \frac{\mathbf{K}}{\mathbf{n} \times 10^{6}} \left[ \sum_{i=1}^{n} \mathbf{V}_{i} \mathbf{C}_{i} \right]$$

Where:

E<sub>a</sub>= Mass flow rate of benzene exiting the treatment process, kg/hr (lb/hr).

K = Density of the waste stream,  $kg/m^3$  (lb/ft<sup>3</sup>).

V<sub>i</sub>= Average volume flow rate of waste exiting the treatment process during each run i, m<sup>3</sup> /hr (ft<sup>3</sup> /hr).

C<sub>i</sub>= Average concentration of benzene in the waste stream exiting the treatment process during each run i, ppmw.

n = Number of runs.

 $10^6$  = Conversion factor for ppmw.

(f) An owner or operator using performance tests to demonstrate compliance of a treatment process with §61.348(a)(1)(iii) of this subpart shall determine the benzene destruction efficiency for the combustion unit by the following procedure:

(1) The test shall be conducted under conditions that exist when the combustion unit is operating at the highest inlet waste stream flow rate and benzene content expected to occur. Operations during periods of startup, shutdown, and malfunction shall not constitute representative conditions for the purpose of a test. The owner or operator shall record all process information necessary to document the operating conditions during the test.

(2) All testing equipment shall be prepared and installed as specified in the appropriate test methods.

(3) The mass flow rate of benzene entering the combustion unit shall be determined by computing the product of the flow rate of the waste stream entering the combustion unit, as determined by the inlet flow meter, and the benzene concentration of the waste stream, as determined using the sampling procedures in paragraph (c)(2) or (c)(3) of this section. Three grab samples of the waste shall be taken at equally spaced time intervals over a 1-hour period. Each 1-hour period constitutes a run, and the performance test shall consist of a minimum of 3 runs conducted over a 3-hour period. The mass flow rate of benzene into the combustion unit is calculated as follows:

$$E_{b} = \frac{K}{n \times 10^{6}} \left[ \sum_{i=1}^{n} V_{i} C_{i} \right]$$

Where:

E<sub>b</sub>= Mass flow rate of benzene entering the combustion unit, kg/hr (lb/hr).

K = Density of the waste stream,  $kg/m^3$  (lb/ft<sup>3</sup>).

 $V_i$ = Average volume flow rate of waste entering the combustion unit during each run i, m<sup>3</sup> /hr (ft<sup>3</sup> /hr).

C<sub>i</sub>= Average concentration of benzene in the waste stream entering the combustion unit during each run i, ppmw.

n = Number of runs.

# $10^6$ = Conversion factor for ppmw.

(4) The mass flow rate of benzene exiting the combustion unit exhaust stack shall be determined as follows:

(i) The time period for the test shall not be less than 3 hours during which at least 3 stack gas samples are collected and be the same time period at which the mass flow rate of benzene entering the treatment process is determined. Each sample shall be collected over a 1-hour period (e.g., in a tedlar bag) to represent a time-integrated composite sample and each 1-hour period shall correspond to the periods when the waste feed is sampled.

(ii) A run shall consist of a 1-hour period during the test. For each run:

(A) The reading from each measurement shall be recorded;

(B) The volume exhausted shall be determined using Method 2, 2A, 2C, or 2D from appendix A of 40 CFR part 60, as appropriate.

(C) The average benzene concentration in the exhaust downstream of the combustion unit shall be determined using Method 18 from appendix A of 40 CFR part 60.

(iii) The mass of benzene emitted during each run shall be calculated as follows:

$$M_i = D_b VC(10^{-6})$$

Where:

M<sub>i</sub>= Mass of benzene emitted during run i, kg (lb).

V = Volume of air-vapor mixture exhausted at standard conditions,  $m^3$  (ft<sup>3</sup>).

C = Concentration of benzene measured in the exhaust, ppmv.

 $D_{b}$ = Density of benzene, 3.24 kg/m<sup>3</sup> (0.202 lb/ft<sup>3</sup>).

 $10^6$  = Conversion factor for ppmv.

(iv) The benzene mass emission rate in the exhaust shall be calculated as follows:

$$E_a = \left(\sum_{i=1}^n \mathcal{M}_i\right) / T$$

Where:

E<sub>a</sub>= Mass flow rate of benzene emitted from the combustion unit, kg/hr (lb/hr).

- M<sub>i</sub>= Mass of benzene emitted from the combustion unit during run i, kg (lb).
- T = Total time of all runs, hr.

n = Number of runs.

(5) The benzene destruction efficiency for the combustion unit shall be calculated as follows:

$$R = \frac{E_{b} - E_{a}}{E_{b}} \times 100$$

Where:

R = Benzene destruction efficiency for the combustion unit, percent.

 $E_b$  = Mass flow rate of benzene entering the combustion unit, kg/hr (lb/hr).

 $E_a$  = Mass flow rate of benzene emitted from the combustion unit, kg/hr (lb/hr).

(g) An owner or operator using performance tests to demonstrate compliance of a wastewater treatment system unit with §61.348(b) shall measure the flow-weighted annual average benzene concentration of the wastewater stream where the waste stream enters an exempt waste management unit by collecting and analyzing a minimum of three representative samples of the waste stream using the procedures in paragraph (c)(3) of this section. The test shall be conducted under conditions that exist when the wastewater treatment system is operating at the highest inlet wastewater stream flow rate and benzene content expected to occur. Operations during periods of startup, shutdown, and malfunction shall not constitute representative conditions for the purpose of a test. The owner or operator shall record all process information as is necessary to document the operating conditions during the test.

(h) An owner or operator shall test equipment for compliance with no detectable emissions as required in §§61.343 through 61.347, and §61.349 of this subpart in accordance with the following requirements:

(1) Monitoring shall comply with Method 21 from appendix A of 40 CFR part 60.

(2) The detection instrument shall meet the performance criteria of Method 21.

(3) The instrument shall be calibrated before use on each day of its use by the procedures specified in Method 21.

(4) Calibration gases shall be:

(i) Zero air (less than 10 ppm of hydrocarbon in air); and

(ii) A mixture of methane or n-hexane and air at a concentration of approximately, but less than, 10,000 ppm methane or n-hexane.

(5) The background level shall be determined as set forth in Method 21.

(6) The instrument probe shall be traversed around all potential leak interfaces as close as possible to the interface as described in Method 21.

(7) The arithmetic difference between the maximum concentration indicated by the instrument and the background level is compared to 500 ppm for determining compliance.

(i) An owner or operator using a performance test to demonstrate compliance of a control device with either the organic reduction efficiency requirement or the benzene reduction efficiency requirement specified under §61.349(a)(2) shall use the following procedures:

(1) The test shall be conducted under conditions that exist when the waste management unit vented to the control device is operating at the highest load or capacity level expected to occur. Operations during periods of startup, shutdown, and malfunction shall not constitute representative conditions for the purpose of a test. The owner or operator shall record all process information necessary to document the operating conditions during the test.

(2) Sampling sites shall be selected using Method 1 or 1A from appendix A of 40 CFR part 60, as appropriate.

(3) The mass flow rate of either the organics or benzene entering and exiting the control device shall be determined as follows:

(i) The time period for the test shall not be less than 3 hours during which at least 3 stack gas samples are collected. Samples of the

vent stream entering and exiting the control device shall be collected during the same time period. Each sample shall be collected over a 1-hour period (e.g., in a tedlar bag) to represent a time-integrated composite sample.

(ii) A run shall consist of a 1-hour period during the test. For each run:

(A) The reading from each measurement shall be recorded;

(B) The volume exhausted shall be determined using Method 2, 2A, 2C, or 2D from appendix A of 40 CFR part 60, as appropriate;

(C) The organic concentration or the benzene concentration, as appropriate, in the vent stream entering and exiting the control shall be determined using Method 18 from appendix A of 40 CFR part 60.

(iii) The mass of organics or benzene entering and exiting the control device during each run shall be calculated as follows:

$$M_{aj} = \frac{K_l V_{aj}}{10^6} \left( \sum_{i=1}^n C_{ai} M W_i \right)$$
$$M_{bj} = \frac{K_l V_{bj}}{10^6} \left( \sum_{i=1}^n C_{bi} M W_i \right)$$

 $M_{ai}$  = Mass of organics or benzene in the vent stream entering the control device during run j, kg (lb).

M<sub>bi</sub>= Mass of organics or benzene in the vent stream exiting the control device during run j, kg (lb).

 $V_{ai}$  = Volume of vent stream entering the control device during run j, at standard conditions, m<sup>3</sup> (ft<sup>3</sup>).

 $V_{bi}$  = Volume of vent stream exiting the control device during run j, at standard conditions, m<sup>3</sup> (ft<sup>3</sup>).

C<sub>ai</sub>= Organic concentration of compound i or the benzene concentration measured in the vent stream entering the control device as determined by Method 18, ppm by volume on a dry basis.

C<sub>bi</sub>= Organic concentration of compound i or the benzene concentration measured in the vent stream exiting the control device as determined by Method 18, ppm by volume on a dry basis.

MW<sub>i</sub>= Molecular weight of organic compound i in the vent stream, or the molecular weight of benzene, kg/kg-mol (lb/lb-mole).

n = Number of organic compounds in the vent stream; if benzene reduction efficiency is being demonstrated, then n=1.

K<sub>1</sub>= Conversion factor for molar volume at standard conditions (293 K and 760 mm Hg (527 R and 14.7 psia))

 $= 0.0416 \text{ kg-mol/m}^3 (0.00118 \text{ lb-mol/ft}^3)$ 

 $10^{-6}$ =Conversion factor for ppmv.

(iv) The mass flow rate of organics or benzene entering and exiting the control device shall be calculated as follows:

$$E_{a} = \left(\sum_{j=1}^{n} M_{aj}\right) / T$$
$$E_{b} = \left(\sum_{j=1}^{n} M_{bj}\right) / T$$

Where:

E<sub>a</sub>= Mass flow rate of organics or benzene entering the control device, kg/hr (lb/hr).

 $E_{b}$ = Mass flow rate of organics or benzene exiting the control device, kg/hr (lb/hr).

M<sub>ai</sub>= Mass of organics or benzene in the vent stream entering the control device during run j, kg (lb).

M<sub>bj</sub>= Mass of organics or benzene in the vent stream exiting the control device during run j, kg (lb).

T = Total time of all runs, hr.

n = Number of runs.

(4) The organic reduction efficiency or the benzene reduction efficiency for the control device shall be calculated as follows:

$$R = \frac{E_a - E_b}{E_a} \times 100$$

Where:

R = Total organic reduction of efficiency or benzene reduction efficiency for the control device, percent.

E<sub>b</sub>= Mass flow rate of organics or benzene entering the control device, kg/hr (lb/hr).

E<sub>a</sub>= Mass flow rate of organic or benzene emitted from the control device, kg/hr (lb/hr).

(j) An owner or operator shall determine the benzene quantity for the purposes of the calculation required by §61.342 (c)(3)(ii)(B) according to the provisions of paragraph (a) of this section, except that the procedures in paragraph (a) of this section shall also apply to wastes with a water content of 10 percent or less.

(k) An owner or operator shall determine the benzene quantity for the purposes of the calculation required by §61.342(e)(2) by the following procedure:

(1) For each waste stream that is not controlled for air emissions in accordance with §61.343. 61.344, 61.345, 61.346, 61.347, or 61.348(a), as applicable to the waste management unit that manages the waste, the benzene quantity shall be determined as specified in paragraph (a) of this section, except that paragraph (b)(4) of this section shall not apply, i.e., the waste quantity for process unit turnaround waste is not annualized but shall be included in the determination of benzene quantity for the year in which the waste is generated for the purposes of the calculation required by §61.342(e)(2).

(2) For each waste stream that is controlled for air emissions in accordance with §61.343. 61.344, 61.345, 61.346, 61.347, or 61.348(a), as applicable to the waste management unit that manages the waste, the determination of annual waste quantity and flow-weighted annual average benzene concentration shall be made at the first applicable location as described in paragraphs (k)(2)(i), (k)(2)(ii), and (k)(2)(iii) of this section and prior to any reduction of benzene concentration through volatilization of the benzene, using

the methods given in (k)(2)(iv) and (k)(2)(v) of this section.

(i) Where the waste stream enters the first waste management unit not complying with §§61.343, 61.344, 61.345, 61.346, 61.347, and 61.348(a) that are applicable to the waste management unit,

(ii) For each waste stream that is managed or treated only in compliance with §§61.343 through 61.348(a) up to the point of final direct discharge from the facility, the determination of benzene quantity shall be prior to any reduction of benzene concentration through volatilization of the benzene, or

(iii) For wastes managed in units controlled for air emissions in accordance with §§61.343, 61.344, 61.345, 61.346, 61.347, and 61.348(a), and then transferred offsite, facilities shall use the first applicable offsite location as described in paragraphs (k)(2)(i) and (k)(2)(ii) of this section if they have documentation from the offsite facility of the benzene quantity at this location. Facilities without this documentation for offsite wastes shall use the benzene quantity determined at the point where the transferred waste leaves the facility.

(iv) Annual waste quantity shall be determined using the procedures in paragraphs (b)(5), (6), or (7) of this section, and

(v) The flow-weighted annual average benzene concentration shall be determined using the procedures in paragraphs (c)(2) or (3) of this section.

(3) The benzene quantity in a waste stream that is generated less than one time per year, including process unit turnaround waste, shall be included in the determination of benzene quantity as determined in paragraph (k)(6) of this section for the year in which the waste is generated. The benzene quantity in this waste stream shall not be annualized or averaged over the time interval between the activities that resulted in generation of the waste for purposes of determining benzene quantity as determined in paragraph (k)(6) of this section.

(4) The benzene in waste entering an enhanced biodegradation unit, as defined in  $\S61.348(b)(2)(ii)(B)$ , shall not be included in the determination of benzene quantity, determined in paragraph (k)(6) of this section, if the following conditions are met:

(i) The benzene concentration for each waste stream entering the enhanced biodegradation unit is less than 10 ppmw on a flowweighted annual average basis, and

(ii) All prior waste management units managing the waste comply with §§61.343, 61.344, 61.345, 61.346, 61.347 and 61.348(a).

(5) The benzene quantity for each waste stream in paragraph (k)(2) of this section shall be determined by multiplying the annual waste quantity of each waste stream times its flow-weighted annual average benzene concentration.

(6) The total benzene quantity for the purposes of the calculation required by  $\S61.342(e)(2)$  shall be determined by adding together the benzene quantities determined in paragraphs (k)(1) and (k)(5) of this section for each applicable waste stream.

(7) If the benzene quantity determined in paragraph (6) of this section exceeds 6.0 Mg/yr (6.6 ton/yr) only because of multiple counting of the benzene quantity for a waste stream, the owner or operator may use the following procedures for the purposes of the calculation required by §61.342(e)(2):

(i) Determine which waste management units are involved in the multiple counting of benzene;

(ii) Determine the quantity of benzene that is emitted, recovered, or removed from the affected units identified in paragraph (k)(7)(i) of this section, or destroyed in the units if applicable, using either direct measurements or the best available estimation techniques developed or approved by the Administrator.

(iii) Adjust the benzene quantity to eliminate the multiple counting of benzene based on the results from paragraph (k)(7)(ii) of this section and determine the total benzene quantity for the purposes of the calculation required by §61.342(e)(2).

(iv) Submit in the annual report required under §61.357(a) a description of the methods used and the resulting calculations for the alternative procedure under paragraph (k)(7) of this section, the benzene quantity determination from paragraph (k)(6) of this section, and the adjusted benzene quantity determination from paragraph (k)(7)(iii) of this section.

[55 FR 8346, Mar. 7, 1990; 55 FR 12444, Apr. 3, 1990, as amended at 55 FR 37231, Sept. 10, 1990; 58 FR 3099, Jan. 7, 1993; 65 FR 62160, Oct. 17, 2000]

# § 61.356 Recordkeeping requirements.

(a) Each owner or operator of a facility subject to the provisions of this subpart shall comply with the recordkeeping requirements of this section. Each record shall be maintained in a readily accessible location at the facility site for a period not less than two years from the date the information is recorded unless otherwise specified.

(b) Each owner or operator shall maintain records that identify each waste stream at the facility subject to this subpart, and indicate whether or not the waste stream is controlled for benzene emissions in accordance with this subpart. In addition the owner or operator shall maintain the following records:

(1) For each waste stream not controlled for benzene emissions in accordance with this subpart, the records shall include all test results, measurements, calculations, and other documentation used to determine the following information for the waste stream: waste stream identification, water content, whether or not the waste stream is a process wastewater stream, annual waste quantity, range of benzene concentrations, annual average flow-weighted benzene concentration, and annual benzene quantity.

(2) For each waste stream exempt from §61.342(c)(1) in accordance with §61.342(c)(3), the records shall include:

(i) All measurements, calculations, and other documentation used to determine that the continuous flow of process wastewater is less than 0.02 liters (0.005 gallons) per minute or the annual waste quantity of process wastewater is less than 10 Mg/yr (11 ton/yr) in accordance with §61.342(c)(3)(i), or

(ii) All measurements, calculations, and other documentation used to determine that the sum of the total annual benzene quantity in all exempt waste streams does not exceed 2.0 Mg/yr (2.2 ton/yr) in accordance with §61.342(c)(3)(ii).

(3) For each facility where process wastewater streams are controlled for benzene emissions in accordance with §61.342(d) of this subpart, the records shall include for each treated process wastewater stream all measurements, calculations, and other documentation used to determine the annual benzene quantity in the process wastewater stream exiting the treatment process.

(4) For each facility where waste streams are controlled for benzene emissions in accordance with §61.342(e), the records shall include for each waste stream all measurements, including the locations of the measurements, calculations, and other documentation used to determine that the total benzene quantity does not exceed 6.0 Mg/yr (6.6 ton/yr).

(5) For each facility where the annual waste quantity for process unit turnaround waste is determined in accordance with §61.355(b)(5), the records shall include all test results, measurements, calculations, and other documentation used to determine the following information: identification of each process unit at the facility that undergoes turnarounds, the date of the most recent turnaround for each process unit, identification of each process unit turnaround waste, the water content of each process unit turnaround waste, the annual waste quantity determined in accordance with §61.355(b)(5), the range of benzene concentrations in the waste, the annual average flow-weighted benzene concentration of the waste, and the annual benzene quantity calculated in accordance with §61.355(a)(1)(iii) of this section.

(6) For each facility where wastewater streams are controlled for benzene emissions in accordance with §61.348(b)(2), the records shall include all measurements, calculations, and other documentation used to determine the annual benzene content of the waste streams and the total annual benzene quantity contained in all waste streams managed or treated in exempt waste management units.

(c) An owner or operator transferring waste off-site to another facility for treatment in accordance with §61.342(f) shall maintain documentation for each offsite waste shipment that includes the following information: Date waste is shipped offsite, quantity of waste shipped offsite, name and address of the facility receiving the waste, and a copy of the notice sent with the waste shipment.

(d) An owner or operator using control equipment in accordance with §§61.343 through 61.347 shall maintain engineering design documentation for all control equipment that is installed on the waste management unit. The documentation shall be retained for the life of the control equipment. If a control device is used, then the owner or operator shall maintain the control device records required by paragraph (f) of this section.

(e) An owner or operator using a treatment process or wastewater treatment system unit in accordance with §61.348 of this subpart shall maintain the following records. The documentation shall be retained for the life of the unit.

(1) A statement signed and dated by the owner or operator certifying that the unit is designed to operate at the documented performance level when the waste stream entering the unit is at the highest waste stream flow rate and benzene content expected to occur.

(2) If engineering calculations are used to determine treatment process or wastewater treatment system unit performance, then the owner or operator shall maintain the complete design analysis for the unit. The design analysis shall include for example the following information: Design specifications, drawings, schematics, piping and instrumentation diagrams, and other documentation necessary to demonstrate the unit performance.

(3) If performance tests are used to determine treatment process or wastewater treatment system unit performance, then the owner or operator shall maintain all test information necessary to demonstrate the unit performance.

(i) A description of the unit including the following information: type of treatment process; manufacturer name and model number; and for each waste stream entering and exiting the unit, the waste stream type (e.g., process wastewater, sludge, slurry, etc.), and the design flow rate and benzene content.

(ii) Documentation describing the test protocol and the means by which sampling variability and analytical variability were accounted for in the determination of the unit performance. The description of the test protocol shall include the following information: sampling locations, sampling method, sampling frequency, and analytical procedures used for sample analysis.

(iii) Records of unit operating conditions during each test run including all key process parameters.

(iv) All test results.

(4) If a control device is used, then the owner or operator shall maintain the control device records required by paragraph (f) of this section.

(f) An owner or operator using a closed-vent system and control device in accordance with §61.349 of this subpart shall maintain the following records. The documentation shall be retained for the life of the control device.

(1) A statement signed and dated by the owner or operator certifying that the closed-vent system and control device is designed to operate at the documented performance level when the waste management unit vented to the control device is or would be operating at the highest load or capacity expected to occur.

(2) If engineering calculations are used to determine control device performance in accordance with §61.349(c), then a design analysis for the control device that includes for example:

(i) Specifications, drawings, schematics, and piping and instrumentation diagrams prepared by the owner or operator, or the control device manufacturer or vendor that describe the control device design based on acceptable engineering texts. The design analysis shall address the following vent stream characteristics and control device operating parameters:

(A) For a thermal vapor incinerator, the design analysis shall consider the vent stream composition, constituent concentrations, and flow rate. The design analysis shall also establish the design minimum and average temperature in the combustion zone and the combustion zone residence time.

(B) For a catalytic vapor incinerator, the design analysis shall consider the vent stream composition, constituent concentrations, and flow rate. The design analysis shall also establish the design minimum and average temperatures across the catalyst bed inlet and outlet.

(C) For a boiler or process heater, the design analysis shall consider the vent stream composition, constituent concentrations, and flow rate. The design analysis shall also establish the design minimum and average flame zone temperatures, combustion zone residence time, and description of method and location where the vent stream is introduced into the flame zone.

(D) For a flare, the design analysis shall consider the vent stream composition, constituent concentrations, and flow rate. The design analysis shall also consider the requirements specified in 40 CFR 60.18.

(E) For a condenser, the design analysis shall consider the vent stream composition, constituent concentration, flow rate, relative humidity, and temperature. The design analysis shall also establish the design outlet organic compound concentration level or the design outlet benzene concentration level, design average temperature of the condenser exhaust vent stream, and the design average temperatures of the coolant fluid at the condenser inlet and outlet.

(F) For a carbon adsorption system that regenerates the carbon bed directly on-site in the control device such as a fixed-bed adsorber, the design analysis shall consider the vent stream composition, constituent concentration, flow rate, relative humidity, and temperature. The design analysis shall also establish the design exhaust vent stream organic compound concentration level or the design exhaust vent stream benzene concentration level, number and capacity of carbon beds, type and working capacity of

activated carbon used for carbon beds, design total steam flow over the period of each complete carbon bed regeneration cycle, duration of the carbon bed steaming and cooling/drying cycles, design carbon bed temperature after regeneration, design carbon bed regeneration time, and design service life of carbon.

(G) For a carbon adsorption system that does not regenerate the carbon bed directly on-site in the control device, such as a carbon canister, the design analysis shall consider the vent stream composition, constituent concentration, flow rate, relative humidity, and temperature. The design analysis shall also establish the design exhaust vent stream organic compound concentration level or the design exhaust vent stream benzene concentration level, capacity of carbon bed, type and working capacity of activated carbon used for carbon bed, and design carbon replacement interval based on the total carbon working capacity of the control device and source operating schedule.

(H) For a control device subject to the requirements of §61.349(a)(2)(iv), the design analysis shall consider the vent stream composition, constituent concentration, and flow rate. The design analysis shall also include all of the information submitted under §61.349 (a)(2)(iv).

(ii) [Reserved]

(3) If performance tests are used to determine control device performance in accordance with §61.349(c) of this subpart:

(i) A description of how it is determined that the test is conducted when the waste management unit or treatment process is operating at the highest load or capacity level. This description shall include the estimated or design flow rate and organic content of each vent stream and definition of the acceptable operating ranges of key process and control parameters during the test program.

(ii) A description of the control device including the type of control device, control device manufacturer's name and model number, control device dimensions, capacity, and construction materials.

(iii) A detailed description of sampling and monitoring procedures, including sampling and monitoring locations in the system, the equipment to be used, sampling and monitoring frequency, and planned analytical procedures for sample analysis.

(iv) All test results.

(g) An owner or operator shall maintain a record for each visual inspection required by §§61.343 through 61.347 of this subpart that identifies a problem (such as a broken seal, gap or other problem) which could result in benzene emissions. The record shall include the date of the inspection, waste management unit and control equipment location where the problem is identified, a description of the problem, a description of the corrective action taken, and the date the corrective action was completed.

(h) An owner or operator shall maintain a record for each test of no detectable emissions required by §§61.343 through 61.347 and §61.349 of this subpart. The record shall include the following information: date the test is performed, background level measured during test, and maximum concentration indicated by the instrument reading measured for each potential leak interface. If detectable emissions are measured at a leak interface, then the record shall also include the waste management unit, control equipment, and leak interface location where detectable emissions were measured, a description of the problem, a description of the corrective action taken, and the date the corrective action was completed.

(i) For each treatment process and wastewater treatment system unit operated to comply with §61.348, the owner or operator shall maintain documentation that includes the following information regarding the unit operation:

(1) Dates of startup and shutdown of the unit.

(2) If measurements of waste stream benzene concentration are performed in accordance with §61.354(a)(1) of this subpart, the owner or operator shall maintain records that include date each test is performed and all test results.

(3) If a process parameter is continuously monitored in accordance with §61.354(a)(2) of this subpart, the owner or operator shall maintain records that include a description of the operating parameter (or parameters) to be monitored to ensure that the unit will be operated in conformance with these standards and the unit's design specifications, and an explanation of the criteria used for selection of that parameter (or parameters). This documentation shall be kept for the life of the unit.

(4) If measurements of waste stream benzene concentration are performed in accordance with §61.354(b), the owner or operator shall maintain records that include the date each test is performed and all test results.

(5) Periods when the unit is not operated as designed.

(j) For each control device, the owner or operator shall maintain documentation that includes the following information regarding the control device operation:

(1) Dates of startup and shutdown of the closed-vent system and control device.

(2) A description of the operating parameter (or parameters) to be monitored to ensure that the control device will be operated in conformance with these standards and the control device's design specifications and an explanation of the criteria used for selection of that parameter (or parameters). This documentation shall be kept for the life of the control device.

(3) Periods when the closed-vent system and control device are not operated as designed including all periods and the duration when:

(i) Any valve car-seal or closure mechanism required under §61.349(a)(1)(ii) is broken or the by-pass line valve position has changed.

(ii) The flow monitoring devices required under §61.349(a)(1)(ii) indicate that vapors are not routed to the control device as required.

(4) If a thermal vapor incinerator is used, then the owner or operator shall maintain continuous records of the temperature of the gas stream in the combustion zone of the incinerator and records of all 3-hour periods of operation during which the average temperature of the gas stream in the combustion zone is more than 28 °C (50 °F) below the design combustion zone temperature.

(5) If a catalytic vapor incinerator is used, then the owner or operator shall maintain continuous records of the temperature of the gas stream both upstream and downstream of the catalyst bed of the incinerator, records of all 3-hour periods of operation during which the average temperature measured before the catalyst bed is more than 28 °C (50 °F) below the design gas stream temperature, and records of all 3-hour periods of operation during which the average temperature difference across the catalyst bed is less than 80 percent of the design temperature difference.

(6) If a boiler or process heater is used, then the owner or operator shall maintain records of each occurrence when there is a change in the location at which the vent stream is introduced into the flame zone as required by  $\S61.349(a)(2)(i)(C)$ . For a boiler or process heater having a design heat input capacity less than 44 MW ( $150 \times 106$  BTU/hr), the owner or operator shall maintain continuous records of the temperature of the gas stream in the combustion zone of the boiler or process heater and records of all 3-hour periods of operation during which the average temperature of the gas stream in the combustion zone is more than 28 °C (50 °F) below the design combustion zone temperature. For a boiler or process heater having a design heat input capacity greater than or equal to 44 MW ( $150 \times 106$  BTU/hr), the owner or operator shall maintain continuous records of the parameter(s) monitored in accordance with the requirements of  $\S61.354(c)(5)$ .

(7) If a flare is used, then the owner or operator shall maintain continuous records of the flare pilot flame monitoring and records of all periods during which the pilot flame is absent.

(8) If a condenser is used, then the owner or operator shall maintain records from the monitoring device of the parameters selected to be monitored in accordance with §61.354(c)(6). If concentration of organics or concentration of benzene in the control device outlet gas stream is monitored, then the owner or operator shall record all 3-hour periods of operation during which the concentration of organics or the concentration of benzene in the exhaust stream is more than 20 percent greater than the design value. If the temperature of the condenser exhaust stream and coolant fluid is monitored, then the owner or operator shall record all 3-hour periods of operation during which the temperature of the condenser exhaust vent stream is more than 6 °C (11 °F) above the design average exhaust vent stream temperature, or the temperature of the condenser is more than 6 °C (11 °F) above the design average coolant fluid temperature at the condenser outlet.

(9) If a carbon adsorber is used, then the owner or operator shall maintain records from the monitoring device of the concentration of organics or the concentration of benzene in the control device outlet gas stream. If the concentration of organics or the concentration of benzene in the control device outlet gas stream is monitored, then the owner or operator shall record all 3-hour periods of operation during which the concentration of organics or the concentration of benzene in the design value. If the carbon bed regeneration interval is monitored, then the owner or operator shall record each occurrence when the vent stream continues to flow through the control device beyond the predetermined carbon bed regeneration time.

(10) If a carbon adsorber that is not regenerated directly on site in the control device is used, then the owner or operator shall maintain records of dates and times when the control device is monitored, when breakthrough is measured, and shall record the date and time then the existing carbon in the control device is replaced with fresh carbon.

(11) If an alternative operational or process parameter is monitored for a control device, as allowed in §61.354(e) of this subpart, then the owner or operator shall maintain records of the continuously monitored parameter, including periods when the device is not

## operated as designed.

(12) If a control device subject to the requirements of §61.349(a)(2)(iv) is used, then the owner or operator shall maintain records of the parameters that are monitored and each occurrence when the parameters monitored are outside the range of values specified in §61.349(a)(2)(iv)(C), or other records as specified by the Administrator.

(k) An owner or operator who elects to install and operate the control equipment in §61.351 of this subpart shall comply with the recordkeeping requirements in 40 CFR 60.115b.

(I) An owner or operator who elects to install and operate the control equipment in §61.352 of this subpart shall maintain records of the following:

(1) The date, location, and corrective action for each visual inspection required by 40 CFR 60.693–2(a)(5), during which a broken seal, gap, or other problem is identified that could result in benzene emissions.

(2) Results of the seal gap measurements required by 40 CFR 60.693-2(a).

(m) If a system is used for emission control that is maintained at a pressure less than atmospheric pressure with openings to provide dilution air, then the owner or operator shall maintain records of the monitoring device and records of all periods during which the pressure in the unit is operated at a pressure that is equal to or greater than atmospheric pressure.

(n) Each owner or operator using a total enclosure to comply with control requirements for tanks in §61.343 or the control requirements for containers in §61.345 must keep the records required in paragraphs (n)(1) and (2) of this section. Owners or operators may use records as required in 40 CFR 264.1089(b)(2)(iv) or 40 CFR 265.1090(b)(2)(iv) for a tank or as required in 40 CFR 264.1089(d)(1) or 40 CFR 265.1090(d)(1) for a container to meet the recordkeeping requirement in paragraph (n)(1) of this section. The owner or operator must make the records of each verification of a total enclosure available for inspection upon request.

(1) Records of the most recent set of calculations and measurements performed to verify that the enclosure meets the criteria of a permanent total enclosure as specified in "Procedure T—Criteria for and Verification of a Permanent or Temporary Total Enclosure" in 40 CFR 52.741, appendix B;

(2) Records required for a closed-vent system and control device according to the requirements in paragraphs (d) (f), and (j) of this section.

[55 FR 8346, Mar. 7, 1990; 55 FR 12444, Apr. 3, 1990; 55 FR 18331, May 2, 1990, as amended at 58 FR 3103, Jan. 7, 1993; 65 FR 62161, Oct. 17, 2000; 67 FR 68533, Nov. 12, 2002]

# § 61.357 Reporting requirements.

(a) Each owner or operator of a chemical plant, petroleum refinery, coke by-product recovery plant, and any facility managing wastes from these industries shall submit to the Administrator within 90 days after January 7, 1993, or by the initial startup for a new source with an initial startup after the effective date, a report that summarizes the regulatory status of each waste stream subject to §61.342 and is determined by the procedures specified in §61.355(c) to contain benzene. Each owner or operator subject to this subpart who has no benzene onsite in wastes, products, by-products, or intermediates shall submit an initial report that is a statement to this effect. For all other owners or operators subject to this subpart, the report shall include the following information:

(1) Total annual benzene quantity from facility waste determined in accordance with §61.355(a) of this subpart.

(2) A table identifying each waste stream and whether or not the waste stream will be controlled for benzene emissions in accordance with the requirements of this subpart.

(3) For each waste stream identified as not being controlled for benzene emissions in accordance with the requirements of this subpart the following information shall be added to the table:

(i) Whether or not the water content of the waste stream is greater than 10 percent;

(ii) Whether or not the waste stream is a process wastewater stream, product tank drawdown, or landfill leachate;

(iii) Annual waste quantity for the waste stream;

(iv) Range of benzene concentrations for the waste stream;

(v) Annual average flow-weighted benzene concentration for the waste stream; and

(vi) Annual benzene quantity for the waste stream.

(4) The information required in paragraphs (a) (1), (2), and (3) of this section should represent the waste stream characteristics based on current configuration and operating conditions. An owner or operator only needs to list in the report those waste streams that contact materials containing benzene. The report does not need to include a description of the controls to be installed to comply with the standard or other information required in §61.10(a).

(b) If the total annual benzene quantity from facility waste is less than 1 Mg/yr (1.1 ton/yr), then the owner or operator shall submit to the Administrator a report that updates the information listed in paragraphs (a)(1) through (a)(3) of this section whenever there is a change in the process generating the waste stream that could cause the total annual benzene quantity from facility waste to increase to 1 Mg/yr (1.1 ton/yr) or more.

(c) If the total annual benzene quantity from facility waste is less than 10 Mg/yr (11 ton/yr) but is equal to or greater than 1 Mg/yr (1.1 ton/yr), then the owner or operator shall submit to the Administrator a report that updates the information listed in paragraphs (a)(1) through (a)(3) of this section. The report shall be submitted annually and whenever there is a change in the process generating the waste stream that could cause the total annual benzene quantity from facility waste to increase to 10 Mg/yr (11 ton/yr) or more. If the information in the annual report required by paragraphs (a)(1) through (a)(3) of this section is not changed in the following year, the owner or operator may submit a statement to that effect.

(d) If the total annual benzene quantity from facility waste is equal to or greater than 10 Mg/yr (11 ton/yr), then the owner or operator shall submit to the Administrator the following reports:

(1) Within 90 days after January 7, 1993, unless a waiver of compliance under §61.11 of this part is granted, or by the date of initial startup for a new source with an initial startup after the effective date, a certification that the equipment necessary to comply with these standards has been installed and that the required initial inspections or tests have been carried out in accordance with this subpart. If a waiver of compliance is granted under §61.11, the certification of equipment necessary to comply with these standards shall be submitted by the date the waiver of compliance expires.

(2) Beginning on the date that the equipment necessary to comply with these standards has been certified in accordance with paragraph (d)(1) of this section, the owner or operator shall submit annually to the Administrator a report that updates the information listed in paragraphs (a)(1) through (a)(3) of this section. If the information in the annual report required by paragraphs (a)(1) through (a)(3) of this section is not changed in the following year, the owner or operator may submit a statement to that effect.

(3) If an owner or operator elects to comply with the requirements of  $\S61.342(c)(3)(ii)$ , then the report required by paragraph (d)(2) of this section shall include a table identifying each waste stream chosen for exemption and the total annual benzene quantity in these exempted streams.

(4) If an owner or operator elects to comply with the alternative requirements of §61.342(d) of this subpart, then he shall include in the report required by paragraph (d)(2) of this section a table presenting the following information for each process wastewater stream:

(i) Whether or not the process wastewater stream is being controlled for benzene emissions in accordance with the requirements of this subpart;

(ii) For each process wastewater stream identified as not being controlled for benzene emissions in accordance with the requirements of this subpart, the table shall report the following information for the process wastewater stream as determined at the point of waste generation: annual waste quantity, range of benzene concentrations, annual average flow-weighted benzene concentration, and annual benzene quantity;

(iii) For each process wastewater stream identified as being controlled for benzene emissions in accordance with the requirements of this subpart, the table shall report the following information for the process wastewater stream as determined at the exit to the treatment process: Annual waste quantity, range of benzene concentrations, annual average flow-weighted benzene concentration, and annual benzene quantity.

(5) If an owner or operator elects to comply with the alternative requirements of  $\S61.342(e)$ , then the report required by paragraph (d)(2) of this section shall include a table presenting the following information for each waste stream:

(i) For each waste stream identified as not being controlled for benzene emissions in accordance with the requirements of this subpart; the table shall report the following information for the waste stream as determined at the point of waste generation: annual waste quantity, range of benzene concentrations, annual average flow-weighted benzene concentration, and annual benzene quantity;

(ii) For each waste stream identified as being controlled for benzene emissions in accordance with the requirements of this subpart; the table shall report the following information for the waste stream as determined at the applicable location described in §61.355(k)(2): Annual waste quantity, range of benzene concentrations, annual average flow-weighted benzene concentration, and annual benzene quantity.

(6) Beginning 3 months after the date that the equipment necessary to comply with these standards has been certified in accordance with paragraph (d)(1) of this section, the owner or operator shall submit quarterly to the Administrator a certification that all of the required inspections have been carried out in accordance with the requirements of this subpart.

(7) Beginning 3 months after the date that the equipment necessary to comply with these standards has been certified in accordance with paragraph (d)(1) of this section, the owner or operator shall submit a report quarterly to the Administrator that includes:

(i) If a treatment process or wastewater treatment system unit is monitored in accordance with §61.354(a)(1) of this subpart, then each period of operation during which the concentration of benzene in the monitored waste stream exiting the unit is equal to or greater than 10 ppmw.

(ii) If a treatment process or wastewater treatment system unit is monitored in accordance with §61.354(a)(2) of this subpart, then each 3-hour period of operation during which the average value of the monitored parameter is outside the range of acceptable values or during which the unit is not operating as designed.

(iii) If a treatment process or wastewater treatment system unit is monitored in accordance with §61.354(b), then each period of operation during which the flow-weighted annual average concentration of benzene in the monitored waste stream entering the unit is equal to or greater than 10 ppmw and/or the total annual benzene quantity is equal to or greater than 1.0 mg/yr.

(iv) For a control device monitored in accordance with §61.354(c) of this subpart, each period of operation monitored during which any of the following conditions occur, as applicable to the control device:

(A) Each 3-hour period of operation during which the average temperature of the gas stream in the combustion zone of a thermal vapor incinerator, as measured by the temperature monitoring device, is more than 28 °C (50 °F) below the design combustion zone temperature.

(B) Each 3-hour period of operation during which the average temperature of the gas stream immediately before the catalyst bed of a catalytic vapor incinerator, as measured by the temperature monitoring device, is more than 28 °C (50 °F) below the design gas stream temperature, and any 3-hour period during which the average temperature difference across the catalyst bed (i.e., the difference between the temperatures of the gas stream immediately before and after the catalyst bed), as measured by the temperature monitoring device, is less than 80 percent of the design temperature difference.

(C) Each 3-hour period of operation during which the average temperature of the gas stream in the combustion zone of a boiler or process heater having a design heat input capacity less than 44 MW (150 × 106 BTU/hr), as mesured by the temperature monitoring device, is more than 28 °C (50 °F) below the design combustion zone temperature.

(D) Each 3-hour period of operation during which the average concentration of organics or the average concentration of benzene in the exhaust gases from a carbon adsorber, condenser, or other vapor recovery system is more than 20 percent greater than the design concentration level of organics or benzene in the exhaust gas.

(E) Each 3-hour period of operation during which the temperature of the condenser exhaust vent stream is more than 6 °C (11 °F) above the design average exhaust vent stream temperature, or the temperature of the coolant fluid exiting the condenser is more than 6 °C (11 °F) above the design average coolant fluid temperature at the condenser outlet.

(F) Each period in which the pilot flame of a flare is absent.

(G) Each occurrence when there is a change in the location at which the vent stream is introduced into the flame zone of a boiler or process heater as required by §61.349(a)(2)(i)(C) of this subpart.

(H) Each occurrence when the carbon in a carbon adsorber system that is regenerated directly on site in the control device is not regenerated at the predetermined carbon bed regeneration time.

(I) Each occurrence when the carbon in a carbon adsorber system that is not regenerated directly on site in the control device is not replaced at the predetermined interval specified in §61.354(c) of this subpart.

(J) Each 3-hour period of operation during which the parameters monitored are outside the range of values specified in §61.349(a)(2)(iv)(C), or any other periods specified by the Administrator for a control device subject to the requirements of §61.349(a)(2)(iv).

(v) For a cover and closed-vent system monitored in accordance with §61.354(g), the owner or operator shall submit a report quarterly to the Administrator that identifies any period in which the pressure in the waste management unit is equal to or greater than atmospheric pressure.

(8) Beginning one year after the date that the equipment necessary to comply with these standards has been certified in accordance with paragraph (d)(1) of this section, the owner or operator shall submit annually to the Administrator a report that summarizes all inspections required by §§61.342 through 61.354 during which detectable emissions are measured or a problem (such as a broken seal, gap or other problem) that could result in benzone emissions is identified, including information about the repairs or corrective action taken.

(e) An owner or operator electing to comply with the provisions of §§61.351 or 61.352 of this subpart shall notify the Administrator of the alternative standard selected in the report required under §61.07 or §61.10 of this part.

(f) An owner or operator who elects to install and operate the control equipment in §61.351 of this subpart shall comply with the reporting requirements in 40 CFR 60.115b.

(g) An owner or operator who elects to install and operate the control equipment in §61.352 of this subpart shall submit initial and quarterly reports that identify all seal gap measurements, as required in 40 CFR 60.693–2(a), that are outside the prescribed limits.

[55 FR 8346, Mar. 7 1990; 55 FR 12444, Apr. 3, 1990, as amended at 55 FR 37231, Sept. 10, 1990; 58 FR 3105, Jan. 7, 1993; 65 FR 62161, Oct. 17, 2000]

# § 61.358 Delegation of authority.

(a) In delegating implementation and enforcement authority to a State under section 112(d) of the Clean Air Act, the authorities contained in paragraph (b) of this section shall be retained by the Administrator and not transferred to a State.

(b) Alternative means of emission limitation under §61.353 of this subpart will not be delegated to States.

# § 61.359 [Reserved]

Appendix A to Part 61

#### AFRENDER A

National Entssion Starlands for separatus ann Egil Libras

Compliance Claber Information

```
1. SOLPCE REFORT
```

10053.001085 Parlame on convertion of storages of constraints pollutents subject to the factors Encision Dardards for replant with a replaying and replay Reports for the information contential information for a subject subject of the content of subject after the Effective date of any starsame of even entrember here, the subject of our information.
4 first of replaying information of even information.
4 first of replaying information of even information.
5 first strain first and provided an UNLOG
5 for even of the subject of even information.
6 first of replaying information of even information.
6 first of replaying information of even information.
7 first information of the subject of even information.
7 first information of the subject of even information.
7 first information of the subject of even information.
7 first information of the subject of even information.
7 first information of the subject of even information.
7 first information of the subject of even information.
7 first information of the subject of even information.
7 first information of the subject of even information.
7 first information of the subject of even information of the subject of even information.
7 first information of the subject of even information of even information of the subject of even information of ev

 Sup 1-10
 ST
 ESter Market
 St State 35
 79

 401
 State Registic Number
 T4
 State 35
 79

 401
 State Registic Number
 T4
 State 35
 79

 401
 State Registic Number
 T4
 State 35
 70

 401
 State 35
 State 35
 State 35
 71
 70

 <u>Contact</u> - Indicate the neme and telephone number of the owner or operator or other responsible official when \$54 may contact concerning this report.

```
View or download PDF
```

	Dup 1-'8	19 20	21	Harp		
	ta 46 Ares Lode	L. hunser				
<b>1</b> .	Source lies alkali Pia	cription - S: nt or Mach	rle" y state the ine Shap"].	rature of the	savree te :	)., "Imlin-
	Des 1-10	19 20 2	ı ———	Erscrigtion		50
	1		CantInced		79	छ
4.	Alternetia metting edi to a locat	e Kalling Ad: dreis / I son tor differen	tress - Indicate respindence is to t than that spec	an alternative o de directed iffed above.		
	Du⊳ 3-'8	2 1 2 1 2 0	21 Aurber	Street or Box H	and an	- ZE 67
	Bup 1-10	4 4 15 20	21 CILy	35	17 33 51117	4 715 14 30
5.	Compliance the entropy prior to So which req.	Status - Thi on Tyrtacroi C Says After 'ne the Subm	e emissions from as cortained in the effective a ission of such i	the national [r ate of any stan storration.	- can Tssion Sta dards on a	Cannot meet ndards te or aandments
Nort: The Envision St Subject to Administration Such walver	Fe entry for andards for Federal pre- for of the C s is listed	Sign From the so Harandous An ancement acts S. (netroname th Section	nature of Dever, corre will access or Pollutants, ti ions unless gram e-tal Protection 11 of this form.	Operator or Ot 1 Chose Trates he source will Let a weiger of Agency. The J	her Tespon Set by the te 1- yil: compliants aformation	NETE (If(I <u>cra)</u> National atton and Ex the regized for

 PAULINS INFORMATION, Part B should be constanted separately for each point of emission for each hegandous pollutant. (Sources subject to 61.22(1) may only number 4. below.)

. . . . . . . . . . . . . . . .

Dup 1 13 B G 5 30 30 31 4 16 17 18 19 25 300 27 20 59 30 31 HE2S & Ref LS STP

- - ---

View or download PDF

1,	Pollucent process.	Enlitted Indicate	- Indicate th "AS" for ast	ne type of Nestos, 'Ei	hazardo.s ' for ber	səllət 7 Univ,	ant eniti or "NG"	ed by the for mercury.
	<u>37 33</u> Pollutant	э <b>г</b>	Fea, lat I	n		यमः व ह	र २	
2,	Process Ge Thydrogen a beryllis	<u>scription</u> and Eax <sup>+</sup> in nachine	n - Provide a Tin a nercury Eshop), Use	brief des chior-aik addictona	cristion aif gipnt i seets	of each , "grlr 1f nece	ding mach sistry.	'P.G., Ine' 18
	50	Process	Jescription	,	• জি			
	Nup 1-18	6 1 19 20	21		•••••••••••		·	5C
	5]		**************************************	•		σ		
1	Dup 1-19	1 <u>5 2</u> 0	21	<u> </u>				50
	iT				79 8	5		
3.	Anount of named in 1 previous t	Pollutant ter 1 whi welve mon	- Indicate ct enters ch ths of opera	the averag e process ston).	e melight i 16 pounds	of the ⇒erπor	hezandous nth (based	neterial on the
	Gup 1-1E	25 70	zi		29	is. (no.	36	33
4.	Control De a. Indica the en cyclos renove	vices te the ty issions f el and th s from th	pe of pollut ron the proc e estimated e process ga	ton contro ess (e.g., percent of s stream,	l desfces venturf the politi	. 1f an; scrubbe: stant wi	y, used to r, baghous wich the p	; reduce e, net evice
	Dup 1-18	15 ZD	21	PRIMARY ()	DINTROL DEN	<u>NCE .</u>	4.1	
	4 <u>5 P</u>	rinary De	vica have	<u> </u>	66 Fercent Ri Efficier	70 TTCVA1 L	72	79
	च्च							

View or download PDF

Due 1-15 - 5 - 5 - 51 - 511 -
47 Cerumdery Device Gra 64 65 70 70 20 Percent Removal Effectency
<ul> <li>E. Asherron Freinkrum Control Deriver Only</li> <li>1. Dia tashawan in nervited in Dim 40, give the following reformation.</li> </ul>
<ul> <li>The are flow permanifully in tubic feet per minute per square foot of factor error.</li> </ul>
Ale Directories by the Cfr/ft <sup>2</sup>
The pressure drip to include water dauge scross the filter at which the bedrouse is coercipe.
OpenAlina cressure drap = inches W.g.
<ul> <li>If the bathouse nativial contains symbletic fill yarm, their whether this material is 2 -2 spin f -2 or not spin.</li> </ul>
It she bagtouse utilities a felter febric, give the minimum trickness in scores and the density in durines per square yerd.
"hickorss r trubes Consity r pz/yd <sup>2</sup>
<ol> <li>If a wet collection device is specified in Item 44, give the designed unit contacting energy or Inches Water gauge.</li> </ol>
- Unit contesting energy Inches w.g
(. <u>013205AL 0F ASBLS103-CCN14IN(NF #451(5</u> Part C should be completed separately for each ascestos-containing waits generation operation arising from sources subject to \$61.27(g), (c), (c), and (r).
0.5 1-13 0.0 5 20 500 27 78 29 30 31 16 17 18 19 20 500 27 78 29 30 31 HEDS X REF CS SIP
4 5 27 33 34 - Ceguletton - 65 66 Pollutare - FC

View or download PDF

<u>Weste Generation</u> - Provide a brief ceschiption of each process that generates astrocommunication (aug. dispess) of control revise wastes;

5. Process Lettrip for 70 ch

2. Appendix control to indicate the overage centerlage appendix content of these materials. Dup to  $\frac{e}{2} = \frac{1}{20} = \frac{1}{21}$  Appendix CONTENTENTION (7) at (7)

38 CZ

- Annuni of mastes increate the average weight of astestos-containing wastes disposed of, reasoned in vg/day.
  - Dup 1-18 <u>6 2</u> 19 20 2 2 2 2 2 8 80
- <u>Control Methods</u> Indicate the existed control methods used in all stages of weste dispusal, from collections processing, and processing to transacting and deposition.
  - Eug 1-18 5.3 19 20 27 Frinery Centrol Hechod 33
  - 45 53 55 Cup 1-18 5 4 19<sup>-00</sup> 73 71 70 70 70 70 50
- 51 75 B3 5. <u>Maste Disposal</u> - Inducte the type of disposal site (canttary letdfill, ppen, covered) or inconstation site (municipal, private, where the waste is disposed of and who operates the site (constry, private, municipal). State the mane and location of the site (closest bity or four, courty, individual)
  - State the mane and location of the site (classes) bity on town, county, state).
  - bup 1 10
     6 i
     TYPE 6<sup>2</sup> (17)::
     13
     35

     19
     20
     1
     53
     53

     51
     75
     83

View or download PDF

 Eup 1.18
 4.6
 21
 02764108
 29
 21
 50

 S1
 75
 85

 Dup 1.18
 6.7
 71
 20
 21

 S1
 70
 71
 70

- HKETE DISPESAL CUTYS. Part Distolic te completed separately for ever attests waste dispectal viral subject to section (1.22000).
- 0.; (-13 12 16 7 15 79 25 51; 77 28 71 35 31 hEDS = Per (2 50) 22 33 32 Peg0'stion to 25 ec'lutert (1 500540 5116 50 68 50
- <u>Ensingilar</u> Previce a brief description of the site, including its size and <u>Configuration</u>, and the distance to the closest city or turn, closest residence, and closest primary road.
- Cup 1-18
   5 1
   5775 35503 (470)
   37
   37
   37
   50

   51
   15
   20
   21
   21
   37
   37
   30
   50

   51
   10
   10
   37
   37
   37
   50
   50

   51
   10
   10
   10
   37
   36
   37
   50

   51
   10
   10
   10
   34
   36
   37
   42
   43
- 4<u>5 (0(0))</u> 4<u>5 − 5</u>1 (0<u>− 5</u>) (<del>1</del> − 5) (<del>1</del> − 5) (1 − 5) 2<sup>7</sup> − 78 80

## View or download PDF

2.	Chactivat used to c undertake	ion - Afre comply with in to natet	r the site is inactivated, indicat the standard and send a list of t aim the inactivated site.	e the method or methods the actions that will be
	Bup 1-18	15 <mark>58</mark>	нетнествае	The site:
	<u>e</u> t		79	BC

#### View or download PDF

#### II. Waiver Requests

A. Waiver of Compliance. Owners or operators of sources unable to operate in compliance with the National Emission Standards for Hazardous Air Pollutants prior to 90 days after the effective date of any standards or amendments which require the submission of such information may request a waiver of compliance from the Administrator of the U.S. Environmental Protection Agency for the time period necessary to install appropriate control devices or make modifications to achieve compliance. The Administrator may grant a waiver of compliance with the standard for a period not exceeding two years from the effective date of the hazardous pollutant standards, if he finds that such period is necessary for the installation of controls and that steps will be taken during the period of the waiver to assure that the health of persons will be protected from imminent endangerment.

The report information provided in Section I must accompany this application. Applications should be sent to the appropriate EPA regional office.

1. Processes Involved —Indicate the process or processes emitting hazardous pollutants to which emission controls are to be applied.

## 2. Controls

a. Describe the proposed type of control device to be added or modification to be made to the process to reduce the emission of hazardous pollutants to an acceptable level. (Use additional sheets if necessary.)

b. Describe the measures that will be taken during the waiver period to assure that the health of persons will be protected from imminent endangerment. (Use additional sheets if necessary.)

Date by which contracts for emission control systems or process modifications will be awarded; or date by which orders will be issued for the purchase of the component parts to accomplish emission control or process modification.

View or download PDF

B. Waiver of Emission Tests. A waiver of emission testing may be granted to owners or operators of sources subject to emmission testing if, in the judgment of the Administrator of the Environmental Protection Agency the emissions from the source comply with the appropriate standard or if the owners or operators of the source have requested a waiver of compliance or have been granted a waiver of compliance.

This application should accompany the report information provided in Section I.

1. Reason —State the reasons for requesting a waiver of emission testing. If the reason stated is that the emissions from the source are within the prescribed limits, documentation of this condition must be attached.

DateSignature of the owner or operator

(Sec. 114, of the Clean Air Act as amended (42 U.S.C. 7414))

[40 FR 48303, Oct. 14, 1975, as amended at 43 FR 8800, Mar. 3, 1978; 50 FR 46295, Sept. 9, 1985]

## Appendix B to Part 61—Test Methods

Method 101-Determination of particulate and gaseous mercury emissions from chlor-alkali plants (air streams)

Method 101A—Determination of particulate and gaseous mercury emissions from sewage sludge incinerators

Method 102—Determination of particulate and gaseous mercury emissions from chlor-alkali plants (hydrogen streams)

Method 103-Beryllium screening method

Method 104-Determination of beryllium emissions from stationary sources

Method 105-Determination of mercury in wastewater treatment plant sewage sludges

Method 106-Determination of vinyl chloride emissions from stationary sources

Method 107---Determination of vinyl chloride content of in-process wastewater samples, and vinyl chloride content of polyvinyl chloride resin slurry, wet cake, and latex samples

Method 107A—Determination of vinyl chloride content of solvents, resin-solvent solution, polyvinyl chloride resin, resin slurry, wet resin, and latex samples

Method 108—Determination of particulate and gaseous arsenic emissions

Method 108A-Determination of arsenic content in ore samples from nonferrous smelters

Method 108B—Determination of arsenic content in ore samples from nonferrous smelters

Method 108C-Determination of arsenic content in ore samples from nonferrous smelters (molybdenum blue photometric procedure)

Method 111-Determination of Polonium-210 emissions from stationary sources

Method 101—Determination of Particulate and Gaseous Mercury Emissions From Chlor-Alkali Plants (Air Streams)

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from methods in appendix A to 40 CFR part 60. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3, and Method 5.

1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.	Sensitivity	
Mercury (Hg)	7439–97–6	Dependent upon recorder and spectrophotometer.	

1.2 Applicability. This method is applicable for the determination of Hg emissions, including both particulate and gaseous Hg, from chlor-alkali plants and other sources (as specified in the regulations) where the carrier-gas stream in the duct or stack is principally air.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

## 2.0 Summary of Method

Particulate and gaseous Hg emissions are withdrawn isokinetically from the source and collected in acidic iodine monochloride (ICI) solution. The Hg collected (in the mercuric form) is reduced to elemental Hg, which is then aerated from the solution into an optical cell and measured by atomic absorption spectrophotometry.

#### 3.0 Definitions[Reserved]

4.0 Interferences

4.1 Sample Collection. Sulfur dioxide (SO<sub>2</sub>) reduces ICI and causes premature depletion of the ICI solution.

#### 4.2 Sample Analysis.

4.2.1 ICI concentrations greater than 10<sup>-4</sup>molar inhibit the reduction of the Hg (II) ion in the aeration cell.

4.2.2 Condensation of water vapor on the optical cell windows causes a positive interference.

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

5.2 Corrosive Reagents. The following reagents are hazardous. Personal protective equipment and safe procedures are useful in preventing chemical splashes. If contact occurs, immediately flush with copious amounts of water for at least 15 minutes. Remove clothing under shower and decontaminate. Treat residual chemical burn as thermal burn.

5.2.1 Hydrochloric Acid (HCl). Highly toxic and corrosive. Causes severe damage to tissues. Vapors are highly irritating to eyes, skin, nose, and lungs, causing severe damage. May cause bronchitis, pneumonia, or edema of lungs. Exposure to concentrations of 0.13 to 0.2 percent can be lethal to humans in a few minutes. Provide ventilation to limit exposure. Reacts with metals, producing hydrogen gas.

5.2.2 Nitric Acid (HNO<sub>3</sub>). Highly corrosive to eyes, skin, nose, and lungs. Vapors cause bronchitis, pneumonia, or edema of lungs. Reaction to inhalation may be delayed as long as 30 hours and still be fatal. Provide ventilation to limit exposure. Strong oxidizer. Hazardous reaction may occur with organic materials such as solvents.

5.2.3 Sulfuric Acid (H<sub>2</sub>SO<sub>4</sub>). Rapidly destructive to body tissue. Will cause third degree burns. Eye damage may result in blindness. Inhalation may be fatal from spasm of the larynx, usually within 30 minutes. 3 mg/m<sup>3</sup> will cause lung damage. 1 mg/m<sup>3</sup> for 8 hours will cause lung damage or, in higher concentrations, death. Provide ventilation to limit inhalation. Reacts violently with metals and organics.

6.0 Equipment and Supplies.

6.1 Sample Collection. A schematic of the sampling train used in performing this method is shown in Figure 101–1; it is similar to the Method 5 sampling train. The following items are required for sample collection:

6.1.1 Probe Nozzle, Pitot Tube, Differential Pressure Gauge, Metering System, Barometer, and Gas Density Determination Equipment. Same as Method 5, Sections 6.1.1.1, 6.1.1.3, 6.1.1.4, 6.1.1.9, 6.1.2, and 6.1.3, respectively.

6.1.2 Probe Liner. Borosilicate or quartz glass tubing. A heating system capable of maintaining a gas temperature of 120 ±14 °C (248 ±25 °F) at the probe exit during sampling may be used to prevent water condensation.

Note: Do not use metal probe liners.

6.1.3 Impingers. Four Greenburg-Smith impingers connected in series with leak-free ground glass fittings or any similar leak-free noncontaminating fittings. For the first, third, and fourth impingers, impingers that are modified by replacing the tip with a 13-mm ID (0.5-in.) glass tube extending to 13 mm (0.5 in.) from the bottom of the flask may be used.

6.1.4 Acid Trap. Mine Safety Appliances air line filter, Catalog number 81857, with acid absorbing cartridge and suitable connections, or equivalent.

6.2 Sample Recovery. The following items are needed for sample recovery:

6.2.1 Glass Sample Bottles. Leakless, with Teflon-lined caps, 1000- and 100-ml.

6.2.2 Graduated Cylinder. 250-ml.

6.2.3 Funnel and Rubber Policeman. To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

6.2.4 Funnel. Glass, to aid in sample recovery.

6.3 Sample Preparation and Analysis. The following items are needed for sample preparation and analysis:

6.3.1 Atomic Absorption Spectrophotometer. Perkin-Elmer 303, or equivalent, containing a hollow-cathode mercury lamp and the optical cell described in Section 6.3.2.

6.3.2 Optical Cell. Cylindrical shape with quartz end windows and having the dimensions shown in Figure 101–2. Wind the cell with approximately 2 meters (6 ft) of 24-gauge Nichrome wire, or equivalent, and wrap with fiberglass insulation tape, or equivalent; do not let the wires touch each other.

6.3.3 Aeration Cell. Constructed according to the specifications in Figure 101–3. Do not use a glass frit as a substitute for the blown glass bubbler tip shown in Figure 101–3.

6.3.4 Recorder. Matched to output of the spectrophotometer described in Section 6.3.1.

6.3.5 Variable Transformer. To vary the voltage on the optical cell from 0 to 40 volts.

6.3.6 Hood. For venting optical cell exhaust.

6.3.7 Flow Metering Valve.

6.3.8 Rate Meter. Rotameter, or equivalent, capable of measuring to within 2 percent a gas flow of 1.5 liters/min (0.053 cfm).

6.3.9 Aeration Gas Cylinder. Nitrogen or dry, Hg-free air, equipped with a single-stage regulator.

6.3.10 Tubing. For making connections. Use glass tubing (ungreased ball and socket connections are recommended) for all tubing connections between the solution cell and the optical cell; do not use Tygon tubing, other types of flexible tubing, or metal tubing as substitutes. Teflon, steel, or copper tubing may be used between the nitrogen tank and flow metering valve (Section 6.3.7), and Tygon, gum, or rubber tubing between the flow metering valve and the aeration cell.

6.3.11 Flow Rate Calibration Equipment. Bubble flow meter or wet-test meter for measuring a gas flow rate of 1.5 ±0.1 liters/min (0.053 ±0.0035 cfm).

6.3.12 Volumetric Flasks. Class A with penny head standard taper stoppers; 100-, 250-, 500-, and 1000-ml.

6.3.13 Volumetric Pipets. Class A; 1-, 2-, 3-, 4-, and 5-ml.

6.3.14 Graduated Cylinder. 50-ml.

6.3.15 Magnetic Stirrer. General-purpose laboratory type.

6.3.16 Magnetic Stirring Bar. Teflon-coated.

6.3.17 Balance. Capable of weighing to ±0.5 g.

6.3.18 Alternative Analytical Apparatus. Alternative systems are allowable as long as they meet the following criteria:

6.3.18.1 A linear calibration curve is generated and two consecutive samples of the same aliquot size and concentration agree within 3 percent of their average.

6.3.18.2 A minimum of 95 percent of the spike is recovered when an aliquot of a source sample is spiked with a known concentration of Hg (II) compound.

6.3.18.3 The reducing agent should be added after the aeration cell is closed.

6.3.18.4 The aeration bottle bubbler should not contain a frit.

6.3.18.5 Any Tygon tubing used should be as short as possible and conditioned prior to use until blanks and standards yield linear and reproducible results.

6.3.18.6 If manual stirring is done before aeration, it should be done with the aeration cell closed.

6.3.18.7 A drying tube should not be used unless it is conditioned as the Tygon tubing above.

7.0 Reagents and Standards

Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society; where such specifications are not available, use the best available grade.

7.1 Sample Collection. The following reagents are required for sample collection:

7.1.1 Water. Deionized distilled, to conform to ASTM D 1193–77 or 91 (incorporated by reference—see §61.18), Type 1. If high concentrations of organic matter are not expected to be present, the analyst may eliminate the KMnO₄test for oxidizable organic matter. Use this water in all dilutions and solution preparations.

7.1.2 Nitric Acid, 50 Percent (v/v). Mix equal volumes of concentrated HNO<sub>3</sub>and water, being careful to add the acid to the water slowly.

7.1.3 Silica Gel. Indicating type, 6- to 16-mesh. If previously used, dry at 175 °C (350 °F) for 2 hours. The tester may use new silica gel as received.

7.1.4 Potassium Iodide (KI) Solution, 25 Percent. Dissolve 250 g of KI in water, and dilute to 1 liter.

7.1.5 Iodine Monochloride Stock Solution, 1.0 M. To 800 ml of 25 percent KI solution, add 800 ml of concentrated HCI. Cool to room temperature. With vigorous stirring, slowly add 135 g of potassium iodate (KIO<sub>3</sub>), and stir until all free iodine has dissolved. A clear orange-red solution occurs when all the KIO<sub>3</sub>has been added. Cool to room temperature, and dilute to 1800 ml with water. Keep the solution in amber glass bottles to prevent degradation.

7.1.6 Absorbing Solution, 0.1 M ICI. Dilute 100 ml of the 1.0 M ICI stock solution to 1 liter with water. Keep the solution in amber glass bottles and in darkness to prevent degradation. This reagent is stable for at least two months.

7.2 Sample Preparation and Analysis. The following reagents and standards are required for sample preparation and analysis:

#### 7.2.1 Reagents.

7.2.1.1 Tin (II) Solution. Prepare fresh daily, and keep sealed when not being used. Completely dissolve 20 g of tin (II) chloride (or 25 g of tin (II) sulfate) crystals (Baker Analyzed reagent grade or any other brand that will give a clear solution) in 25 ml of concentrated HCI. Dilute to 250 ml with water. Do not substitute  $HNO_3$ ,  $H_2SO_4$ , or other strong acids for the HCI.

7.2.1.2 Sulfuric Acid, 5 Percent (v/v). Dilute 25 ml of concentrated H₂SO₄to 500 ml with water.

#### 7.2.2 Standards

7.2.2.1 Hg Stock Solution, 1 mg Hg/ml. Prepare and store all Hg standard solutions in borosilicate glass containers. Completely dissolve 0.1354 g of Hg (II) chloride in 75 ml of water in a 100-ml glass volumetric flask. Add 10 ml of concentrated HNO<sub>3</sub>, and adjust the volume to exactly 100 ml with water. Mix thoroughly. This solution is stable for at least one month.

7.2.2.2 Intermediate Hg Standard Solution, 10  $\mu$ g Hg/ml. Prepare fresh weekly. Pipet 5.0 ml of the Hg stock solution (Section 7.2.2.1) into a 500-ml glass volumetric flask, and add 20 ml of the 5 percent H<sub>2</sub>SO<sub>4</sub>solution. Dilute to exactly 500 ml with water. Thoroughly mix the solution.

7.2.2.3 Working Hg Standard Solution, 200 ng Hg/ml. Prepare fresh daily. Pipet 5.0 ml of the intermediate Hg standard solution (Section 7.2.2.2) into a 250-ml volumetric glass flask. Add 10 ml of the 5 percent H<sub>2</sub>SO<sub>4</sub>and 2 ml of the 0.1 M ICl absorbing solution taken as a blank (Section 8.7.4.3), and dilute to 250 ml with water. Mix thoroughly.

#### 8.0 Sample Collection, Preservation, Transport, and Storage

Because of the complexity of this method, testers should be trained and experienced with the test procedures to ensure reliable results. Since the amount of Hg that is collected generally is small, the method must be carefully applied to prevent contamination or loss of sample.

8.1 Pretest Preparation. Follow the general procedure outlined in Method 5, Section 8.1, except omit Sections 8.1.2 and 8.1.3.

8.2 Preliminary Determinations. Follow the general procedure outlined in Method 5, Section 8.2, with the exception of the following:

8.2.1 Select a nozzle size based on the range of velocity heads to assure that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates below 28 liters/min (1.0 cfm).

8.2.2 Perform test runs such that samples are obtained over a period or periods that accurately determine the maximum emissions that occur in a 24-hour period. In the case of cyclic operations, run sufficient tests for the accurate determination of the emissions that occur over the duration of the cycle. A minimum sample time of 2 hours is recommended. In some instances, high Hg or high SO<sub>2</sub>concentrations make it impossible to sample for the desired minimum time. This is indicated by reddening (liberation of free iodine) in the first impinger. In these cases, the sample run may be divided into two or more subruns to ensure that the absorbing solution is not depleted.

## 8.3 Preparation of Sampling Train.

8.3.1 Clean all glassware (probe, impingers, and connectors) by rinsing with 50 percent HNO<sub>3</sub>, tap water, 0.1 M ICI, tap water, and finally deionized distilled water. Place 100 ml of 0.1 M ICI in each of the first three impingers. Take care to prevent the absorbing solution from contacting any greased surfaces. Place approximately 200 g of preweighed silica gel in the fourth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the silica gel container in a clean place for later use in the sample recovery. Alternatively, determine and record the weight of the silica gel plus impinger to the nearest 0.5 g.

8.3.2 Install the selected nozzle using a Viton A O-ring when stack temperatures are less than 260 °C (500 °F). Use a fiberglass string gasket if temperatures are higher. See APTD–0576 (Reference 3 in Method 5) for details. Other connecting systems using either 316 stainless steel or Teflon ferrules may be used. Mark the probe with heat-resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

8.3.3 Assemble the train as shown in Figure 101–1, using (if necessary) a very light coat of silicone grease on all ground glass joints. Grease only the outer portion (see APTD–0576) to avoid the possibility of contamination by the silicone grease.

Note: An empty impinger may be inserted between the third impinger and the silica gel to remove excess moisture from the sample stream.

8.3.4 After the sampling train has been assembled, turn on and set the probe heating system, if applicable, at the desired operating temperature. Allow time for the temperatures to stabilize. Place crushed ice around the impingers.

8.4 Leak-Check Procedures. Follow the leak-check procedures outlined in Method 5, Section 8.4.

8.5 Sampling Train Operation. Follow the general procedure outlined in Method 5, Section 8.5. For each run, record the data required on a data sheet such as the one shown in Figure 101–4.

8.6 Calculation of Percent Isokinetic. Same as Method 5, Section 8.6.

8.7 Sample Recovery. Begin proper cleanup procedure as soon as the probe is removed from the stack at the end of the sampling period.

8.7.1 Allow the probe to cool. When it can be safely handled, wipe off any external particulate matter near the tip of the probe nozzle, and place a cap over it. Do not cap off the probe tip tightly while the sampling train is cooling. Capping would create a vacuum and draw liquid out from the impingers.

8.7.2 Before moving the sampling train to the cleanup site, remove the probe from the train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the impinger. Use either ground-glass stoppers, plastic caps, or serum caps to close these openings.

8.7.3 Transfer the probe and impinger assembly to a cleanup area that is clean, protected from the wind, and free of Hg contamination. The ambient air in laboratories located in the immediate vicinity of Hg-using facilities is not normally free of Hg contamination.

8.7.4 Inspect the train before and during disassembly, and note any abnormal conditions. Treat the samples as follows.

8.7.4.1 Container No. 1 (Impingers and Probe).

8.7.4.1.1 Using a graduated cylinder, measure the liquid in the first three impingers to within 1 ml. Record the volume of liquid present (*e.g.*, see Figure 5–6 of Method 5). This information is needed to calculate the moisture content of the effluent gas. (Use only glass storage bottles and graduated cylinders that have been precleaned as in Section 8.3.1) Place the contents of the first three impingers into a 1000-ml glass sample bottle.

8.7.4.1.2 Taking care that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover the Hg (and any condensate) from the probe nozzle, probe fitting, and probe liner as follows: Rinse these components with two 50-ml portions of 0.1 M ICI. Next, rinse the probe nozzle, fitting and liner, and each piece of connecting glassware between the probe liner and the back half of the third impinger with a maximum of 400 ml of water. Add all washings to the 1000-ml glass sample bottle containing the liquid from the first three impingers.

8.7.4.1.3 After all washings have been collected in the sample container, tighten the lid on the container to prevent leakage during shipment to the laboratory. Mark the height of the liquid to determine later whether leakage occurred during transport. Label the container to identify clearly its contents.

8.7.4.2 Container No. 2 (Silica Gel). Same as Method 5, Section 8.7.6.3.

8.7.4.3 Container No. 3 (Absorbing Solution Blank). Place 50 ml of the 0.1 M ICI absorbing solution in a 100-ml sample bottle. Seal the container. Use this blank to prepare the working Hg standard solution (Section 7.2.2.3).

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
8.4 10.2	Sampling equipment leak-checks and calibration	Ensure accuracy and precision of sampling measurements.
10.5, 10.6	Spectrophotometer calibration	Ensure linearity of spectrophotometer response to standards.
11.3.3	Check for matrix effects	Eliminate matrix effects.

9.2 Volume Metering System Checks. Same as Method 5, Section 9.2.

10.0 Calibration and Standardizations

Note: Maintain a laboratory log of all calibrations.

10.1 Before use, clean all glassware, both new and used, as follows: brush with soap and tap water, liberally rinse with tap water, soak for 1 hour in 50 percent HNO<sub>3</sub>, and then rinse with deionized distilled water.

10.2 Sampling Equipment. Calibrate the sampling equipment according to the procedures outlined in the following sections of Method 5: Section 10.1 (Probe Nozzle), Section 10.2 (Pitot Tube Assembly), Section 10.3 (Metering System), Section 10.5 (Temperature Sensors), Section 10.6 (Barometer).

10.3 Aeration System Flow Rate Meter. Assemble the aeration system as shown in Figure 101–5. Set the outlet pressure on the aeration gas cylinder regulator to a minimum pressure of 500 mm Hg (10 psi), and use the flow metering valve and a bubble flowmeter or wet-test meter to obtain a flow rate of  $1.5 \pm 0.1$  liters/min (0.053 ±0.0035 cfm) through the aeration cell. After the calibration of the aeration system flow rate meter is complete, remove the bubble flowmeter from the system.

10.4 Optical Cell Heating System. Using a 50-ml graduated cylinder, add 50 ml of water to the bottle section of the aeration cell, and attach the bottle section to the bubbler section of the cell. Attach the aeration cell to the optical cell and while aerating at  $1.5 \pm 0.1$  liters/min (0.053 ±0.0035 cfm), determine the minimum variable transformer setting necessary to prevent condensation of moisture in the optical cell and in the connecting tubing. (This setting should not exceed 20 volts.)

10.5 Spectrophotometer and Recorder.

10.5.1 The Hg response may be measured by either peak height or peak area.

Note: The temperature of the solution affects the rate at which elemental Hg is released from a solution and, consequently, it affects the shape of the absorption curve (area) and the point of maximum absorbance (peak height). Therefore, to obtain reproducible results, bring all solutions to room temperature before use.

10.5.2 Set the spectrophotometer wavelength at 253.7 nm, and make certain the optical cell is at the minimum temperature that will prevent water condensation. Then set the recorder scale as follows: Using a 50-ml graduated cylinder, add 50 ml of water to the aeration cell bottle. Add three drops of Antifoam B to the bottle, and then pipet 5.0 ml of the working Hg standard solution into the aeration cell.

Note: Always add the Hg-containing solution to the aeration cell after the 50 ml of water.

10.5.3 Place a Teflon-coated stirring bar in the bottle. Before attaching the bottle section to the bubbler section of the aeration cell, make certain that (1) the aeration cell exit arm stopcock (Figure 101–3) is closed (so that Hg will not prematurely enter the optical cell when the reducing agent is being added) and (2) there is no flow through the bubbler. If conditions (1) and (2) are met, attach the bottle section to the bubbler section of the aeration cell. Pipet 5 ml of tin (II) reducing solution into the aeration cell through the side arm, and immediately stopper the side arm. Stir the solution for 15 seconds, turn on the recorder, open the aeration cell exit arm stopcock, and immediately initiate aeration with continued stirring. Determine the maximum absorbance of the standard, and set this value to read 90 percent of the recorder full scale.

## 10.6 Calibration Curve.

10.6.1 After setting the recorder scale, repeat the procedure in Section 10.5 using 0.0-, 1.0-, 2.0-, 3.0-, 4.0-, and 5.0-ml aliquots of the working standard solution (final amount of Hg in the aeration cell is 0, 200, 400, 600, 800, and 1000 ng, respectively). Repeat this procedure on each aliquot size until two consecutive peaks agree within 3 percent of their average value.

# Note: To prevent Hg carryover from one sample to another, do not close the aeration cell from the optical cell until the recorder pen has returned to the baseline.)

10.6.2 It should not be necessary to disconnect the aeration gas inlet line from the aeration cell when changing samples. After separating the bottle and bubbler sections of the aeration cell, place the bubbler section into a 600-ml beaker containing approximately 400 ml of water. Rinse the bottle section of the aeration cell with a stream of water to remove all traces of the tin (II) reducing agent. Also, to prevent the loss of Hg before aeration, remove all traces of the reducing agent between samples by washing with water. It will be necessary, however, to wash the aeration cell parts with concentrated HCI if any of the following conditions occur: (1) A white film appears on any inside surface of the aeration cell, (2) the calibration curve changes suddenly, or (3) the replicate samples do not yield reproducible results.

10.6.3 Subtract the average peak height (or peak area) of the blank (0.0-ml aliquot)—which must be less than 2 percent of recorder full scale—from the averaged peak heights of the 1.0-, 2.0-, 3.0-, 4.0-, and 5.0-ml aliquot standards. If the blank absorbance is greater than 2 percent of full-scale, the probable cause is Hg contamination of a reagent or carry-over of Hg from a previous sample. Prepare the calibration curve by plotting the corrected peak height of each standard solution versus the corresponding final total Hg weight in the aeration cell (in ng), and draw the best fit straight line. This line should either pass through the origin or pass through a point no further from the origin than ±2 percent of the recorder full scale. If the line does not pass through or very near to the origin, check for nonlinearity of the curve and for incorrectly prepared standards.

## 11.0 Analytical Procedure

11.1 Sample Loss Check. Check the liquid level in each container to see whether liquid was lost during transport. If a noticeable amount of leakage occurred, either void the sample or use methods subject to the approval of the Administrator to account for the losses.

11.2 Sample Preparation. Treat each sample as follows:

11.2.1 Container No. 1 (Impingers and Probe). Carefully transfer the contents of Container No. 1 into a 1000-ml volumetric flask, and adjust the volume to exactly 1000 ml with water.

11.2.2 Dilutions. Pipet a 2-ml aliquot from the diluted sample from Section 11.2.1 into a 250-ml volumetric flask. Add 10 ml of 5 percent  $H_2SO_4$ , and adjust the volume to exactly 250 ml with water. This solution is stable for at least 72 hours.

## Note: The dilution factor will be 250/2 for this solution.

11.3 Analysis. Calibrate the analytical equipment and develop a calibration curve as outlined in Sections 10.3 through 10.6.

11.3.1 Mercury Samples. Repeat the procedure used to establish the calibration curve with an appropriately sized aliquot (1 to 5 ml) of the diluted sample (from Section 11.2.2) until two consecutive peak heights agree within 3 percent of their average value. The peak maximum of an aliquot (except the 5-ml aliquot) must be greater than 10 percent of the recorder full scale. If the peak maximum of a 1.0-ml aliquot is off scale on the recorder, further dilute the original source sample to bring the Hg concentration into the calibration range of the spectrophotometer.

11.3.2 Run a blank and standard at least after every five samples to check the spectrophotometer calibration. The peak height of the blank must pass through a point no further from the origin than ±2 percent of the recorder full scale. The difference between the measured concentration of the standard (the product of the corrected peak height and the reciprocal of the least squares slope) and the actual concentration of the standard must be less than 7 percent, or recalibration of the analyzer is required.

11.3.3 Check for Matrix Effects (optional). Use the Method of Standard Additions as follows to check at least one sample from each source for matrix effects on the Hg results. The Method of Standard Additions procedures described on pages 9–4 and 9–5 of the section entitled "General Information" of the Perkin Elmer Corporation Atomic Absorption Spectrophotometry Manual, Number 303–0152 (Reference 16 in Section 16.0) are recommended. If the results of the Method of Standard Additions procedure used on the single source sample do not agree to within ±5 percent of the value obtained by the routine atomic absorption analysis, then reanalyze all samples from the source using the Method of Standard Additions procedure.

11.4 Container No. 2 (Silica Gel). Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. (This step may be conducted in the field.)

12.0 Data Analysis and Calculations

Carry out calculations, retaining at least one extra decimal significant figure beyond that of the acquired data. Round off figures only after the final calculation. Other forms of the equations may be used as long as they give equivalent results.

12.1 Average Dry Gas Meter Temperature and Average Orifice Pressure Drop, Dry Gas Volume, Volume of Water Vapor Condensed, Moisture Content, and Isokinetic Variation. Same as Method 5, Sections 12.2 through 12.5 and 12.11, respectively.

12.2 Stack Gas Velocity. Using the data from this test and Equation 2-9 of Method 2, calculate the average stack gas velocity vs.

12.3 Total Mercury.

12.3.1 For each source sample, correct the average maximum absorbance of the two consecutive samples whose peak heights agree within 3 percent of their average for the contribution of the solution blank (see Section 10.6.3). Use the calibration curve and these corrected averages to determine the final total weight of Hg in ng in the aeration cell for each source sample.

12.3.2 Correct for any dilutions made to bring the sample into the working range of the spectrophotometer. Then calculate the Hg in the original solution,  $m_{Hg}$ , as follows:

$$m_{Hg} = \left[C_{Hg(AC)}(DF)(V_{f})(10^{-3})\right]/S$$
 Eq. 101-1

Where:

CHg(AC)= Total ng of Hg in aliquot analyzed (reagent blank subtracted).

DF = Dilution factor for the Hg-containing solution (before adding to the aeration cell; e.g., DF = 250/2 if the source samples were diluted as described in Section 11.2.2)

V<sub>f</sub>= Solution volume of original sample, 1000 ml for samples diluted as described in Section 11.2.1.

 $10^{-3}$ = Conversion factor, µg/ng.

S = Aliguot volume added to aeration cell, ml.

12.4 Mercury Emission Rate. Calculate the daily Hg emission rate, R, using Equation 101–2. For continuous operations, the operating time is equal to 86,400 seconds per day. For cyclic operations, use only the time per day each stack is in operation. The total Hg emission rate from a source will be the summation of results from all stacks.

$$R = \frac{Km_{Hg}V_{s}A_{s}\left(86,400\times10^{-6}\right)}{\left[V_{m(std)} + V_{w(std)}\right]\left(T_{s}/P_{s}\right)} \qquad \text{Eq. 101-2}$$

Where:

K<sub>1</sub>= 0.3858 °K/mm Hg for metric units.

K<sub>1</sub>= 17.64 °R/in. Hg for English units.

K<sub>3</sub>= 10<sup>-6</sup>g/µg for metric units.

= 2.2046 "  $\times$  10<sup>-9</sup>lb/µg for English units.

P<sub>s</sub>= Absolute stack gas pressure, mm Hg (in. Hg).

t = Daily operating time, sec/day.

T<sub>s</sub>= Absolute average stack gas temperature, °K (°R).

Vm(std)= Dry gas sample volume at standard conditions, scm (scf).

Vw(std)= Volume of water vapor at standard conditions, scm (scf).

12.5 Determination of Compliance. Each performance test consists of three repetitions of the applicable test method. For the purpose of determining compliance with an applicable national emission standard, use the average of the results of all repetitions.

13.0 Method Performance

The following estimates are based on collaborative tests, wherein 13 laboratories performed duplicate analyses on two Hg-containing samples from a chlor-alkali plant and on one laboratory-prepared sample of known Hg concentration. The sample concentrations ranged from 2 to 65 µg Hg/ml.

13.1 Precision. The estimated intra-laboratory and inter-laboratory standard deviations are 1.6 and 1.8 µg Hg/ml, respectively.

13.2 Accuracy. The participating laboratories that analyzed a 64.3 µg Hg/ml (in 0.1 M ICI) standard obtained a mean of 63.7 µg Hg/ml.

13.3 Analytical Range. After initial dilution, the range of this method is 0.5 to 120 µg Hg/ml. The upper limit can be extended by further dilution of the sample.

14.0 Pollution Prevention.[Reserved]

15.0 Waste Management.[Reserved]

16.0 References

Same as Method 5, Section 17.0, References 1-3, 5, and 6, with the addition of the following:

1. Determining Dust Concentration in a Gas Stream. ASME Performance Test Code No. 27. New York, NY. 1957.

2. DeVorkin, Howard, et al. Air Pollution Source Testing Manual. Air Pollution Control District. Los Angeles, CA. November 1963.

3. Hatch, W.R., and W.I. Ott. Determination of Sub-Microgram Quantities of Mercury by Atomic Absorption Spectrophotometry. Anal. Chem. 40:2085–87. 1968.

4. Mark, L.S. Mechanical Engineers' Handbook. McGraw-Hill Book Co., Inc. New York, NY. 1951.

5. Western Precipitation Division of Joy Manufacturing Co. Methods for Determination of Velocity, Volume, Dust and Mist Content of Gases. Bulletin WP–50. Los Angeles, CA. 1968.

6. Perry, J.H. Chemical Engineers' Handbook. McGraw-Hill Book Co., Inc. New York, NY. 1960.

7. Shigehara, R.T., W.F. Todd, and W.S. Smith. Significance of Errors in Stack Sampling Measurements. Stack Sampling News. 1 (3):6–18. September 1973.

8. Smith, W.S., R.T. Shigehara, and W.F. Todd. A Method of Interpreting Stack Sampling Data. Stack Sampling News. 1 (2):8–17. August 1973.

9. Standard Method for Sampling Stacks for Particulate Matter. In: 1971 Annual Book of ASTM Standards, Part 23. ASTM Designation D 2928–71. Philadelphia, PA 1971.

10. Vennard, J.K. Elementary Fluid Mechanics. John Wiley and Sons, Inc. New York. 1947.

11. Mitchell, W.J. and M.R. Midgett. Improved Procedure for Determining Mercury Emissions from Mercury Cell Chlor-Alkali Plants. J. APCA. 26:674–677. July 1976.

12. Shigehara, R.T. Adjustments in the EPA Nomograph for Different Pitot Tube Coefficients and Dry Molecular Weights. Stack Sampling News. 2:4–11. October 1974.

13. Vollaro, R.F. Recommended Procedure for Sample Traverses in Ducts Smaller than 12 Inches in Diameter. U.S. Environmental Protection Agency, Emission Measurement Branch. Research Triangle Park, NC. November 1976.

14. Klein, R. and C. Hach. Standard Additions: Uses and Limitation in Spectrophotometric Measurements. Amer. Lab. 9:21. 1977.

15. Perkin Elmer Corporation. Analytical Methods for Atomic Absorption Spectrophotometry. Norwalk, Connecticut. September 1976.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

View or download PDF










Method 101A-Determination of Particulate and Gaseous Mercury Emissions From Sewage Sludge Incinerators

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from methods in appendix A to 40 CFR part 60 and in this part. Therefore, to obtain reliable results, persons using this method should also have a thorough knowledge of at least the following additional test methods: Methods 1, Method 2, Method 3, and Method 5 of part 60 (appendix A), and Method 101 part 61 (appendix B).

1.0 Scope and Application

## 1.1 Analytes.

Analyte	CAS No.	Sensitivity
Mercury (Hg)	7439–97–6	Dependent upon spectrophotometer and recorder.

1.2 Applicability. This method is applicable for the determination of Hg emissions from sewage sludge incinerators and other sources as specified in an applicable subpart of the regulations.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 Particulate and gaseous Hg emissions are withdrawn isokinetically from the source and are collected in acidic potassium permanganate (KMnO<sub>4</sub>) solution. The Hg collected (in the mercuric form) is reduced to elemental Hg, which is then aerated from the solution into an optical cell and measured by atomic absorption spectrophotometry.

3.0 Definitions.[Reserved]

4.0 Interferences

4.1 Sample Collection. Excessive oxidizable organic matter in the stack gas prematurely depletes the KMnO<sub>4</sub>solution and thereby prevents further collection of Hg.

4.2 Analysis. Condensation of water vapor on the optical cell windows causes a positive interference.

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

5.2 Corrosive Reagents. The following reagents are hazardous. Personal protective equipment and safe procedures are useful in preventing chemical splashes. If contact occurs, immediately flush with copious amounts of water for at least 15 minutes. Remove clothing under shower and decontaminate. Treat residual chemical burns as thermal burns.

5.2.1 Hydrochloric Acid (HCl). Highly toxic. Vapors are highly irritating to eyes, skin, nose, and lungs, causing severe damage. May cause bronchitis, pneumonia, or edema of lungs. Exposure to concentrations of 0.13 to 0.2 percent can be lethal to humans in a few minutes. Provide ventilation to limit exposure. Reacts with metals, producing hydrogen gas.

5.2.2 Nitric Acid (HNO<sub>3</sub>). Highly corrosive to eyes, skin, nose, and lungs. Vapors cause bronchitis, pneumonia, or edema of lungs. Reaction to inhalation may be delayed as long as 30 hours and still be fatal. Provide ventilation to limit exposure. Strong oxidizer. Hazardous reaction may occur with organic materials such as solvents.

5.2.3 Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>). Rapidly destructive to body tissue. Will cause third degree burns. Eye damage may result in blindness. Inhalation may be fatal from spasm of the larynx, usually within 30 minutes. May cause lung tissue damage with edema. 3 mg/m<sup>3</sup> will cause lung damage in uninitiated. 1 mg/m<sup>3</sup> for 8 hours will cause lung damage or, in higher concentrations, death. Provide ventilation to limit inhalation. Reacts violently with metals and organics.

5.3 Chlorine Evolution. Hydrochloric acid reacts with KMnO<sub>4</sub>to liberate chlorine gas. Although this is a minimal concern when small quantities of HCI (5–10 ml) are used in the impinger rinse, a potential safety hazard may still exist. At sources that emit higher concentrations of oxidizable materials (*e.g.*, power plants), more HCI may be required to remove the larger amounts of brown deposit formed in the impingers. In such cases, the potential safety hazards due to sample container pressurization are greater, because of the larger volume of HCI rinse added to the recovered sample. These hazards are eliminated by storing and analyzing the HCI impinger wash separately from the permanganate impinger sample.

6.0 Equipment and Supplies

6.1 Sample Collection and Sample Recovery. Same as Method 101, Sections 6.1 and 6.2, respectively, with the following exceptions:

6.1.1 Probe Liner. Same as in Method 101, Section 6.1.2, except that if a filter is used ahead of the impingers, the probe heating system must be used to minimize the condensation of gaseous Hg.

6.1.2 Filter Holder (Optional). Borosilicate glass with a rigid stainless-steel wire-screen filter support (do not use glass frit supports) and a silicone rubber or Teflon gasket, designed to provide a positive seal against leakage from outside or around the filter. The filter holder must be equipped with a filter heating system capable of maintaining a temperature around the filter holder of 120 ±14 °C (248 ±25 °F) during sampling to minimize both water and gaseous Hg condensation. A filter may also be used in cases where the stream contains large quantities of particulate matter.

6.2 Sample Analysis. Same as Method 101, Section 6.3, with the following additions and exceptions:

6.2.1 Volumetric Pipets. Class A; 1-, 2-, 3-, 4-, 5-, 10-, and 20-ml.

6.2.2 Graduated Cylinder. 25-ml.

6.2.3 Steam Bath.

6.2.4 Atomic Absorption Spectrophotometer or Equivalent. Any atomic absorption unit with an open sample presentation area in which to mount the optical cell is suitable. Instrument settings recommended by the particular manufacturer should be followed. Instruments designed specifically for the measurement of mercury using the cold-vapor technique are commercially available and may be substituted for the atomic absorption spectrophotometer.

6.2.5 Optical Cell. Alternatively, a heat lamp mounted above the cell or a moisture trap installed upstream of the cell may be used.

6.2.6 Aeration Cell. Alternatively, aeration cells available with commercial cold vapor instrumentation may be used.

6.2.7 Aeration Gas Cylinder. Nitrogen, argon, or dry, Hg-free air, equipped with a single-stage regulator. Alternatively, aeration may be provided by a peristaltic metering pump. If a commercial cold vapor instrument is used, follow the manufacturer's recommendations.

7.0 Reagents and Standards

Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise, use the best available grade.

7.1 Sample Collection and Recovery. The following reagents are required for sample collection and recovery:

7.1.1 Water. Deionized distilled, to conform to ASTM D 1193–77 or 91 Type 1. If high concentrations of organic matter are not expected to be present, the analyst may eliminate the KMnO<sub>4</sub>test for oxidizable organic matter. Use this water in all dilutions and solution preparations.

7.1.2 Nitric Acid, 50 Percent (V/V). Mix equal volumes of concentrated HNO<sub>3</sub> and water, being careful to add the acid to the water slowly.

7.1.3 Silica Gel. Indicating type, 6 to 16 mesh. If previously used, dry at 175 °C (350 °F) for 2 hours. New silica gel may be used as received.

7.1.4 Filter (Optional). Glass fiber filter, without organic binder, exhibiting at least 99.95 percent efficiency on 0.3-µm dioctyl phthalate smoke particles. The filter in cases where the gas stream contains large quantities of particulate matter, but blank filters should be analyzed for Hg content.

7.1.5 Sulfuric Acid, 10 Percent (V/V). Carefully add and mix 100 ml of concentrated H<sub>2</sub>SO<sub>4</sub>to 900 ml of water.

7.1.6 Absorbing Solution, 4 Percent KMnO<sub>4</sub>(W/V). Prepare fresh daily. Dissolve 40 g of KMnO<sub>4</sub>in sufficient 10 percent H<sub>2</sub>SO<sub>4</sub>to make 1 liter. Prepare and store in glass bottles to prevent degradation.

7.1.7 Hydrochloric Acid, 8 N. Carefully add and mix 67 ml of concentrated HCl to 33 ml of water.

7.2 Sample Analysis. The following reagents and standards are required for sample analysis:

7.2.1 Water. Same as in Section 7.1.1.

7.2.2 Tin (II) Solution. Prepare fresh daily, and keep sealed when not being used. Completely dissolve 20 g of tin (II) chloride (or 25 g of tin (II) sulfate) crystals (Baker Analyzed reagent grade or any other brand that will give a clear solution) in 25 ml of concentrated HCI. Dilute to 250 ml with water. Do not substitute HNO<sub>3</sub>H2SO<sub>4</sub>, or other strong acids for the HCI.

7.2.3 Sodium Chloride-Hydroxylamine Solution. Dissolve 12 g of sodium chloride and 12 g of hydroxylamine sulfate (or 12 g of hydroxylamine hydrochloride) in water and dilute to 100 ml.

7.2.4 Hydrochloric Acid, 8 N. Same as Section 7.1.7.

7.2.5 Nitric Acid, 15 Percent (V/V). Carefully add 15 ml HNO<sub>3</sub>to 85 ml of water.

7.2.6 Antifoam B Silicon Emulsion. J.T. Baker Company (or equivalent).

7.2.7 Mercury Stock Solution, 1 mg Hg/ml. Prepare and store all Hg standard solutions in borosilicate glass containers. Completely dissolve 0.1354 g of Hg (II) chloride in 75 ml of water. Add 10 ml of concentrated HNO<sub>3</sub>, and adjust the volume to exactly 100 ml with water. Mix thoroughly. This solution is stable for at least one month.

7.2.8 Intermediate Hg Standard Solution, 10 µg/ml. Prepare fresh weekly. Pipet 5.0 ml of the Hg stock solution (Section 7.2.7) into a 500 ml volumetric flask, and add 20 ml of 15 percent HNO<sub>3</sub>solution. Adjust the volume to exactly 500 ml with water. Thoroughly mix the solution.

7.2.9 Working Hg Standard Solution, 200 ng Hg/ml. Prepare fresh daily. Pipet 5.0 ml from the "Intermediate Hg Standard Solution" (Section 7.2.8) into a 250-ml volumetric flask. Add 5 ml of 4 percent KMnO₄absorbing solution and 5 ml of 15 percent HNO₃. Adjust the volume to exactly 250 ml with water. Mix thoroughly.

7.2.10 Potassium Permanganate, 5 Percent (W/V). Dissolve 5 g of KMnO₄in water and dilute to 100 ml.

7.2.11 Filter. Whatman No. 40, or equivalent.

8.0 Sample Collection, Preservation, Transport, and Storage

Same as Method 101, Section 8.0, with the exception of the following:

8.1 Preliminary Determinations. Same as Method 101, Section 8.2, except that the liberation of free iodine in the first impinger due to high Hg or sulfur dioxide concentrations is not applicable. In this method, high oxidizable organic content may make it impossible to sample for the desired minimum time. This problem is indicated by the complete bleaching of the purple color of the KMnO<sub>4</sub>solution. In cases where an excess of water condensation is encountered, collect two runs to make one sample, or add an extra impinger in front of the first impinger (also containing acidified KMnO<sub>4</sub>solution).

8.2 Preparation of Sampling Train. Same as Method 101, Section 8.3, with the exception of the following:

8.2.1 In this method, clean all the glass components by rinsing with 50 percent HNO<sub>3</sub>, tap water, 8 N HCI, tap water, and finally with deionized distilled water. Then place 50 ml of absorbing solution in the first impinger and 100 ml in each of the second and third impingers.

8.2.2 If a filter is used, use a pair of tweezers to place the filter in the filter holder. Be sure to center the filter, and place the gasket in the proper position to prevent the sample gas stream from bypassing the filter. Check the filter for tears after assembly is completed. Be sure also to set the filter heating system at the desired operating temperature after the sampling train has been assembled.

8.3 Sampling Train Operation. In addition to the procedure outlined in Method 101, Section 8.5, maintain a temperature around the filter (if applicable) of 120 ±14 °C (248 ±25 °F).

8.4 Sample Recovery. Same as Method 101, Section 8.7, with the exception of the following:

8.4.1 Transfer the probe, impinger assembly, and (if applicable) filter assembly to the cleanup area.

8.4.2 Treat the sample as follows:

8.4.2.1 Container No. 1 (Impinger, Probe, and Filter Holder) and, if applicable, Container No. 1A (HCI rinse).

8.4.2.1.1 Using a graduated cylinder, measure the liquid in the first three impingers to within 1 ml. Record the volume of liquid present (*e.g.*, see Figure 5–6 of Method 5). This information is needed to calculate the moisture content of the effluent gas. (Use only graduated cylinder and glass storage bottles that have been precleaned as in Section 8.2.1.) Place the contents of the first three impingers (four if an extra impinger was added as described in Section 8.1) into a 1000-ml glass sample bottle labeled Container No. 1.

Note: If a filter is used, remove the filter from its holder as outlined under Section 8.4.3.

8.4.2.1.2 Taking care that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover the Hg (and any condensate) from the probe nozzle, probe fitting, probe liner, front half of the filter holder (if applicable), and impingers as follows: Rinse these components with a total of 400 ml (350 ml if an extra impinger was added as described in Section 8.1) of fresh absorbing solution, carefully assuring removal of all loose particulate matter from the impingers; add all washings to the 1000 ml glass sample bottle. To remove any residual brown deposits on the glassware following the permanganate rinse, rinse with approximately 100 ml of water, carefully assuring removal of all loose particulate matter from the impingers. Add this rinse to Container No. 1.

8.4.2.1.3 If no visible deposits remain after this water rinse, do not rinse with 8 N HCl. If deposits do remain on the glassware after the water rinse, wash impinger walls and stems with 25 ml of 8 N HCl, and place the wash in a separate container labeled Container No. 1A as follows: Place 200 ml of water in a sample container labeled Container No. 1A. Wash the impinger walls and stem with the HCl by turning the impinger on its side and rotating it so that the HCl contacts all inside surfaces. Pour the HCl wash carefully with stirring into Container No. 1A.

8.4.2.1.4 After all washings have been collected in the appropriate sample container(s), tighten the lid(s) on the container(s) to prevent leakage during shipment to the laboratory. Mark the height of the fluid level to allow subsequent determination of whether leakage has occurred during transport. Label each container to identify its contents clearly.

8.4.3 Container No. 2 (Silica Gel). Same as Method 5, Section 8.7.6.3.

8.4.4 Container No. 3 (Filter). If a filter was used, carefully remove it from the filter holder, place it in a 100-ml glass sample bottle, and add 20 to 40 ml of absorbing solution. If it is necessary to fold the filter, be sure that the particulate cake is inside the fold. Carefully transfer to the 100-ml sample bottle any particulate matter and filter fibers that adhere to the filter holder gasket by using a dry Nylon bristle brush and a sharp-edged blade. Seal the container. Label the container to identify its contents clearly. Mark the height of the fluid level to allow subsequent determination of whether leakage has occurred during transport.

8.4.5 Container No. 4 (Filter Blank). If a filter was used, treat an unused filter from the same filter lot as that used for sampling according to the procedures outlined in Section 8.4.4.

8.4.6 Container No. 5 (Absorbing Solution Blank). Place 650 ml of 4 percent KMnO₄absorbing solution in a 1000-ml sample bottle. Seal the container.

8.4.7 Container No. 6 (HCI Rinse Blank). Place 200 ml of water in a 1000-ml sample bottle, and add 25 ml of 8 N HCl carefully with stirring. Seal the container. Only one blank sample per 3 runs is required.

9.0 Quality Control

### 9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
8.0,	Sampling equipment leak-checks and	Ensure accuracy and precision of sampling

10.0	calibration	measurements.
10.2	Spectrophotometer calibration	Ensure linearity of spectrophotometer response to standards.
11.3.3	Check for matrix effects	Eliminate matrix effects.

9.2 Volume Metering System Checks. Same as Method 5, Section 9.2.

## 10.0 Calibration and Standardization

Same as Method 101, Section 10.0, with the following exceptions:

10.1 Optical Cell Heating System Calibration. Same as in Method 101, Section 10.4, except use a-25 ml graduated cylinder to add 25 ml of water to the bottle section of the aeration cell.

10.2 Spectrophotometer and Recorder Calibration.

10.2.1 The Hg response may be measured by either peak height or peak area.

Note: The temperature of the solution affects the rate at which elemental Hg is released from a solution and, consequently, it affects the shape of the absorption curve (area) and the point of maximum absorbance (peak height). To obtain reproducible results, all solutions must be brought to room temperature before use.

10.2.2 Set the spectrophotometer wave length at 253.7 nm, and make certain the optical cell is at the minimum temperature that will prevent water condensation. Then set the recorder scale as follows: Using a 25-ml graduated cylinder, add 25 ml of water to the aeration cell bottle. Add three drops of Antifoam B to the bottle, and then pipet 5.0 ml of the working Hg standard solution into the aeration cell.

Note: Always add the Hg-containing solution to the aeration cell after the 25 ml of water.

10.2.3 Place a Teflon-coated stirring bar in the bottle. Add 5 ml of absorbing solution to the aeration bottle, and mix well. Before attaching the bottle section to the bubbler section of the aeration cell, make certain that (1) the aeration cell exit arm stopcock (Figure 101–3 of Method 101) is closed (so that Hg will not prematurely enter the optical cell when the reducing agent is being added) and (2) there is no flow through the bubbler. If conditions (1) and (2) are met, attach the bottle section to the bubbler section of the aeration cell. Add sodium chloride-hydroxylamine in 1 ml increments until the solution is colorless. Now add 5 ml of tin (II) solution to the aeration bottle through the side arm, and immediately stopper the side arm. Stir the solution for 15 seconds, turn on the recorder, open the aeration cell exit arm stopcock, and immediately initiate aeration with continued stirring. Determine the maximum absorbance of the standard, and set this value to read 90 percent of the recorder full scale.

## 11.0 Analytical Procedure

11.1 Sample Loss Check. Check the liquid level in each container to see if liquid was lost during transport. If a noticeable amount of leakage occurred, either void the sample or use methods subject to the approval of the Administrator to account for the losses.

11.2 Sample Preparation. Treat sample containers as follows:

11.2.1 Containers No. 3 and No. 4 (Filter and Filter Blank).

11.2.1.1 If a filter is used, place the contents, including the filter, of Containers No. 3 and No. 4 in separate 250-ml beakers, and heat the beakers on a steam bath until most of the liquid has evaporated. Do not heat to dryness. Add 20 ml of concentrated HNO<sub>3</sub>to the beakers, cover them with a watch glass, and heat on a hot plate at 70 °C (160 °F) for 2 hours. Remove from the hot plate.

11.2.1.2 Filter the solution from digestion of the Container No. 3 contents through Whatman No. 40 filter paper, and save the filtrate for addition to the Container No. 1 filtrate as described in Section 11.2.2. Discard the filter paper.

11.2.1.3 Filter the solution from digestion of the Container No. 4 contents through Whatman No. 40 filter paper, and save the filtrate for addition to Container No. 5 filtrate as described in Section 11.2.3 below. Discard the filter paper.

11.2.2 Container No. 1 (Impingers, Probe, and Filter Holder) and, if applicable, No. 1A (HCI rinse).

11.2.2.1 Filter the contents of Container No. 1 through Whatman No. 40 filter paper into a 1 liter volumetric flask to remove the brown manganese dioxide (MnO<sub>2</sub>) precipitate. Save the filter for digestion of the brown MnO<sub>2</sub>precipitate. Add the sample filtrate from Container No. 3 to the 1-liter volumetric flask, and dilute to volume with water. If the combined filtrates are greater than 1000 ml, determine the volume to the nearest ml and make the appropriate corrections for blank subtractions. Mix thoroughly. Mark the filtrate as analysis Sample No. A.1 and analyze for Hg within 48 hr of the filtration step. Place the saved filter, which was used to remove the brown MnO<sub>2</sub>precipitate, into an appropriate sized container. In a laboratory hood, add 25 ml of 8 N HCl to the filter and allow to digest for a minimum of 24 hours at room temperature.

11.2.2.2 Filter the contents of Container 1A through Whatman No. 40 filter paper into a 500-ml volumetric flask. Then filter the digestate of the brown MnO<sub>2</sub>precipitate from Container No. 1 through Whatman No. 40 filter paper into the same 500-ml volumetric flask, and dilute to volume with water. Mark this combined 500 ml dilute solution as analysis Sample No. A.2. Discard the filters.

11.2.3 Container No. 5 (Absorbing Solution Blank) and No. 6 (HCI Rinse Blank).

11.2.3.1 Treat Container No. 5 as Container No. 1 (as described in Section 11.2.2), except substitute the filter blank filtrate from Container No. 4 for the sample filtrate from Container No. 3, and mark as Sample A.1 Blank.

11.2.3.2 Treat Container No. 6 as Container No. 1A, (as described in Section 11.2.2, except substitute the filtrate from the digested blank MnO<sub>2</sub>precipitate for the filtrate from the digested sample MnO<sub>2</sub>precipitate, and mark as Sample No. A.2 Blank.

Note: When analyzing samples A.1 Blank and HCI A.2 Blank, always begin with 10 ml aliquots. This applies specifically to blank samples.

11.3 Analysis. Calibrate the analytical equipment and develop a calibration curve as outlined in Section 10.0.

11.3.1 Mercury Samples. Then repeat the procedure used to establish the calibration curve with appropriately sized aliquots (1 to 10 ml) of the samples (from Sections 11.2.2 and 11.2.3) until two consecutive peak heights agree within 3 percent of their average value. If the 10 ml sample is below the detectable limit, use a larger aliquot (up to 20 ml), but decrease the volume of water added to the aeration cell accordingly to prevent the solution volume from exceeding the capacity of the aeration bottle. If the peak maximum of a 1.0 ml aliquot is off scale, further dilute the original sample to bring the Hg concentration into the calibration range of the spectrophotometer. If the Hg content of the absorbing solution and filter blank is below the working range of the analytical method, use zero for the blank.

11.3.2 Run a blank and standard at least after every five samples to check the spectrophotometer calibration; recalibrate as necessary.

11.3.3 Check for Matrix Effects (optional). Same as Method 101, Section 11.3.3.

12.0 Data Analysis and Calculations

Note: Carry out calculations, retaining at least one extra decimal significant figure beyond that of the acquired data. Round off figures only after the final calculation. Other forms of the equations may be used as long as they give equivalent results.

12.1 Nomenclature.

C(fltr)Hg= Total ng of Hg in aliquot of KMnO<sub>4</sub>filtrate and HNO<sub>3</sub>digestion of filter analyzed (aliquot of analysis Sample No. A.1).

C(fltr blk)Hg= Total ng of Hg in aliquot of KMnO<sub>4</sub>blank and HNO<sub>3</sub>digestion of blank filter analyzed (aliquot of analysis Sample No. A.1 blank).

C(HC1 blk)Hg= Total ng of Hg analyzed in aliquot of the 500-ml analysis Sample No. HCl A.2 blank.

C(HCl)Hg= Total ng of Hg analyzed in the aliquot from the 500-ml analysis Sample No. HCl A.2.

DF = Dilution factor for the HCI-digested Hg-containing solution, Analysis Sample No. "HCI A.2."

DF<sub>bik</sub>= Dilution factor for the HCI-digested Hg containing solution, Analysis Sample No. "HCI A.2 blank." (Refer to sample No. "HCI A.2" dilution factor above.)

m(fltr)Hg= Total blank corrected µg of Hg in KMnO<sub>4</sub>filtrate and HNO<sub>3</sub>digestion of filter sample.

m(HCl)Hg= Total blank corrected µg of Hg in HCl rinse and HCl digestate of filter sample.

m<sub>Hg</sub>= Total blank corrected Hg content in each sample, µg.

S = Aliquot volume of sample added to aeration cell, ml.

S<sub>blk</sub>= Aliquot volume of blank added to aeration cell, ml.

Vf(blk)= Solution volume of blank sample, 1000 ml for samples diluted as described in Section 11.2.2.

Vf(fltr)= Solution volume of original sample, normally 1000 ml for samples diluted as described in Section 11.2.2.

Vf(HCl)= Solution volume of original sample, 500 ml for samples diluted as described in Section 11.2.1.

10<sup>-3</sup>= Conversion factor, µg/ng.

12.2 Average Dry Gas Meter Temperature and Average Orifice Pressure Drop, Dry Gas Volume, Volume of Water Vapor Condensed, Moisture Content, Isokinetic Variation, and Stack Gas Velocity and Volumetric Flow Rate. Same as Method 5, Sections 12.2 through 12.5, 12.11, and 12.12, respectively.

12.3 Total Mercury.

12.3.1 For each source sample, correct the average maximum absorbance of the two consecutive samples whose peak heights agree within 3 percent of their average for the contribution of the blank. Use the calibration curve and these corrected averages to determine the final total weight of Hg in ng in the aeration cell for each source sample.

12.3.2 Correct for any dilutions made to bring the sample into the working range of the spectrophotometer.

$$m_{(\text{HCI})\text{Hg}} = \frac{\left[C_{(\text{HCI})\text{Hg}}\text{DF}\right]}{S} - \frac{\left[C_{(\text{HCIblk})\text{Hg}}\text{DF}_{bk}\right]}{S_{bk}}Vf_{(\text{HCI})}\left(10^{-3}\right) \qquad \text{Eq. 101A-1}$$

Note: This dilution factor applies only to the intermediate dilution steps, since the original sample volume  $[(V_f)_{HCL}]$  of "HCI A.2" has been factored out in the equation along with the sample aliquot (S). In Eq. 101A–1, the sample aliquot, S, is introduced directly into the aeration cell for analysis according to the procedure outlined in Section 11.3.1. A dilution factor is required only if it is necessary to bring the sample into the analytical instrument's calibration range.

Note: The maximum allowable blank subtraction for the HCI is the lesser of the two following values: (1) the actual blank measured value (analysis Sample No. HCI A.2 blank), or (2) 5% of the Hg content in the combined HCI rinse and digested sample (analysis Sample No. HCI A.2).

$$m_{(\mathbf{fir})\mathbf{Hg}} = \frac{\left[C_{(\mathbf{fir})\mathbf{Hg}} DF V_{\mathbf{f}(\mathbf{fir})}\right]}{S} - \frac{\left[C_{(\mathbf{fir}b\mathbf{k})\mathbf{Hg}} DF_{\mathbf{b}\mathbf{k}} V_{\mathbf{f}(\mathbf{b}\mathbf{k})}\right]}{S_{\mathbf{b}\mathbf{k}}} \qquad \text{Eq. 101A-2}$$

Note: The maximum allowable blank subtraction for the HCl is the lesser of the two following values: (1) the actual blank measured value (analysis Sample No. "A.1 blank"), or (2) 5% of the Hg content in the filtrate (analysis Sample No. "A.1").

 $m_{Hg} = m_{(HCI)Hg} + m_{(fhr)Hg} = Eq. 101A-3$ 

12.3 Mercury Emission Rate. Same as Method 101, Section 12.3.

12.4 Determination of Compliance. Same as Method 101, Section 12.4.

13.0 Method Performance

13.1 Precision. Based on eight paired-train tests, the intra-laboratory standard deviation was estimated to be 4.8  $\mu$ g/ml in the concentration range of 50 to 130  $\mu$ g/m3.

13.2 Bias. [Reserved]

13.3 Range. After initial dilution, the range of this method is 20 to 800 ng Hg/ml. The upper limit can be extended by further dilution of the sample.

14.0 Pollution Prevention[Reserved]

- 15.0 Waste Management[Reserved]
- 16.0 References

Same as Section 16.0 of Method 101, with the addition of the following:

1. Mitchell, W.J., *et al.* Test Methods to Determine the Mercury Emissions from Sludge Incineration Plants. U.S. Environmental Protection Agency. Research Triangle Park, NC. Publication No. EPA–600/4–79–058. September 1979.

2. Wilshire, Frank W., *et al.* Reliability Study of the U.S. EPA's Method 101A—Determination of Particulate and Gaseous Mercury Emissions. U.S. Environmental Protection Agency. Research Triangle Park, NC. Report No. 600/D–31/219 AREAL 367, NTIS Acc No. PB91–233361.

3. Memorandum from William J. Mitchell to Roger T. Shigehara discussing the potential safety hazard in Section 7.2 of Method 101A. February 28, 1990.

17.0 Tables, Diagrams, Flowcharts, And Validation Data[Reserved]

Method 102—Determination of Particulate and Gaseous Mercury Emissions From Chlor-Alkali Plants (Hydrogen Streams)

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part and in appendix A to 40 CFR part 60. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3, Method 5, and Method 101.

1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.	Sensitivity
Mercury (Hg)	7439–97–6	Dependent upon recorder and spectrophotometer.

1.2 Applicability. This method is applicable for the determination of Hg emissions, including both particulate and gaseous Hg, from chlor-alkali plants and other sources (as specified in the regulations) where the carrier-gas stream in the duct or stack is principally hydrogen.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

## 2.0 Summary of Method

2.1 Particulate and gaseous Hg emissions are withdrawn isokinetically from the source and collected in acidic iodine monochloride (ICI) solution. The Hg collected (in the mercuric form) is reduced to elemental Hg, which is then aerated from the solution into an optical cell and measured by atomic absorption spectrophotometry.

3.0 Definitions[Reserved]

4.0 Interferences

Same as Method 101, Section 4.2.

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

5.2 Corrosive Reagents. Same as Method 101, Section 5.2.

5.3 Explosive Mixtures. The sampler must conduct the source test under conditions of utmost safety because hydrogen and air mixtures are explosive. Since the sampling train essentially is leakless, attention to safe operation can be concentrated at the inlet and outlet. If a leak does occur, however, remove the meter box cover to avoid a possible explosive mixture. The following specific precautions are recommended:

5.3.1 Operate only the vacuum pump during the test. The other electrical equipment, e.g., heaters, fans, and timers, normally are not essential to the success of a hydrogen stream test.

5.3.2 Seal the sample port to minimize leakage of hydrogen from the stack.

5.3.3 Vent sampled hydrogen at least 3 m (10 ft) away from the train. This can be accomplished by attaching a 13-mm (0.50-in.) ID Tygon tube to the exhaust from the orifice meter.

Note: A smaller ID tubing may cause the orifice meter calibration to be erroneous. Take care to ensure that the exhaust line is not bent or pinched.

# 6.0 Equipment and Supplies

Same as Method 101, Section 6.0, with the exception of the following:

6.1 Probe Heating System. Do not use, unless otherwise specified.

6.2 Glass Fiber Filter. Do not use, unless otherwise specified.

7.0 Reagents and Standards

Same as Method 101, Section 7.0.

8.0 Sample Collection, Preservation, Transport, and Storage

Same as Method 101, Section 8.0, with the exception of the following:

8.1 Setting of Isokinetic Rates.

8.1.1 If a nomograph is used, take special care in the calculation of the molecular weight of the stack gas and in the setting of the nomograph to maintain isokinetic conditions during sampling (Sections 8.1.1.1 through 8.1.1.3 below).

8.1.1.1 Calibrate the meter box orifice. Use the techniques described in APTD-0576 (see Reference 9 in Section 17.0 of Method 5). Calibration of the orifice meter at flow conditions that simulate the conditions at the source is suggested. Calibration should either be done with hydrogen or with some other gas having similar Reynolds Number so that there is similarity between the Reynolds Numbers during calibration and during sampling.

8.1.1.2 The nomograph described in APTD–0576 cannot be used to calculate the C factor because the nomograph is designed for use when the stack gas dry molecular weight is 29 ±4. Instead, the following calculation should be made to determine the proper C factor:

C = 0.00154
$$\Delta H @ C_p^{-2} T_m (P_s/P_m) \frac{(1-B_{ws})^2}{(1-B_{ws})+18B_{ws}}$$
 Eq. 102-1

Where:

B<sub>ws</sub>= Fraction by volume of water vapor in the stack gas.

C<sub>p</sub>= Pitot tube calibration coefficient, dimensionless.

M<sub>d</sub>= Dry molecular weight of stack gas, lb/lb-mole.

Ps= Absolute pressure of stack gas, in. Hg.

P<sub>m</sub>= Absolute pressure of gas at the meter, in. Hg.

T<sub>m</sub>= Absolute temperature of gas at the orifice, °R.

 $\Delta$ H@= Meter box calibration factor obtained in Section 8.1.1.1, in. H<sub>2</sub>O.

 $0.00154 = (in. H_2O/^{\circ}R).$ 

Note: This calculation is left in English units, and is not converted to metric units because nomographs are based on English units.

8.1.1.3 Set the calculated C factor on the operating nomograph, and select the proper nozzle diameter and K factor as specified in APTD–0576. If the C factor obtained in Section 8.1.1.2 exceeds the values specified on the existing operating nomograph, expand the C scale logarithmically so that the values can be properly located.

8.1.2 If a calculator is used to set isokinetic rates, it is suggested that the isokinetic equation presented in Reference 13 in Section 17.0 of Method 101 be consulted.

8.2 Sampling in Small (<12-in. Diameter) Stacks. When the stack diameter (or equivalent diameter) is less than 12 inches, conventional pitot tube-probe assemblies should not be used. For sampling guidelines, see Reference 14 in Section 17.0 of Method 101.

9.0 Quality Control

Same as Method 101, Section 9.0.

10.0 Calibration and Standardizations

Same as Method 101, Section 10.0.

11.0 Analytical Procedure

Same as Method 101, Section 11.0.

12.0 Data Analysis and Calculations

Same as Method 101, Section 12.0.

13.0 Method Performance

Same as Method 101, Section 13.0.

13.1 Analytical Range. After initial dilution, the range of this method is 0.5 to 120 µg Hg/ml. The upper limit can be extended by further dilution of the sample.

14.0 Pollution Prevention.[Reserved]

15.0 Waste Management [Reserved]

16.0 References

Same as Method 101, Section 16.0.

17.0 Tables, Diagrams, Flowcharts, and Validation Data.[Reserved]

Method 103-Beryllium Screening Method

1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.	Sensitivity
Beryllium (Be)	7440-41-7	Dependent upon analytical procedure used.

1.2 Applicability. This procedure details guidelines and requirements for methods acceptable for use in determining Be emissions in ducts or stacks at stationary sources.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 Particulate Be emissions are withdrawn isokinetically from three points in a duct or stack and are collected on a filter. The collected sample is analyzed for Be using an appropriate technique.

# 3.0 Definitions.[Reserved]

## 4.0 Interferences.[Reserved]

## 5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

5.2 Hydrochloric Acid (HCl). Highly corrosive and toxic. Vapors are highly irritating to eyes, skin, nose, and lungs, causing severe damage. May cause bronchitis, pneumonia, or edema of lungs. Exposure to concentrations of 0.13 to 0.2 percent can be lethal to humans in a few minutes. Provide ventilation to limit exposure. Reacts with metals, producing hydrogen gas. Personal protective equipment and safe procedures are useful in preventing chemical splashes. If contact occurs, immediately flush with copious amounts of water at least 15 minutes. Remove clothing under shower and decontaminate. Treat residual chemical burn as thermal burn.

## 6.0 Equipment and Supplies

6.1 Sample Collection. A schematic of the required sampling train configuration is shown in Figure 103–1 in Section 17.0. The essential components of the train are as follows:

6.1.1 Nozzle. Stainless steel, or equivalent, with sharp, tapered leading edge.

## 6.1.2 Probe. Sheathed borosilicate or quartz glass tubing.

6.1.3 Filter. Millipore AA, or equivalent, with appropriate filter holder that provides a positive seal against leakage from outside or around the filter. It is suggested that a Whatman 41, or equivalent, be placed immediately against the back side of the Millipore filter as a guard against breakage of the Millipore. Include the backup filter in the analysis. To be equivalent, other filters shall exhibit at least 99.95 percent efficiency (0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles, and be amenable to the Be analysis procedure. The filter efficiency tests shall be conducted in accordance with ASTM D 2986–71, 78, 95a (incorporated by reference—see §61.18). Test data from the supplier's quality control program are sufficient for this purpose.

6.1.4 Meter-Pump System. Any system that will maintain isokinetic sampling rate, determine sample volume, and is capable of a sampling rate of greater than 14 lpm (0.5 cfm).

6.2 Measurement of Stack Conditions. The following equipment is used to measure stack conditions:

6.2.1 Pitot Tube. Type S, or equivalent, with a constant coefficient (±5 percent) over the working range.

6.2.2 Inclined Manometer, or Equivalent. To measure velocity head to ±10 percent of the minimum value.

6.2.3 Temperature Measuring Device. To measure stack temperature to ±1.5 percent of the minimum absolute stack temperature.

6.2.4 Pressure Measuring Device. To measure stack pressure to ±2.5 mm Hg (0.1 in. Hg).

6.2.5 Barometer. To measure atmospheric pressure to ±2.5 mm Hg (0.1 in. Hg).

6.2.6 Wet and Dry Bulb Thermometers, Drying Tubes, Condensers, or Equivalent. To determine stack gas moisture content to ±1 percent.

6.3 Sample Recovery.

6.3.1 Probe Cleaning Equipment. Probe brush or cleaning rod at least as long as probe, or equivalent. Clean cotton balls, or equivalent, should be used with the rod.

6.3.2 Leakless Glass Sample Bottles. To contain sample.

6.4 Analysis. All equipment necessary to perform an atomic absorption, spectrographic, fluorometric, chromatographic, or equivalent analysis.

7.0 Reagents and Standards

7.1 Sample Recovery.

7.1.1 Water. Deionized distilled, to conform to ASTM D 1193-77, 91 (incorporated by reference-see §61.18), Type 3.

7.1.2 Acetone. Reagent grade.

7.1.3 Wash Acid, 50 Percent (V/V) Hydrochloric Acid (HCl). Mix equal volumes of concentrated HCl and water, being careful to add the acid slowly to the water.

7.2 Analysis. Reagents and standards as necessary for the selected analytical procedure.

8.0 Sample Collection, Preservation, Transport, and Storage

Guidelines for source testing are detailed in the following sections. These guidelines are generally applicable; however, most sample sites differ to some degree and temporary alterations such as stack extensions or expansions often are required to insure the best possible sample site. Further, since Be is hazardous, care should be taken to minimize exposure. Finally, since the total quantity of Be to be collected is quite small, the test must be carefully conducted to prevent contamination or loss of sample.

8.1 Selection of a Sampling Site and Number of Sample Runs. Select a suitable sample site that is as close as practicable to the point of atmospheric emission. If possible, stacks smaller than one foot in diameter should not be sampled.

8.1.1 Ideal Sampling Site. The ideal sampling site is at least eight stack or duct diameters downstream and two diameters upstream from any flow disturbance such as a bend, expansion or contraction. For rectangular cross sections, use Equation 103–1 in Section 12.2 to determine an equivalent diameter,  $D_{e}$ .

8.1.2 Alternate Sampling Site. Some sampling situations may render the above sampling site criteria impractical. In such cases, select an alternate site no less than two diameters downstream and one-half diameter upstream from any point of flow disturbance. Additional sample runs are recommended at any sample site not meeting the criteria of Section 8.1.1.

8.1.3 Number of Sample Runs Per Test. Three sample runs constitute a test. Conduct each run at one of three different points. Select three points that proportionately divide the diameter, or are located at 25, 50, and 75 percent of the diameter from the inside wall. For horizontal ducts, sample on a vertical line through the centroid. For rectangular ducts, sample on a line through the centroid and parallel to a side. If additional sample runs are performed per Section 8.1.2, proportionately divide the duct to accommodate the total number of runs.

8.2 Measurement of Stack Conditions. Using the equipment described in Section 6.2, measure the stack gas pressure, moisture, and temperature to determine the molecular weight of the stack gas. Sound engineering estimates may be made in lieu of direct measurements. Describe the basis for such estimates in the test report.

8.3 Preparation of Sampling Train.

8.3.1 Assemble the sampling train as shown in Figure 103–1. It is recommended that all glassware be precleaned by soaking in wash acid for two hours.

8.3.2 Leak check the sampling train at the sampling site. The leakage rate should not be in excess of 1 percent of the desired sample rate.

8.4 Sampling Train Operation.

8.4.1 For each run, measure the velocity at the selected sampling point. Determine the isokinetic sampling rate. Record the velocity head and the required sampling rate. Place the nozzle at the sampling point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. At the conclusion of the test, record the sampling rate. Again measure the velocity head at the sampling point. The required isokinetic rate at the end of the period should not have deviated more than 20 percent from that originally calculated. Describe the reason for any deviation beyond 20 percent in the test report.

8.4.2 Sample at a minimum rate of 14 liters/min (0.5 cfm). Obtain samples over such a period or periods of time as are necessary to determine the maximum emissions which would occur in a 24-hour period. In the case of cyclic operations, perform sufficient sample runs so as to allow determination or calculation of the emissions that occur over the duration of the cycle. A minimum sampling time of two hours per run is recommended.

8.5 Sample Recovery.

8.5.1 It is recommended that all glassware be precleaned as in Section 8.3. Sample recovery should also be performed in an area free of possible Be contamination. When the sampling train is moved, exercise care to prevent breakage and contamination. Set aside a portion of the acetone used in the sample recovery as a blank for analysis. The total amount of acetone used should be measured for accurate blank correction. Blanks can be eliminated if prior analysis shows negligible amounts.

8.5.2 Remove the filter (and backup filter, if used) and any loose particulate matter from filter holder, and place in a container.

8.5.3 Clean the probe with acetone and a brush or long rod and cotton balls. Wash into the container with the filter. Wash out the filter holder with acetone, and add to the same container.

9.0 Quality Control.[Reserved]

10.0 Calibration and Standardization

10.1 Sampling Train. As a procedural check, compare the sampling rate regulation with a dry gas meter, spirometer, rotameter (calibrated for prevailing atmospheric conditions), or equivalent, attached to the nozzle inlet of the complete sampling train.

10.2 Analysis. Perform the analysis standardization as suggested by the manufacturer of the instrument, or the procedures for the analytical method in use.

11.0 Analytical Procedure

Make the necessary preparation of samples and analyze for Be. Any currently acceptable method (e.g., atomic absorption, spectrographic, fluorometric, chromatographic) may be used.

12.0 Data Analysis and Calculations

12.1 Nomenclature.

 $A_s(avg) = Stack area, m^2 (ft^2).$ 

L = Length.

R = Be emission rate, g/day.

V<sub>s</sub>(avg) = Average stack gas velocity, m/sec (ft/sec).

 $V_{total}$ = Total volume of gas sampled, m<sup>3</sup> (ft<sup>3</sup>).

W = Width.

Wt= Total weight of Be collected, mg.

 $10^{-6}$ = Conversion factor, g/µg.

86,400 = Conversion factor, sec/day.

12.2 Calculate the equivalent diameter, De, for a rectangular cross section as follows:

$$D_e = \frac{2 \cdot L \cdot W}{L + W} \qquad \text{Eq. 103-1}$$

12.3 Calculate the Be emission rate, R, in g/day for each stack using Equation 103–2. For cyclic operations, use only the time per day each stack is in operation. The total Be emission rate from a source is the summation of results from all stacks.

$$R = \frac{W_{\rm t} V_{s(wg)} A_s (86,400) (10^{-6})}{V_{\rm total}} \qquad \text{Eq. 103-2}$$

12.4 Test Report. Prepare a test report that includes as a minimum: A detailed description of the sampling train used, results of the procedural check described in Section 10.1 with all data and calculations made, all pertinent data taken during the test, the basis for any estimates made, isokinetic sampling calculations, and emission results. Include a description of the test site, with a block diagram and brief description of the process, location of the sample points in the stack cross section, and stack dimensions and distances from any point of disturbance.

13.0 Method Performance.[Reserved]

- 14.0 Pollution Prevention.[Reserved]
- 15.0 Waste Management.[Reserved]
- 16.0 References.[Reserved]
- 17.0 Tables, Diagrams, Flow Charts, and Validation Data



Method 104-Determination of Beryllium Emissions From Stationary Sources

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from methods in appendix A to 40 CFR part 60. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3, and Method 5 in appendix A, part 60.

1.0 Scope and Application

## 1.1 Analytes.

Analyte	CAS No.	Sensitivity
Beryllium (Be)	7440-41-7	Dependent upon recorder and spectrophotometer.

1.2 Applicability. This method is applicable for the determination of Be emissions in ducts or stacks at stationary sources. Unless otherwise specified, this method is not intended to apply to gas streams other than those emitted directly to the atmosphere without further processing.

1.3 Data Quality Objectives. Adherences to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

#### 2.0 Summary of Method

2.1 Particulate and gaseous Be emissions are withdrawn isokinetically from the source and are collected on a glass fiber filter and in water. The collected sample is digested in an acid solution and is analyzed by atomic absorption spectrophotometry.

3.0 Definitions[Reserved]

4.0 Interferences

4.1 Matrix Effects. Analysis for Be by flame atomic absorption spectrophotometry is sensitive to the chemical composition and to the physical properties (*e.g.*, viscosity, pH) of the sample. Aluminum and silicon in particular are known to interfere when present in appreciable quantities. The analytical procedure includes (optionally) the use of the Method of Standard Additions to check for these matrix effects, and sample analysis using the Method of Standard Additions if significant matrix effects are found to be present (see Reference 2 in Section 16.0).

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

5.2 Corrosive reagents. The following reagents are hazardous. Personal protective equipment and safe procedures are useful in preventing chemical splashes. If contact occurs, immediately flush with copious amounts of water at least 15 minutes. Remove clothing under shower and decontaminate. Treat residual chemical burn as thermal burn.

5.2.1 Hydrochloric Acid (HC<sub>i</sub>). Highly toxic. Vapors are highly irritating to eyes, skin, nose, and lungs, causing severe damage. May cause bronchitis, pneumonia, or edema of lungs. Exposure to concentrations of 0.13 to 0.2 percent can be lethal to humans in a few minutes. Provide ventilation to limit exposure. Reacts with metals, producing hydrogen gas.

5.2.2 Hydrogen Peroxide (H<sub>2</sub>O<sub>2</sub>). Irritating to eyes, skin, nose, and lungs.

5.2.3 Nitric Acid (HNO<sub>3</sub>). Highly corrosive to eyes, skin, nose, and lungs. Vapors cause bronchitis, pneumonia, or edema of lungs. Reaction to inhalation may be delayed as long as 30 hours and still be fatal. Provide ventilation to limit exposure. Strong oxidizer. Hazardous reaction may occur with organic materials such as solvents.

5.2.4 Sodium Hydroxide (NaOH). Causes severe damage to eyes and skin. Inhalation causes irritation to nose, throat, and lungs. Reacts exothermically with limited amounts of water.

5.3 Beryllium is hazardous, and precautions should be taken to minimize exposure.

- 6.0 Equipment and Supplies
- 6.1 Sample Collection. Same as Method 5, Section 6.1, with the exception of the following:

6.1.1 Sampling Train. Same as Method 5, Section 6.1.1, with the exception of the following:

6.1.2 Probe Liner. Borosilicate or quartz glass tubing. A heating system capable of maintaining a gas temperature of 120 ±14 °C (248 ±25 °F) at the probe exit during sampling to prevent water condensation may be used.

Note: Do not use metal probe liners.

6.1.3 Filter Holder. Borosilicate glass, with a glass frit filter support and a silicone rubber gasket. Other materials of construction ( e.g., stainless steel, Teflon, Viton) may be used, subject to the approval of the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter. The holder shall be attached immediately at the outlet of the probe. A heating system capable of maintaining the filter at a minimum temperature in the range of the stack temperature may be used to prevent condensation from occurring.

6.1.4 Impingers. Four Greenburg-Smith impingers connected in series with leak-free ground glass fittings or any similar leak-free noncontaminating fittings. For the first, third, and fourth impingers, use impingers that are modified by replacing the tip with a 13 mm-ID (0.5 in.) glass tube extending to 13 mm (0.5 in.) from the bottom of the flask may be used.

6.2 Sample Recovery. The following items are needed for sample recovery:

- 6.2.1 Probe Cleaning Rod. At least as long as probe.
- 6.2.2 Glass Sample Bottles. Leakless, with Teflon-lined caps, 1000 ml.
- 6.2.3 Petri Dishes. For filter samples, glass or polyethylene, unless otherwise specified by the Administrator.
- 6.2.4 Graduated Cylinder. 250 ml.
- 6.2.5 Funnel and Rubber Policeman. To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.
- 6.2.6 Funnel. Glass, to aid in sample recovery.
- 6.2.7 Plastic Jar. Approximately 300 mL
- 6.3 Analysis. The following items are needed for sample analysis:
- 6.3.1 Atomic Absorption Spectrophotometer. Perkin-Elmer 303, or equivalent, with nitrous oxide/acetylene burner.
- 6.3.2 Hot Plate.
- 6.3.3 Perchloric Acid Fume Hood.
- 7.0 Reagents and Standards

Note: Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise, use the best available grade.

7.1 Sample Collection. Same as Method 5, Section 7.1, including deionized distilled water conforming to ASTM D 1193–77 or 91 (incorporated by reference—see §61.18), Type 3. The Millipore AA filter is recommended.

7.2 Sample Recovery. Same as Method 5 in appendix A, part 60, Section 7.2, with the addition of the following:

7.2.1 Wash Acid, 50 Percent (V/V) Hydrochloric Acid (HCI). Mix equal volumes of concentrated HCI and water, being careful to add the acid slowly to the water.

7.3 Sample Preparation and Analysis. The following reagents and standards and standards are needed for sample preparation and

Page 75 of 183 (40CFR61 Subpart FF)

analysis:

7.3.1 Water. Same as in Section 7.1.

7.3.2. Perchloric Acid (HCIO<sub>4</sub>). Concentrated (70 percent V/V).

7.3.3 Nitric Acid (HNO<sub>3</sub>). Concentrated.

7.3.4 Beryllium Powder. Minimum purity 98 percent.

7.3.5 Sulfuric Acid (H<sub>2</sub>SO<sub>4</sub>) Solution, 12 N. Dilute 33 ml of concentrated H<sub>2</sub>SO<sub>4</sub>to 1 liter with water.

7.3.6 Hydrochloric Acid Solution, 25 Percent HCI (V/V).

7.3.7 Stock Beryllium Standard Solution, 10  $\mu$ g Be/ml. Dissolve 10.0 mg of Be in 80 ml of 12 N H<sub>2</sub>SO<sub>4</sub>in a 1000-ml volumetric flask. Dilute to volume with water. This solution is stable for at least one month. Equivalent strength Be stock solutions may be prepared from Be salts such as BeCl<sub>2</sub>and Be(NO<sub>3</sub>)<sub>2</sub>(98 percent minimum purity).

7.3.8 Working Beryllium Standard Solution, 1 µg Be/ml. Dilute a 10 ml aliquot of the stock beryllium standard solution to 100 ml with 25 percent HCI solution to give a concentration of 1 mg/ml. Prepare this dilute stock solution fresh daily.

8.0 Sample Collection, Preservation, Transport, and Storage

The amount of Be that is collected is generally small, therefore, it is necessary to exercise particular care to prevent contamination or loss of sample.

8.1 Pretest Preparation. Same as Method 5, Section 8.1, except omit Section 8.1.3.

8.2 Preliminary Determinations. Same as Method 5, Section 8.2, with the exception of the following:

8.2.1 Select a nozzle size based on the range of velocity heads to assure that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates below 28 liters/min (1.0 cfm).

8.2.2 Obtain samples over a period or periods of time that accurately determine the maximum emissions that occur in a 24-hour period. In the case of cyclic operations, perform sufficient sample runs for the accurate determination of the emissions that occur over the duration of the cycle. A minimum sample time of 2 hours per run is recommended.

8.3 Preparation of Sampling Train. Same as Method 5, Section 8.3, with the exception of the following:

8.3.1 Prior to assembly, clean all glassware (probe, impingers, and connectors) by first soaking in wash acid for 2 hours, followed by rinsing with water.

8.3.2 Save a portion of the water for a blank analysis.

8.3.3 Procedures relating to the use of metal probe liners are not applicable.

8.3.4 Probe and filter heating systems are needed only if water condensation is a problem. If this is the case, adjust the heaters to provide a temperature at or above the stack temperature. However, membrane filters such as the Millipore AA are limited to about 107 °C (225 °F). If the stack gas is in excess of about 93 °C (200 °F), consideration should be given to an alternate procedure such as moving the filter holder downstream of the first impinger to insure that the filter does not exceed its temperature limit. After the sampling train has been assembled, turn on and set the probe heating system, if applicable, at the desired operating temperature. Allow time for the temperatures to stabilize. Place crushed ice around the impingers.

Note: An empty impinger may be inserted between the third impinger and the silica gel to remove excess moisture from the sample stream.

8.4 Leak Check Procedures, Sampling Train Operation, and Calculation of Percent Isokinetic. Same as Method 5, Sections 8.4, 8.5,

and 8.6, respectively.

8.5 Sample Recovery. Same as Method 5, Section 8.7, except treat the sample as follows: Transfer the probe and impinger assembly to a cleanup area that is clean, protected from the wind, and free of Be contamination. Inspect the train before and during this assembly, and note any abnormal conditions. Treat the sample as follows: Disconnect the probe from the impinger train.

8.5.1 Container No. 1. Same as Method 5, Section 8.7.6.1.

8.5.2 Container No. 2. Place the contents (measured to 1 ml) of the first three impingers into a glass sample bottle. Use the procedures outlined in Section 8.7.6.2 of Method 5, where applicable, to rinse the probe nozzle, probe fitting, probe liner, filter holder, and all glassware between the filter holder and the back half of the third impinger with water. Repeat this procedure with acetone. Place both water and acetone rinse solutions in the sample bottle with the contents of the impingers.

8.5.3 Container No. 3. Same as Method 5, Section 8.7.6.3.

8.6 Blanks.

8.6.1 Water Blank. Save a portion of the water as a blank. Take 200 ml directly from the wash bottle being used and place it in a plastic sample container labeled "H<sub>2</sub>O blank."

8.6.2 Filter. Save two filters from each lot of filters used in sampling. Place these filters in a container labeled "filter blank."

8.7 Post-test Glassware Rinsing. If an additional test is desired, the glassware can be carefully double rinsed with water and reassembled. However, if the glassware is out of use more than 2 days, repeat the initial acid wash procedure.

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
8.4, 10.1	Sampling equipment leak checks and calibration	Ensure accuracy and precision of sampling measurements.
10.2	Spectrophotometer calibration	Ensure linearity of spectrophotometer response to standards.
11.5	Check for matrix effects	Eliminate matrix effects.
11.6	Audit sample analysis	Evaluate analyst's technique and standards preparation.

9.2 Volume Metering System Checks. Same as Method 5, Section 9.2.

10.0 Calibration and Standardization

Note: Maintain a laboratory log of all calibrations.

10.1 Sampling Equipment. Same as Method 5, Section 10.0.

10.2 Preparation of Standard Solutions. Pipet 1, 3, 5, 8, and 10 ml of the 1.0  $\mu$ g Be/ml working standard solution into separate 100 ml volumetric flasks, and dilute to the mark with water. The total amounts of Be in these standards are 1, 3, 5, 8, and 10  $\mu$ g, respectively.

10.3 Spectrophotometer and Recorder. The Be response may be measured by either peak height or peak area. Analyze an aliquot

of the 10-µg standard at 234.8 nm using a nitrous oxide/acetylene flame. Determine the maximum absorbance of the standard, and set this value to read 90 percent of the recorder full scale.

## 10.4 Calibration Curve.

10.4.1 After setting the recorder scale, analyze an appropriately sized aliquot of each standard and the BLANK (see Section 11) until two consecutive peaks agree within 3 percent of their average value.

10.4.3 Subtract the average peak height (or peak area) of the blank—which must be less than 2 percent of recorder full scale—from the averaged peak heights of the standards. If the blank absorbance is greater than 2 percent of full-scale, the probable cause is Be contamination of a reagent or carry-over of Be from a previous sample. Prepare the calibration curve by plotting the corrected peak height of each standard solution versus the corresponding total Be weight in the standard (in µg).

10.5 Spectrophotometer Calibration Quality Control. Calculate the least squares slope of the calibration curve. The line must pass through the origin or through a point no further from the origin than  $\pm 2$  percent of the recorder full scale. Multiply the corrected peak height by the reciprocal of the least squares slope to determine the distance each calibration point lies from the theoretical calibration line. The difference between the calculated concentration values and the actual concentrations (i.e., 1, 3, 5, 8, and 10 µg Be) must be less than 7 percent for all standards.

# 11.0 Analytical Procedure

11.1 Sample Loss Check. Prior to analysis, check the liquid level in Container No. 2. Note on the analytical data sheet whether leakage occurred during transport. If a noticeable amount of leakage occurred, either void the sample or take steps, subject to the approval of the Administrator, to adjust the final results.

11.2 Glassware Cleaning. Before use, clean all glassware according to the procedure of Section 8.3.1.

11.3 Sample Preparation. The digestion of Be samples is accomplished in part in concentrated HCIO<sub>4</sub>.

Note: The sample must be heated to light brown fumes after the initial HNO3 addition; otherwise, dangerous perchlorates may result from the subsequent HClO<sub>4</sub>digestion. HClO<sub>4</sub>should be used only under a hood.

11.3.1 Container No. 1. Transfer the filter and any loose particulate matter from Container No. 1 to a 150-ml beaker. Add 35 ml concentrated  $HNO_3$ . To oxidize all organic matter, heat on a hotplate until light brown fumes are evident. Cool to room temperature, and add 5 ml 12 N  $H_2SO_4$ and 5 ml concentrated  $HCIO_4$ .

11.3.2 Container No. 2. Place a portion of the water and acetone sample into a 150 ml beaker, and put on a hotplate. Add portions of the remainder as evaporation proceeds and evaporate to dryness. Cool the residue, and add 35 ml concentrated  $HNO_3$ . To oxidize all organic matter, heat on a hotplate until light brown fumes are evident. Cool to room temperature, and add 5 ml 12 N H<sub>2</sub>SO<sub>4</sub>and 5 ml concentrated  $HCIO_4$ . Then proceed with step 11.3.4.

11.3.3 Final Sample Preparation. Add the sample from Section 11.3.2 to the 150-ml beaker from Section 11.3.1. Replace on a hotplate, and evaporate to dryness in a HClO₄hood. Cool the residue to room temperature, add 10.0 ml of 25 percent V/V HCl, and mix to dissolve the residue.

11.3.4 Filter and Water Blanks. Cut each filter into strips, and treat each filter individually as directed in Section 11.3.1. Treat the 200-ml water blank as directed in Section 11.3.2. Combine and treat these blanks as directed in Section 11.3.3.

11.4 Spectrophotometer Preparation. Turn on the power; set the wavelength, slit width, and lamp current; and adjust the background corrector as instructed by the manufacturer's manual for the particular atomic absorption spectrophotometer. Adjust the burner and flame characteristics as necessary.

11.5 Analysis. Calibrate the analytical equipment and develop a calibration curve as outlined in Sections 10.4 and 10.5.

11.5.1 Beryllium Samples. Repeat the procedure used to establish the calibration curve with an appropriately sized aliquot of each sample (from Section 11.3.3) until two consecutive peak heights agree within 3 percent of their average value. The peak height of each sample must be greater than 10 percent of the recorder full scale. If the peak height of the sample is off scale on the recorder, further dilute the original source sample to bring the Be concentration into the calibration range of the spectrophotometer.

11.5.2 Run a blank and standard at least after every five samples to check the spectrophotometer calibration. The peak height of the blank must pass through a point no further from the origin than  $\pm 2$  percent of the recorder full scale. The difference between the measured concentration of the standard (the product of the corrected peak height and the reciprocal of the least squares slope) and the actual concentration of the standard must be less than 7 percent, or recalibration of the analyzer is required.

11.5.3 Check for Matrix Effects (optional). Use the Method of Standard Additions (see Reference 2 in Section 16.0) to check at least one sample from each source for matrix effects on the Be results. If the results of the Method of Standard Additions procedure used on the single source sample do not agree to within 5 percent of the value obtained by the routine atomic absorption analysis, then reanalyze all samples from the source using the Method of Standard Additions procedure.

11.6 Container No. 2 (Silica Gel). Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. (This step may be conducted in the field.)

12.0 Data Analysis and Calculations

Carry out calculations, retaining at least one extra decimal significant figure beyond that of the acquired data. Round off figures only after the final calculation. Other forms of the equations may be used as long as they give equivalent results.

12.1 Nomenclature.

 $K_1$ = 0.3858 °K/mm Hg for metric units.

= 17.64 °R/in. Hg for English units.

 $K_3 = 10^{-6} g/\mu g$  for metric units.

=  $2.2046 \times 10^{-9}$ lb/µg for English units.

m<sub>Be</sub>= Total weight of beryllium in the source sample.

P<sub>s</sub>= Absolute stack gas pressure, mm Hg (in. Hg).

t = Daily operating time, sec/day.

T<sub>s</sub>= Absolute average stack gas temperature, °K (°R).

Vm(std)= Dry gas sample volume at standard conditions, scm (scf).

Vw(std)= Volume of water vapor at standard conditions, scm (scf).

12.2 Average Dry Gas Meter Temperature and Average Orifice Pressure Drop, Dry Gas Volume, Volume of Water Vapor Condensed, Moisture Content, Isokinetic Variation, and Stack Gas Velocity and Volumetric Flow Rate. Same as Method 5, Sections 12.2 through 12.5, 12.11, and 12.12, respectively.

12.3 Total Beryllium. For each source sample, correct the average maximum absorbance of the two consecutive samples whose peak heights agree within 3 percent of their average for the contribution of the solution blank (see Sections 11.3.4 and 11.5.2). Correcting for any dilutions if necessary, use the calibration curve and these corrected averages to determine the total weight of Be in each source sample.

12.4 Beryllium Emission Rate. Calculate the daily Hg emission rate, R, using Equation 104–1. For continuous operations, the operating time is equal to 86,400 seconds per day. For cyclic operations, use only the time per day each stack is in operation. The total Hg emission rate from a source will be the summation of results from all stacks.

$$R = \frac{K_1 K_3 t m_{Be} P_s v_s A_s}{T_s \left( V_{m(stil)} + V_{\omega(stil)} \right)} \qquad \text{Eq. 104-1}$$

12.5 Determination of Compliance. Each performance test consists of three sample runs. For the purpose of determining compliance with an applicable national emission standard, use the average of the results of all sample runs.

13.0 Method Performance.[Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management.[Reserved]

16.0 References

Same as References 1, 2, and 4-11 of Section 16.0 of Method 101 with the addition of the following:

1. Amos, M.D., and J.B. Willis. Use of High-Temperature Pre-Mixed Flames in Atomic Absorption Spectroscopy. Spectrochim. Acta. 22:1325. 1966.

2. Fleet, B., K.V. Liberty, and T. S. West. A Study of Some Matrix Effects in the Determination of Beryllium by Atomic Absorption Spectroscopy in the Nitrous Oxide-Acetylene Flame. Talanta 17:203. 1970.

17.0 Tables, Diagrams, Flowcharts, And Validation Data[Reserved]

Method 105-Determination of Mercury in Wastewater Treatment Plant Sewage Sludges

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should also have a thorough knowledge of at least the following additional test methods: Method 101 and Method 101A.

1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.	Sensitivity
Mercury (Hg)	7439–97–6	Dependent upon spectrophotometer and recorder.

1.2 Applicability. This method is applicable for the determination of total organic and inorganic Hg content in sewage sludges.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 Time-composite sludge samples are withdrawn from the conveyor belt subsequent to dewatering and before incineration or drying. A weighed portion of the sludge is digested in aqua regia and is oxidized by potassium permanganate (KMnO<sub>4</sub>). Mercury in the digested sample is then measured by the conventional spectrophotometric cold-vapor technique.

3.0 Definitions[Reserved]

4.0 Interferences[Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of

the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

5.2 Corrosive Reagents. The following reagents are hazardous. Personal protective equipment and safe procedures are useful in preventing chemical splashes. If contact occurs, immediately flush with copious amounts of water at least 15 minutes. Remove clothing under shower and decontaminate. Treat residual chemical burn as thermal burn.

5.2.1 Hydrochloric Acid (HCl). Highly toxic. Vapors are highly irritating to eyes, skin, nose, and lungs, causing severe damage. May cause bronchitis, pneumonia, or edema of lungs. Exposure to concentrations of 0.13 to 0.2 percent can be lethal to humans in a few minutes. Provide ventilation to limit exposure. Reacts with metals, producing hydrogen gas.

5.2.2 Nitric Acid (HNO<sub>3</sub>). Highly corrosive to eyes, skin, nose, and lungs. Vapors cause bronchitis, pneumonia, or edema of lungs. Reaction to inhalation may be delayed as long as 30 hours and still be fatal. Provide ventilation to limit exposure. Strong oxidizer. Hazardous reaction may occur with organic materials such as solvents.

6.0 Equipment and Supplies

6.1 Sample Collection and Mixing. The following items are required for collection and mixing of the sludge samples:

- 6.1.1 Container. Plastic, 50-liter.
- 6.1.2 Scoop. To remove 950-ml (1 quart.) sludge sample.
- 6.1.3 Mixer. Mortar mixer, wheelbarrow-type, 57-liter (or equivalent) with electricity-driven motor.
- 6.1.4 Blender. Waring-type, 2-liter.
- 6.1.5 Scoop. To remove 100-ml and 20-ml samples of blended sludge.
- 6.1.6 Erlenmeyer Flasks. Four, 125-ml.
- 6.1.7 Beakers. Glass beakers in the following sizes: 50 ml (1), 200 ml (1), 400 ml (2).
- 6.2 Sample Preparation and Analysis. Same as Method 101, Section 6.3, with the addition of the following:
- 6.2.1 Hot Plate.
- 6.2.2 Desiccator.
- 6.2.3 Filter Paper. S and S No. 588 (or equivalent).
- 6.2.4 Beakers. Glass beakers, 200 ml and 400 ml (2 each).
- 7.0 Reagents and Standards

Note: Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise, use the best available grade.

7.1 Sample Analysis. Same as Method 101A, Section 7.2, with the following additions and exceptions:

7.1.1 Hydrochloric Acid. The concentrated HCI specified in Method 101A, Section 7.2.4, is not required.

7.1.2 Aqua Regia. Prepare immediately before use. Carefully add one volume of concentrated HNO<sub>3</sub>to three volumes of concentrated HCI.

## 8.0 Sample Collection, Preservation, Storage, and Transport

8.1 Sludge Sampling. Withdraw equal volume increments of sludge [for a total of at least 15 liters (16 quarts)] at intervals of 30 min over an 8-hr period, and combine in a rigid plastic container.

8.2 Sludge Mixing. Transfer the entire 15-liter sample to a mortar mixer. Mix the sample for a minimum of 30 min at 30 rpm. Take six 100-ml portions of sludge, and combine in a 2-liter blender. Blend sludge for 5 min; add water as necessary to give a fluid consistency. Immediately after stopping the blender, withdraw four 20-ml portions of blended sludge, and place them in separate, tared 125-ml Erlenmeyer flasks. Reweigh each flask to determine the exact amount of sludge added.

8.3 Sample Holding Time. Samples shall be analyzed within the time specified in the applicable subpart of the regulations.

9.0 Quality Control

Section	Quality control measure	Effect
10.0	Spectrophotometer calibration	Ensure linearity of spectrophotometer response to standards.
11.0	Check for matrix effects	Eliminate matrix effects.

10.0 Calibration and Standardization

Same as Method 101A, Section 10.2.

### 11.0 Analytical Procedures

11.1 Solids Content of Blended Sludge. Dry one of the 20-ml blended samples from Section 8.2 in an oven at 105 °C (221 °F) to constant weight. Cool in a desiccator, weigh and record the dry weight of the sample.

11.2 Aqua Regia Digestion of Blended Samples.

11.2.1 To each of the three remaining 20-ml samples from Section 8.2 add 25 ml of aqua regia, and digest the on a hot plate at low heat (do not boil) for 30 min, or until samples are a pale yellow-brown color and are void of the dark brown color characteristic of organic matter. Remove from hotplate and allow to cool.

11.2.2 Filter each digested sample separately through an S and S No. 588 filter or equivalent, and rinse the filter contents with 50 ml of water. Transfer the filtrate and filter washing to a 100-ml volumetric flask, and carefully dilute to volume with water.

11.3 Solids Content of the Sludge Before Blending. Remove two 100-ml portions of mixed sludge from the mortar mixer and place in separate, tared 400-ml beakers. Reweigh each beaker to determine the exact amount of sludge added. Dry in oven at 105 °C (221 °F) and cool in a desiccator to constant weight.

11.4 Analysis for Mercury. Analyze the three aqua regia-digested samples using the procedures outlined in Method 101A, Section 11.0.

12.0 Data Analysis and Calculations

12.1 Nomenclature.

 $C_{\text{m}}\text{=}$  Concentration of Hg in the digested sample,  $\mu\text{g/g}.$ 

F<sub>sb</sub>= Weight fraction of solids in the blended sludge.

F<sub>sm</sub>= Weight fraction of solids in the collected sludge after mixing.

M = Hg content of the sewage sludge (on a dry basis),  $\mu g/g$ .

- m = Mass of Hg in the aliquot of digested sample analyzed, µg.
- n = number of digested samples (specified in Section 11.2 as three).
- V<sub>a</sub>= Volume of digested sample analyzed, ml.
- V<sub>s</sub>= Volume of digested sample, ml.
- Wb= Weight of empty sample beaker, g
- W<sub>bs</sub>= Weight of sample beaker and sample, g.
- Wbd= Weight of sample beaker and sample after drying, g.
- W<sub>f</sub>= Weight of empty sample flask, g.

W<sub>fd</sub>= Weight of sample flask and sample after drying, g.

W<sub>fs</sub>= Weight of sample flask and sample, g.

12.2 Mercury Content of Digested Sample (Wet Basis).

12.2.1 For each sample analyzed for Hg content, calculate the arithmetic mean maximum absorbance of the two consecutive samples whose peak heights agree ±3 percent of their average. Correct this average value for the contribution of the blank. Use the calibration curve and these corrected averages to determine the final Hg concentration in the solution cell for each sludge sample.

12.2.2 Calculate the average Hg concentration of the digested samples by correcting for any dilutions made to bring the sample into the working range of the spectrophotometer and for the weight of the sludge portion digested, using Equation 105–1.

$$\bar{C}_{m} = \sum_{i=1}^{n} \left[ \frac{mV_{s}}{V_{a} \left( W_{f^{*}} - W_{f} \right)} \right]_{i} \qquad \text{Eq. 105-1}$$

12.3 Solids Content of Blended Sludge. Determine the solids content of the blended sludge using Equation 105-2.

$$F_{s\delta} = 1 - \frac{W_{fs} - W_{fd}}{W_{fs} - W_f}$$
 Eq. 105-2

12.4 Solids Content of Bulk Sample (before blending but, after mixing in mortar mixer). Determine the solids content of each 100 ml aliquot (Section 11.3), and average the results.

$$F_{sm} = 1 - \frac{W_{\delta s} - W_{\delta d}}{W_{\delta s} - W_{\delta}} \qquad \text{Eq. 105-3}$$

12.5 Mercury Content of Bulk Sample (Dry Basis). Average the results from the three samples from each 8-hr composite sample, and calculate the Hg concentration of the composite sample on a dry basis.

$$M = \frac{\overline{C}_m}{F_{sb}} \qquad \text{Eq. 105-4}$$

Page 83 of 183 (40CFR61 Subpart FF)

13.0 Method Performance

13.1 Range. The range of this method is 0.2 to 5 micrograms per gram; it may be extended by increasing or decreasing sample size.

14.0 Pollution Prevention.[Reserved]

15.0 Waste Management.[Reserved]

16.0 References

1. Bishop, J.N. Mercury in Sediments. Ontario Water Resources Commission. Toronto, Ontario, Canada. 1971.

2. Salma, M. Private Communication. EPA California/Nevada Basin Office. Alameda, California.

3. Hatch, W.R. and W.L. Ott. Determination of Sub-Microgram Quantities of Mercury by Atomic Absorption Spectrophotometry. Analytical Chemistry. 40:2085. 1968.

4. Bradenberger, H., and H. Bader. The Determination of Nanogram Levels of Mercury in Solution by a Flameless Atomic Absorption Technique. Atomic Absorption Newsletter. 6:101. 1967.

5. Analytical Quality Control Laboratory (AQCL). Mercury in Sediment (Cold Vapor Technique) (Provisional Method). U.S. Environmental Protection Agency. Cincinnati, Ohio. April 1972.

6. Kopp, J.F., M.C. Longbottom, and L.B. Lobring. "Cold Vapor" Method for Determining Mercury. Journal AWWA. 64(1):20–25. 1972.

7. Manual of Methods for Chemical Analysis of Water and Wastes. U.S. Environmental Protection Agency. Cincinnati, Ohio. Publication No. EPA-624/2-74-003. December 1974. pp. 118-138.

8. Mitchell, W.J., M.R. Midgett, J. Suggs, R.J. Velton, and D. Albrink. Sampling and Homogenizing Sewage for Analysis. Environmental Monitoring and Support Laboratory, Office of Research and Development, U.S. Environmental Protection Agency. Research Triangle Park, N.C. March 1979. p. 7.

17.0 Tables, Diagrams, Flowcharts, and Validation Data.[Reserved]

Method 106-Determination of Vinyl Chloride Emissions From Stationary Sources

1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.	Sensitivity
Vinyl Chloride (CH <sub>2</sub> :CHCl)	75–01–4	Dependent upon analytical equipment.

1.2 Applicability. This method is applicable for the determination of vinyl chloride emissions from ethylene dichloride, vinyl chloride, and polyvinyl chloride manufacturing processes. This method does not measure vinyl chloride contained in particulate matter.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 An integrated bag sample of stack gas containing vinyl chloride is subjected to GC analysis using a flame ionization detector (FID).

## 3.0 Definitions.[Reserved]

#### 4.0 Interferences

4.1 Resolution interferences of vinyl chloride may be encountered on some sources. Therefore, the chromatograph operator should select the column and operating parameters best suited to the particular analysis requirements. The selection made is subject to approval of the Administrator. Approval is automatic, provided that confirming data are produced through an adequate supplemental analytical technique, and that the data are available for review by the Administrator. An example of this would be analysis with a different column or GC/mass spectroscopy.

# 5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

5.2 Toxic Analyte. Care must be exercised to prevent exposure of sampling personnel to vinyl chloride, which is a carcinogen.

6.0 Equipment and Supplies

6.1 Sample Collection (see Figure 106–1). The sampling train consists of the following components:

6.1.1 Probe. Stainless steel, borosilicate glass, Teflon tubing (as stack temperature permits), or equivalent, equipped with a glass wool plug to remove particulate matter.

6.1.2 Sample Lines. Teflon, 6.4-mm outside diameter, of sufficient length to connect probe to bag. Use a new unused piece for each series of bag samples that constitutes an emission test, and discard upon completion of the test.

6.1.3 Quick Connects. Stainless steel, male (2) and female (2), with ball checks (one pair without), located as shown in Figure 106–1.

6.1.4 Tedlar Bags. 50- to 100-liter capacity, to contain sample. Aluminized Mylar bags may be used if the samples are analyzed within 24 hours of collection.

6.1.5 Bag Containers. Rigid leak-proof containers for sample bags, with covering to protect contents from sunlight.

6.1.6 Needle Valve. To adjust sample flow rates.

6.1.7 Pump. Leak-free, with minimum of 2-liter/min capacity.

6.1.8 Charcoal Tube. To prevent admission of vinyl chloride and other organics to the atmosphere in the vicinity of samplers.

6.1.9 Flowmeter. For observing sampling flow rate; capable of measuring a flow range from 0.10 to 1.00 liter/min.

6.1.10 Connecting Tubing. Teflon, 6.4-mm outside diameter, to assemble sampling train (Figure 106-1).

6.1.11 Tubing Fittings and Connectors. Teflon or stainless steel, to assemble sampling training.

6.2 Sample Recovery. Teflon tubing, 6.4-mm outside diameter, to connect bag to GC sample loop. Use a new unused piece for each series of bag samples that constitutes an emission test, and discard upon conclusion of analysis of those bags.

6.3 Analysis. The following equipment is required:

6.3.1 Gas Chromatograph. With FID potentiometric strip chart recorder and 1.0 to 5.0-ml heated sampling loop in automatic sample valve. The chromatographic system shall be capable of producing a response to 0.1-ppmv vinyl chloride that is at least as great as the average noise level. (Response is measured from the average value of the base line to the maximum of the wave form, while standard operating conditions are in use.)

6.3.2 Chromatographic Columns. Columns as listed below. Other columns may be used provided that the precision and accuracy of the analysis of vinyl chloride standards are not impaired and that information is available for review confirming that there is adequate resolution of vinyl chloride peak. (Adequate resolution is defined as an area overlap of not more than 10 percent of the vinyl chloride peak by an interferent peak. Calculation of area overlap is explained in Procedure 1 of appendix C to this part: "Determination of Adequate Chromatographic Peak Resolution.")

6.3.2.1 Column A. Stainless steel, 2.0 m by 3.2 mm, containing 80/100-mesh Chromasorb 102.

6.3.2.2 Column B. Stainless steel, 2.0 m by 3.2 mm, containing 20 percent GE SF–96 on 60/ip-mesh Chromasorb P AW; or stainless steel, 1.0 m by 3.2 mm containing 80/100-mesh Porapak T. Column B is required as a secondary column if acetaldehyde is present. If used, column B is placed after column A. The combined columns should be operated at 120 °C (250 °F).

6.3.3 Rate Meters (2). Rotameter, or equivalent, 100-ml/min capacity, with flow control valves.

6.3.4 Gas Regulators. For required gas cylinders.

6.3.5 Temperature Sensor. Accurate to ±1 °C (±2 °F), to measure temperature of heated sample loop at time of sample injection.

6.3.6 Barometer. Accurate to ±5 mm Hg, to measure atmospheric pressure around GC during sample analysis.

6.3.7 Pump. Leak-free, with minimum of 100-ml/min capacity.

6.3.8 Recorder. Strip chart type, optionally equipped with either disc or electronic integrator.

6.3.9 Planimeter. Optional, in place of disc or electronic integrator on recorder, to measure chromatograph peak areas.

6.4 Calibration and Standardization.

6.4.1 Tubing. Teflon, 6.4-mm outside diameter, separate pieces marked for each calibration concentration.

Note: The following items are required only if the optional standard gas preparation procedures (Section 10.1) are followed.

6.4.2 Tedlar Bags. Sixteen-inch-square size, with valve; separate bag marked for each calibration concentration.

6.4.3 Syringes. 0.5-ml and 50-µl, gas tight, individually calibrated to dispense gaseous vinyl chloride.

6.4.4 Dry Gas Meter with Temperature and Pressure Gauges. Singer Model DTM-115 with 802 index, or equivalent, to meter nitrogen in preparation of standard gas mixtures, calibrated at the flow rate used to prepare standards.

- 7.0 Reagents and Standards
- 7.1 Analysis. The following reagents are required for analysis.

7.1.1 Helium or Nitrogen. Purity 99.9995 percent or greater, for chromatographic carrier gas.

7.1.2 Hydrogen. Purity 99.9995 percent or greater.

7.1.3 Oxygen or Air. Either oxygen (purity 99.99 percent or greater) or air (less than 0.1 ppmv total hydrocarbon content), as required by detector.

7.2 Calibration. Use one of the following options: either Sections 7.2.1 and 7.2.2, or Section 7.2.3.

7.2.1 Vinyl Chloride. Pure vinyl chloride gas certified by the manufacturer to contain a minimum of 99.9 percent vinyl chloride. If the gas manufacturer maintains a bulk cylinder supply of 99.9+ percent vinyl chloride, the certification analysis may have been performed on this supply, rather than on each gas cylinder prepared from this bulk supply. The date of gas cylinder preparation and the certified analysis must have been affixed to the cylinder before shipment from the gas manufacturer to the buyer.

## 7.2.2 Nitrogen. Same as described in Section 7.1.1.

7.2.3 Cylinder Standards. Gas mixture standards (50-,10-, and 5 ppmv vinyl chloride) in nitrogen cylinders may be used to directly prepare a chromatograph calibration curve as described in Section 10.3 if the following conditions are met: (a) The manufacturer certifies the gas composition with an accuracy of ±3 percent or better. (b) The manufacturer recommends a maximum shelf life over which the gas concentration does not change by greater than ±5 percent from the certified value. (c) The manufacturer affixes the date of gas cylinder preparation, certified vinyl chloride concentration, and recommended maximum shelf to the cylinder before shipment to the buyer.

7.2.3.1 Cylinder Standards Certification. The manufacturer shall certify the concentration of vinyl chloride in nitrogen in each cylinder by (a) directly analyzing each cylinder and (b) calibrating his analytical procedure on the day of cylinder analysis. To calibrate his analytical procedure, the manufacturer shall use as a minimum, a three point calibration curve. It is recommended that the manufacturer maintain (1) a high concentration calibration standard (between 50 and 100 ppmv) to prepare his calibration curve by an appropriate dilution technique and (2) a low-concentration calibration standard (between 5 and 10 ppmv) to verify the dilution technique used. If the difference between the apparent concentration read from the calibration curve and the true concentration assigned to the low-concentration standard exceeds 5 percent of the true concentration, the manufacturer shall determine the source of error and correct it, then repeat the three-point calibration.

7.2.3.2 Verification of Manufacturer's Calibration Standards. Before using a standard, the manufacturer shall verify each calibration standard (a) by comparing it to gas mixtures prepared (with 99 mole percent vinyl chloride) in accordance with the procedure described in Section 7.2.1 or (b) calibrating it against vinyl chloride cylinder Standard Reference Materials (SRM's) prepared by the National Institute of Standards and Technology, if such SRM's are available. The agreement between the initially determined concentration value and the verification concentration value must be ±5 percent. The manufacturer must reverify all calibration standards on a time interval consistent with the shelf life of the cylinder standards sold.

7.2.4 Audit Cylinder Standards.

7.2.4.1 Gas mixture standards with concentrations known only to the person supervising the analysis of samples. The concentrations of the audit cylinders should be: one low-concentration cylinder in the range of 5 to 20 ppmv vinyl chloride and one high-concentration cylinder in the range of 20 to 50 ppmv. When available, obtain audit samples from the appropriate EPA Regional Office or from the responsible enforcement authority.

Note: The responsible enforcement agency should be notified at least 30 days prior to the test date to allow sufficient time for sample delivery.

7.2.4.2 Alternatively, audit cylinders obtained from a commercial gas manufacturer may be used provided: (a) the gas meets the conditions described in Section 7.2.3, (b) the gas manufacturer certifies the audit cylinder as described in Section 7.2.3.1, and (c) the gas manufacturer obtains an independent analysis of the audit cylinders to verify this analysis. Independent analysis is defined here to mean analysis performed by an individual different than the individual who performs the gas manufacturer's analysis, while using calibration standards and analysis equipment different from those used for the gas manufacturer's analysis. Verification is complete and acceptable when the independent analysis concentration is within 5 percent of the gas manufacturer's concentration.

# 8.0 Sample Collection, Preservation, Storage, and Transport

Note: Performance of this method should not be attempted by persons unfamiliar with the operation of a gas chromatograph (GC) nor by those who are unfamiliar with source sampling, because knowledge beyond the scope of this presentation is required.

8.1 Bag Leak-Check. The following leak-check procedure is recommended, but not required, prior to sample collection. The post-test leak-check procedure is mandatory. Connect a water manometer and pressurize the bag to 5 to 10 cm  $H_2O$  (2 to 4 in.  $H_2O$ ). Allow to stand for 10 min. Any displacement in the water manometer indicates a leak. Also, check the rigid container for leaks in this manner.

Note: An alternative leak-check method is to pressurize the bag to 5 to 10 cm H2O and allow it to stand overnight. A deflated bag indicates a leak. For each sample bag in its rigid container, place a rotameter in line between the bag and the pump inlet. Evacuate the bag. Failure of the rotameter to register zero flow when the bag appears to be empty indicates a leak.

8.2 Sample Collection. Assemble the sample train as shown in Figure 106–1. Join the quick connects as illustrated, and determine that all connection between the bag and the probe are tight. Place the end of the probe at the centroid of the stack and start the pump with the needle valve adjusted to yield a flow that will fill over 50 percent of bag volume in the specific sample period. After

allowing sufficient time to purge the line several times, change the vacuum line from the container to the bag and evacuate the bag until the rotameter indicates no flow. Then reposition the sample and vacuum lines and begin the actual sampling, keeping the rate proportional to the stack velocity. At all times, direct the gas exiting the rotameter away from sampling personnel. At the end of the sample period, shut off the pump, disconnect the sample line from the bag, and disconnect the vacuum line from the bag container. Protect the bag container from sunlight.

8.3 Sample Storage. Keep the sample bags out of direct sunlight. When at all possible, analysis is to be performed within 24 hours, but in no case in excess of 72 hours of sample collection. Aluminized Mylar bag samples must be analyzed within 24 hours.

8.4 Post-test Bag Leak-Check. Subsequent to recovery and analysis of the sample, leak-check the sample bag according to the procedure outlined in Section 8.1.

9.0 Quality Control

9.1 Miscellaneous Quality Control

Section	Quality control measure	Effect
10.3	Chromatograph calibration	Ensure precision and accuracy of chromatograph.
11.1	Audit sample analysis	Evaluate analytical technique and standards preparation.

9.2 Immediately after the preparation of the calibration curve and prior to the sample analyses, perform the analysis audit described in appendix C, Procedure 2: "Procedure for Field Auditing GC Analysis."

10.0 Calibration and Standardization

Note: Maintain a laboratory log of all calibrations.

10.1 Preparation of Vinyl Chloride Standard Gas Mixtures. (Optional Procedure-delete if cylinder standards are used.) Evacuate a 16-inch square Tedlar bag that has passed a leak-check (described in Section 8.1) and meter in 5.0 liters of nitrogen. While the bag is filling, use the 0.5-ml syringe to inject 250 µl of 99.9+ percent vinyl chloride gas through the wall of the bag. Upon withdrawing the syringe, immediately cover the resulting hole with a piece of adhesive tape. The bag now contains a vinyl chloride concentration of 50 ppmv. In a like manner use the 50 µl syringe to prepare gas mixtures having 10-and 5-ppmv vinyl chloride concentrations. Place each bag on a smooth surface and alternately depress opposite sides of the bag 50 times to further mix the gases. These gas mixture standards may be used for 10 days from the date of preparation, after which time new gas mixtures must be prepared. (Caution: Contamination may be a problem when a bag is reused if the new gas mixture standard is a lower concentration than the previous gas mixture standard.)

10.2 Determination of Vinyl Chloride Retention Time. (This section can be performed simultaneously with Section 10.3.) Establish chromatograph conditions identical with those in Section 11.3. Determine proper attenuator position. Flush the sampling loop with helium or nitrogen and activate the sample valve. Record the injection time, sample loop temperature, column temperature, carrier gas flow rate, chart speed, and attenuator setting. Record peaks and detector responses that occur in the absence of vinyl chloride. Maintain conditions with the equipment plumbing arranged identically to Section 11.2, and flush the sample loop for 30 seconds at the rate of 100 ml/min with one of the vinyl chloride calibration mixtures. Then activate the sample valve. Record the injection time. Select the peak that corresponds to vinyl chloride. Measure the distance on the chart from the injection time to the time at which the peak maximum occurs. This quantity divided by the chart speed is defined as the retention time. Since other organics may be present in the sample, positive identification of the vinyl chloride peak must be made.

10.3 Preparation of Chromatograph Calibration Curve. Make a GC measurement of each gas mixture standard (described in Section 7.2.3 or 10.1) using conditions identical to those listed in Sections 11.2 and 11.3. Flush the sampling loop for 30 seconds at the rate of 100 ml/min with one of the standard mixtures, and activate the sample valve. Record the concentration of vinyl chloride injected ( $C_c$ ), attenuator setting, chart speed, peak area, sample loop temperature, column temperature, carrier gas flow rate, and retention time. Record the barometric pressure. Calculate  $A_c$ , the peak area multiplied by the attenuator setting. Repeat until two consecutive injection areas are within 5 percent, then plot the average of those two values versus  $C_c$ . When the other standard gas mixtures have been similarly analyzed and plotted, draw a straight line through the points derived by the least squares method. Perform calibration daily, or before and after the analysis of each emission test set of bag samples, whichever is more frequent. For each group of sample analyses, use the average of the two calibration curves which bracket that group to determine the respective sample concentrations. If the two calibration curves differ by more than 5 percent from their mean value, then report the final results by both calibration curves.

## 11.0 Analytical Procedure

11.1 Audit Sample Analysis. Immediately after the preparation of the calibration curve and prior to the sample analyses, perform the analysis audit described in Procedure 2 of appendix C to this part: "Procedure for Field Auditing GC Analysis."

11.2 Sample Recovery. With a new piece of Teflon tubing identified for that bag, connect a bag inlet valve to the gas chromatograph sample valve. Switch the valve to receive gas from the bag through the sample loop. Arrange the equipment so the sample gas passes from the sample valve to 100-ml/min rotameter with flow control valve followed by a charcoal tube and a 1-in. H<sub>2</sub>O pressure gauge. Maintain the sample flow either by a vacuum pump or container pressurization if the collection bag remains in the rigid container. After sample loop purging is ceased, allow the pressure gauge to return to zero before activating the gas sampling valve.

## 11.3 Analysis.

11.3.1 Set the column temperature to 100 °C (210 °F) and the detector temperature to 150 °C (300 °F). When optimum hydrogen and oxygen (or air) flow rates have been determined, verify and maintain these flow rates during all chromatography operations. Using helium or nitrogen as the carrier gas, establish a flow rate in the range consistent with the manufacturer's requirements for satisfactory detector operation. A flow rate of approximately 40 ml/min should produce adequate separations. Observe the base line periodically and determine that the noise level has stabilized and that base line drift has ceased. Purge the sample loop for 30 seconds at the rate of 100 ml/min, shut off flow, allow the sample loop pressure to reach atmospheric pressure as indicated by the H<sub>2</sub>O manometer, then activate the sample valve. Record the injection time (the position of the pen on the chart at the time of sample injection), sample number, sample loop temperature, column temperature, carrier gas flow rate, chart speed, and attenuator setting. Record the barometric pressure. From the chart, note the peak having the retention time corresponding to vinyl chloride as determined in Section 10.2. Measure the vinyl chloride peak area, A<sub>m</sub>, by use of a disc integrator, electronic integrator, or a planimeter. Measure and record the peak heights, H<sub>m</sub>. Record A<sub>m</sub>and retention time. Repeat the injection at least two times or until two consecutive values for the total area of the vinyl chloride peak agree within 5 percent of their average. Use the average value for these two total areas to compute the bag concentration.

11.3.2 Compare the ratio of  $H_m$  to  $A_m$  for the vinyl chloride sample with the same ratio for the standard peak that is closest in height. If these ratios differ by more than 10 percent, the vinyl chloride peak may not be pure (possibly acetaldehyde is present) and the secondary column should be employed (see Section 6.3.2.2).

11.4 Determination of Bag Water Vapor Content. Measure the ambient temperature and barometric pressure near the bag. From a water saturation vapor pressure table, determine and record the water vapor content of the bag,  $B_{wb}$ , as a decimal figure. (Assume the relative humidity to be 100 percent unless a lesser value is known.)

12.0 Calculations and Data Analysis

12.1 Nomenclature.

A<sub>m</sub>= Measured peak area.

A<sub>f</sub>= Attenuation factor.

B<sub>wb</sub>= Water vapor content of the bag sample, as analyzed, volume fraction.

- C<sub>b</sub>= Concentration of vinyl chloride in the bag, ppmv.
- C<sub>c</sub>= Concentration of vinyl chloride in the standard sample, ppmv.
- P<sub>i</sub>= Laboratory pressure at time of analysis, mm Hg.
- P<sub>r</sub>= Reference pressure, the laboratory pressure recorded during calibration, mm Hg.
- $T_i\text{=}$  Absolute sample loop temperature at the time of analysis, °K (°R).
- T<sub>r</sub>= Reference temperature, the sample loop temperature recorded during calibration, °K (°R).
- 12.2 Sample Peak Area. Determine the sample peak area, A<sub>c</sub>, as follows:
$$A_r = A_m A_f \qquad \text{Eq. 106-1}$$

12.3 Vinyl Chloride Concentration. From the calibration curves prepared in Section 10.3, determine the average concentration value of vinyl chloride,  $C_{c}$ , that corresponds to  $A_{c}$ , the sample peak area. Calculate the concentration of vinyl chloride in the bag,  $C_{b}$ , as follows:

$$C_{\phi} = \frac{C_{c}P_{r}T_{i}}{P_{i}T_{r}\left(1 - B_{\psi\phi}\right)} \qquad \text{Eq. 106-2}$$

#### 13.0 Method Performance

13.1 Analytical Range. This method is designed for the 0.1 to 50 parts per million by volume (ppmv) range. However, common gas chromatograph (GC) instruments are capable of detecting 0.02 ppmv vinyl chloride. With proper calibration, the upper limit may be extended as needed.

- 14.0 Pollution Prevention, [Reserved]
- 15.0 Waste Management, [Reserved]
- 16.0 References

1. Brown D.W., E.W. Loy, and M.H. Stephenson. Vinyl Chloride Monitoring Near the B. F. Goodrich Chemical Company in Louisville, KY. Region IV, U.S. Environmental Protection Agency, Surveillance and Analysis Division, Athens, GA. June 24, 1974.

2. G.D. Clayton and Associates. Evaluation of a Collection and Analytical Procedure for Vinyl Chloride in Air. U.S. Environmental Protection Agency, Research Triangle Park, N.C. EPA Contract No. 68–02–1408, Task Order No. 2, EPA Report No. 75–VCL–1. December 13, 1974.

3. Midwest Research Institute. Standardization of Stationary Source Emission Method for Vinyl Chloride. U.S. Environmental Protection Agency, Research Triangle Park, N.C. Publication No. EPA–600/4–77–026. May 1977.

4. Scheil, G. and M.C. Sharp. Collaborative Testing of EPA Method 106 (Vinyl Chloride) that Will Provide for a Standardized Stationary Source Emission Measurement Method. U.S. Environmental Protection Agency, Research Triangle Park, N.C. Publication No. EPA 600/4–78–058. October 1978.

17.0 Tables, Diagrams Flowcharts, and Validation Data.



#### View or download PDF

Method 107-Determination of Vinyl Chloride Content of In-Process Wastewater Samples, and Vinyl Chloride Content of Polyvinyl Chloride Resin Slurry, Wet Cake, and Latex Samples

Note: Performance of this method should not be attempted by persons unfamiliar with the operation of a gas chromatograph (GC) nor by those who are unfamiliar with source sampling, because knowledge beyond the scope of this presentation is required. This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 106.

#### 1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.	Sensitivity
Vinyl Chloride (CH <sub>2</sub> :CHCl)	75-01-4	Dependent upon analytical equipment.

1.2 Applicability. This method is applicable for the determination of the vinyl chloride monomer (VCM) content of in-process wastewater samples, and the residual vinyl chloride monomer (RCVM) content of polyvinyl chloride (PVC) resins, wet, cake, slurry, and latex samples. It cannot be used for polymer in fused forms, such as sheet or cubes. This method is not acceptable where methods from section 304(h) of the Clean Water Act, 33 U.S.C. 1251 *et seq.* (the Federal Water Pollution Control Amendments of 1972 as amended by the Clean Water Act of 1977) are required.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

#### 2.0 Summary of Method

2.1 The basis for this method relates to the vapor equilibrium that is established at a constant known temperature in a closed system between RVCM, PVC resin, water, and air. The RVCM in a PVC resin will equilibrate rapidly in a closed vessel, provided that the temperature of the PVC resin is maintained above the glass transition temperature of that specific resin.

2.2 A sample of PVC or in-process wastewater is collected in a vial or bottle and is conditioned. The headspace in the vial or bottle is then analyzed for vinyl chloride using gas chromatography with a flame ionization detector.

#### 3.0 Definitions[Reserved]

#### 4.0 Interferences

4.1 The chromatograph columns and the corresponding operating parameters herein described normally provide an adequate resolution of vinyl chloride; however, resolution interferences may be encountered on some sources. Therefore, the chromatograph operator shall select the column and operating parameters best suited to his particular analysis requirements, subject to the approval of the Administrator. Approval is automatic provided that confirming data are produced through an adequate supplemental analytical technique, such as analysis with a different column or GC/mass spectroscopy, and that these data are made available for review by the Administrator.

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

5.2 Toxic Analyte. Care must be exercised to prevent exposure of sampling personnel to vinyl chloride, which is a carcinogen. Do not release vinyl chloride to the laboratory atmosphere during preparation of standards. Venting or purging with VCM/air mixtures must be held to a minimum. When they are required, the vapor must be routed to outside air. Vinyl chloride, even at low ppm levels, must never be vented inside the laboratory. After vials have been analyzed, the gas must be vented prior to removal of the vial from the instrument turntable. Vials must be vented through a hypodermic needle connected to an activated charcoal tube to prevent release of vinyl chloride into the laboratory atmosphere. The charcoal must be replaced prior to vinyl chloride breakthrough.

#### 6.0 Equipment and Supplies

6.1 Sample Collection. The following equipment is required:

6.1.1 Glass bottles. 60-ml (2-oz) capacity, with wax-lined screw-on tops, for PVC samples.

6.1.2 Glass Vials. Headspace vials, with Teflon-faced butyl rubber sealing discs, for water samples.

- 6.1.3 Adhesive Tape. To prevent loosening of bottle tops.
- 6.2 Sample Recovery. The following equipment is required:
- 6.2.1 Glass Vials. Headspace vials, with butyl rubber septa and aluminum caps. Silicone rubber is not acceptable.
- 6.2.2 Analytical Balance. Capable of determining sample weight within an accuracy of ±1 percent
- 6.2.3 Vial Sealer. To seal headspace vials.
- 6.2.4 Syringe, 100-ml capacity.
- 6.3 Analysis. The following equipment is required:

6.3.1 Headspace Sampler and Chromatograph. Capable of sampling and analyzing a constant amount of headspace gas from a sealed vial, while maintaining that vial at a temperature of 90 °C ±0.5 °C (194 °F ±0.9 °F). The chromatograph shall be equipped with a flame ionization detector (FID). Perkin-Elmer Corporation Models F–40, F–42, F–45, HS–6, and HS–100, and Hewlett-Packard Corporation Model 19395A have been found satisfactory. Chromatograph backflush capability may be required.

6.3.2 Chromatographic Columns. Stainless steel 1 m by 3.2 mm and 2 m by 3.2 mm, both containing 50/80-mesh Porapak Q. Other columns may be used provided that the precision and accuracy of the analysis of vinyl chloride standards are not impaired and information confirming that there is adequate resolution of the vinyl chloride peak are available for review. (Adequate resolution is defined as an area overlap of not more than 10 percent of the vinyl chloride peak by an interferant peak. Calculation of area overlap is explained in Procedure 1 of appendix C to this part: "Determination of Adequate Chromatographic Peak Resolution.") Two 1.83 m columns, each containing 1 percent Carbowax 1500 on Carbopak B, have been found satisfactory for samples containing acetaldehyde.

- 6.3.3 Temperature Sensor. Range 0 to 100 °C (32 to 212 °F) accurate to 0.1 °C.
- 6.3.4 Integrator-Recorder. To record chromatograms.
- 6.3.5 Barometer. Accurate to 1 mm Hg.
- 6.3.6 Regulators. For required gas cylinders.
- 6.3.7 Headspace Vial Pre-Pressurizer. Nitrogen pressurized hypodermic needle inside protective shield.
- 7.0 Reagents and Standards
- 7.1 Analysis. Same as Method 106, Section 7.1, with the addition of the following:
- 7.1.1 Water. Interference-free.
- 7.2 Calibration. The following items are required for calibration:

7.2.1 Cylinder Standards (4). Gas mixture standards (50-, 500-, 2000- and 4000-ppm vinyl chloride in nitrogen cylinders). Cylinder standards may be used directly to prepare a chromatograph calibration curve as described in Section 10.3, if the following conditions are met: (a) The manufacturer certifies the gas composition with an accuracy of ±3 percent or better (see Section 7.2.1.1). (b) The manufacturer recommends a maximum shelf life over which the gas concentration does not change by greater than ±5 percent from the certified value. (c) The manufacturer affixes the date of gas cylinder preparation, certified vinyl chloride concentration, and recommended maximum shelf life to the cylinder before shipment to the buyer.

7.2.1.1 Cylinder Standards Certification. The manufacturer shall certify the concentration of vinyl chloride in nitrogen in each cylinder by (a) directly analyzing each cylinder and (b) calibrating the analytical procedure on the day of cylinder analysis. To calibrate the analytical procedure, the manufacturer shall use, as a minimum, a 3-point calibration curve. It is recommended that the manufacturer maintain (1) a high-concentration calibration standard (between 4000 and 8000 ppm) to prepare the calibration curve by an appropriate dilution technique and (2) a low-concentration calibration standard (between 50 and 500 ppm) to verify the dilution technique used. If the difference between the apparent concentration read from the calibration curve and the true concentration assigned to the low-concentration standard exceeds 5 percent of the true concentration, the manufacturer shall determine

the source of error and correct it, then repeat the 3-point calibration.

7.2.1.2 Verification of Manufacturer's Calibration Standards. Before using, the manufacturer shall verify each calibration standard by (a) comparing it to gas mixtures prepared (with 99 mole percent vinyl chloride) in accordance with the procedure described in Section 10.1 of Method 106 or by (b) calibrating it against vinyl chloride cylinder Standard Reference Materials (SRMs) prepared by the National Institute of Standards and Technology, if such SRMs are available. The agreement between the initially determined concentration value and the verification concentration value must be within 5 percent. The manufacturer must reverify all calibration standards on a time interval consistent with the shelf life of the cylinder standards sold.

8.0 Sample Collection, Preservation, Storage, and Transport

8.1 Sample Collection.

8.1.1 PVC Sampling. Allow the resin or slurry to flow from a tap on the tank or silo until the tap line has been well purged. Extend and fill a 60-ml sample bottle under the tap, and immediately tighten a cap on the bottle. Wrap adhesive tape around the cap and bottle to prevent the cap from loosening. Place an identifying label on each bottle, and record the date, time, and sample location both on the bottles and in a log book.

8.1.2 Water Sampling. At the sampling location fill the vials bubble-free to overflowing so that a convex meniscus forms at the top. The excess water is displaced as the sealing disc is carefully placed, with the Teflon side down, on the opening of the vial. Place the aluminum seal over the disc and the neck of the vial, and crimp into place. Affix an identifying label on the bottle, and record the date, time, and sample location both on the vials and in a log book.

8.2 Sample Storage. All samples must be analyzed within 24 hours of collection, and must be refrigerated during this period.

9.0 Quality Control

Section	Quality control measure	Effect
10.3	Chromatograph calibration	Ensure precision and accuracy of chromatograph.

#### 10.0 Calibration and Standardization

Note: Maintain a laboratory log of all calibrations.

10.1 Preparation of Standards. Calibration standards are prepared as follows: Place 100 µl or about two equal drops of distilled water in the sample vial, then fill the vial with the VCM/nitrogen standard, rapidly seat the septum, and seal with the aluminum cap. Use a1/8-in. stainless steel line from the cylinder to the vial. Do not use rubber or Tygon tubing. The sample line from the cylinder must be purged (into a properly vented hood) for several minutes prior to filling the vials. After purging, reduce the flow rate to between 500 and 1000 cc/min. Place end of tubing into vial (near bottom). Position a septum on top of the vial, pressing it against the1/8-in. filling tube to minimize the size of the vent opening. This is necessary to minimize mixing air with the standard in the vial. Each vial is to be purged with standard for 90 seconds, during which time the filling tube is gradually slid to the top of the vial. After the 90 seconds, the tube is removed with the septum, simultaneously sealing the vial. Practice will be necessary to develop good technique. Rubber gloves should be worn during the above operations. The sealed vial must then be pressurized for 60 seconds using the vial prepressurizer. Test the vial for leakage by placing a drop of water on the septum at the needle hole. Prepressurization of standards is not required unless samples have been prepressurized.

10.2 Analyzer Calibration. Calibration is to be performed each 8-hour period the chromatograph is used. Alternatively, calibration with duplicate 50-, 500-, 2,000-, and 4,000-ppm standards (hereafter described as a four-point calibration) may be performed on a monthly basis, provided that a calibration confirmation test consisting of duplicate analyses of an appropriate standard is performed once per plant shift, or once per chromatograph carrousel operation (if the chromatograph operation is less frequent than once per shift). The criterion for acceptance of each calibration confirmation test is that both analyses of 500-ppm standards [2,000-ppm standards if dispersion resin (excluding latex resin) samples are being analyzed] must be within 5 percent of the most recent four-point calibration curve. If this criterion is not met, then a complete four-point calibration must be performed before sample analyses can proceed.

10.3 Preparation of Chromatograph Calibration Curve. Prepare two vials each of 50-, 500-, 2,000-, and 4,000-ppm standards. Run the calibration samples in exactly the same manner as regular samples. Plot A<sub>s</sub>, the integrator area counts for each standard sample, versus C<sub>c</sub>, the concentration of vinyl chloride in each standard sample. Draw a straight line through the points derived by the least squares method.

#### 11.0 Analytical Procedure

11.1 Preparation of Equipment. Install the chromatographic column and condition overnight at 160 °C (320 °F). In the first operation, Porapak columns must be purged for 1 hour at 230 °C (450 °F).

Do not connect the exit end of the column to the detector while conditioning. Hydrogen and air to the detector must be turned off while the column is disconnected.

11.2 Flow Rate Adjustments. Adjust flow rates as follows:

11.2.1. Nitrogen Carrier Gas. Set regulator on cylinder to read 50 psig. Set regulator on chromatograph to produce a flow rate of 30.0 cc/min. Accurately measure the flow rate at the exit end of the column using the soap film flowmeter and a stopwatch, with the oven and column at the analysis temperature. After the instrument program advances to the "B" (backflush) mode, adjust the nitrogen pressure regulator to exactly balance the nitrogen flow rate at the detector as was obtained in the "A" mode.

11.2.2. Vial Prepressurizer Nitrogen.

11.2.2.1 After the nitrogen carrier is set, solve the following equation and adjust the pressure on the vial prepressurizer accordingly.

$$P = \frac{T_1}{T_2} \left[ P_1 - \frac{P_{w1} - P_{w2}}{7.50} \right] - 10kPa \qquad \text{Eq. 107-1}$$

Where:

T<sub>1</sub>= Ambient temperature, °K (°R).

T<sub>2</sub>= Conditioning bath temperature, °K (°R).

P1= Gas chromatograph absolute dosing pressure (analysis mode), k Pa.

Pw1= Water vapor pressure 525.8 mm Hg @ 90 °C.

Pw2= Water vapor pressure 19.8 mm Hg @ 22 °C.

7.50 = mm Hg per k Pa.

10 kPa = Factor to adjust the prepressurized pressure to slightly less than the dosing pressure.

11.2.2.2 Because of gauge errors, the apparatus may over-pressurize the vial. If the vial pressure is at or higher than the dosing pressure, an audible double injection will occur. If the vial pressure is too low, errors will occur on resin samples because of inadequate time for head-space gas equilibrium. This condition can be avoided by running several standard gas samples at various pressures around the calculated pressure, and then selecting the highest pressure that does not produce a double injection. All samples and standards must be pressurized for 60 seconds using the vial prepressurizer. The vial is then placed into the 90 °C conditioning bath and tested for leakage by placing a drop of water on the septum at the needle hole. A clean, burr-free needle is mandatory.

11.2.3. Burner Air Supply. Set regulator on cylinder to read 50 psig. Set regulator on chromatograph to supply air to burner at a rate between 250 and 300 cc/min. Check with bubble flowmeter.

11.2.4. Hydrogen Supply. Set regulator on cylinder to read 30 psig. Set regulator on chromatograph to supply approximately 35 ±5 cc/min. Optimize hydrogen flow to yield the most sensitive detector response without extinguishing the flame. Check flow with bubble meter and record this flow.

11.3 Temperature Adjustments. Set temperatures as follows:

11.3.1. Oven (chromatograph column), 140 °C (280 °F).

11.3.2. Dosing Line, 150 °C (300 °F).

11.3.3. Injection Block, 170 °C (340 °F).

11.3.4. Sample Chamber, Water Temperature, 90 °C ±1.0 °C (194 °F ±1.8 °F).

11.4 Ignition of Flame Ionization Detector. Ignite the detector according to the manufacturer's instructions.

11.5 Amplifier Balance. Balance the amplifier according to the manufacturer's instructions.

11.6 Programming the Chromatograph. Program the chromatograph as follows:

11.6.1. I-Dosing or Injection Time. The normal setting is 2 seconds.

11.6.2. A—Analysis Time. The normal setting is approximately 70 percent of the VCM retention time. When this timer terminates, the programmer initiates backflushing of the first column.

11.6.3. B-Backflushing Time. The normal setting is double the analysis time.

11.6.4. W-Stabilization Time. The normal setting is 0.5 min to 1.0 min.

11.6.5. X—Number of Analyses Per Sample. The normal setting is one.

11.7. Sample Treatment. All samples must be recovered and analyzed within 24 hours after collection.

11.7.1 Resin Samples. The weight of the resin used must be between 0.1 and 4.5 grams. An exact weight must be obtained (within ±1 percent) for each sample. In the case of suspension resins, a volumetric cup can be prepared for holding the required amount of sample. When the cup is used, open the sample bottle, and add the cup volume of resin to the tared sample vial (tared, including septum and aluminum cap). Obtain the exact sample weight, add 100 ml or about two equal drops of water, and immediately seal the vial. Report this value on the data sheet; it is required for calculation of RVCM. In the case of dispersion resins, the cup cannot be used. Weigh the sample in an aluminum dish, transfer the sample to the tared vial, and accurately weigh it in the vial. After prepressurization of the samples, condition them for a minimum of 1 hour in the 90 °C (190 °F) bath. Do not exceed 5 hours. Prepressurization equation yields an absolute prepressurization value that is within 30 percent of the atmospheric pressure.

Note: Some aluminum vial caps have a center section that must be removed prior to placing into sample tray. If the cap is not removed, the injection needle will be damaged.

11.7.2 Suspension Resin Slurry and Wet Cake Samples. Decant the water from a wet cake sample, and turn the sample bottle upside down onto a paper towel. Wait for the water to drain, place approximately 0.2 to 4.0 grams of the wet cake sample in a tared vial (tared, including septum and aluminum cap) and seal immediately. Then determine the sample weight (1 percent). All samples weighing over 0.2 gram, must be prepressurized prior to conditioning for 1 hour at 90 °C (190 °F), except as noted in Section 11.7.1. A sample of wet cake is used to determine total solids (TS). This is required for calculating the RVCM.

11.7.3 Dispersion Resin Slurry and Geon Latex Samples. The materials should not be filtered. Sample must be thoroughly mixed. Using a tared vial (tared, including septum and aluminum cap) add approximately eight drops (0.25 to 0.35 g) of slurry or latex using a medicine dropper. This should be done immediately after mixing. Seal the vial as soon as possible. Determine sample weight (1 percent). Condition the vial for 1 hour at 90 °C (190 °F) in the analyzer bath. Determine the TS on the slurry sample (Section 11.10).

11.7.4 In-process Wastewater Samples. Using a tared vial (tared, including septum and aluminum cap) quickly add approximately 1 cc of water using a medicine dropper. Seal the vial as soon as possible. Determine sample weight (1 percent). Condition the vial for 1 hour at 90 °C (190 °F) in the analyzer bath.

11.8 Preparation of Sample Turntable.

11.8.1 Before placing any sample into turntable, be certain that the center section of the aluminum cap has been removed. The numbered sample vials should be placed in the corresponding numbered positions in the turntable. Insert samples in the following order:

11.8.1.1 Positions 1 and 2. Old 2000-ppm standards for conditioning. These are necessary only after the analyzer has not been used for 24 hours or longer.

11.8.1.2 Position 3. 50-ppm standard, freshly prepared.

11.8.1.3 Position 4. 500-ppm standard, freshly prepared.

11.8.1.4 Position 5. 2000-ppm standard, freshly prepared.

11.8.1.5 Position 6. 4000-ppm standard, freshly prepared.

11.8.1.6 Position 7. Sample No. 7 (This is the first sample of the day, but is given as 7 to be consistent with the turntable and the integrator printout.)

11.8.2 After all samples have been positioned, insert the second set of 50-, 500-, 2000-, and 4000-ppm standards. Samples, including standards, must be conditioned in the bath of 90 °C (190 °F) for a minimum of one hour and a maximum of five hours.

11.9 Start Chromatograph Program. When all samples, including standards, have been conditioned at 90 °C (190 °F) for at least one hour, start the analysis program according to the manufacturer's instructions. These instructions must be carefully followed when starting and stopping a program to prevent damage to the dosing assembly.

11.10 Determination of Total Solids. For wet cake, slurry, resin solution, and PVC latex samples, determine TS for each sample by accurately weighing approximately 3 to 4 grams of sample in an aluminum pan before and after placing in a draft oven (105 to 110 °C (221 to 230 °F)). Samples must be dried to constant weight. After first weighing, return the pan to the oven for a short period of time, and then reweigh to verify complete dryness. The TS are then calculated as the final sample weight divided by initial sample weight.

- 12.0 Calculations and Data Analysis
- 12.1 Nomenclature.

As= Chromatogram area counts of vinyl chloride for the sample, area counts.

- A<sub>s</sub>= Chromatogram area counts of vinyl chloride for the sample.
- C<sub>c</sub>= Concentration of vinyl chloride in the standard sample, ppm.

K<sub>p</sub>= Henry's Law Constant for VCM in PVC 90 °C, 6.52 × 10<sup>-6</sup>g/g/mm Hg.

- K<sub>w</sub>= Henry's Law Constant for VCM in water 90 °C,  $7 \times 10^{-7}$ g/g/mm Hg.
- M<sub>v</sub>= Molecular weight of VCM, 62.5 g/mole.
- m = Sample weight, g.
- Pa= Ambient atmospheric pressure, mm Hg.
- R = Gas constant, (62360<sup>3</sup> ml) (mm Hg)/(mole)(°K).
- R<sub>f</sub>= Response factor in area counts per ppm VCM.
- R<sub>s</sub>= Response factor, area counts/ppm.
- T<sub>i</sub>= Ambient laboratory temperature, °K.
- TS = Total solids expressed as a decimal fraction.

T<sub>2</sub>= Equilibrium temperature, °K.

V<sub>g</sub>= Volume of vapor phase, ml.

$$=V_{\gamma}-\frac{m(TS)}{1.36}-\frac{m(1-TS)}{0.9653}$$

V<sub>v</sub>= Vial volume,<sup>3</sup> ml.

1.36 = Density of PVC at 90 °C,  $g/^3$  ml.

0.9653 = Density of water at 90 °C, g/<sup>3</sup> ml.

12.2 Response Factor. If the calibration curve described in Section 10.3 passes through zero, an average response factor, R<sub>I</sub>, may be used to facilitate computation of vinyl chloride sample concentrations.

12.2.1 To compute  $R_f$ , first compute a response factor,  $R_s$ , for each sample as follows:

$$R_{s} = \frac{A_{s}}{C_{c}}$$
 Eq. 107-2

12.2.2 Sum the individual response factors, and calculate  $R_r$ . If the calibration curve does not pass through zero, use the calibration curve to determine each sample concentration.

12.3 Residual Vinyl Chloride Monomer Concentration, (C<sub>rvc</sub>) or Vinyl Chloride Monomer Concentration. Calculate C<sub>rvc</sub>in ppm or mg/kg as follows:

$$C_{rw} = \frac{A_{s}P_{a}}{R_{f}T_{1}} \left[ \frac{M_{v}V_{g}}{Rm} + K_{p}(TS)T_{2}K_{w}(1-TS)T_{2} \right] \qquad \text{Eq. 107-3}$$

Note: Results calculated using these equations represent concentration based on the total sample. To obtain results based on dry PVC content, divide by TS.

#### 13.0 Method Performance

13.1 Range and Sensitivity. The lower limit of detection of vinyl chloride will vary according to the sampling and chromatographic system. The system should be capable of producing a measurement for a 50-ppm vinyl chloride standard that is at least 10 times the standard deviation of the system background noise level.

13.2 An interlaboratory comparison between seven laboratories of three resin samples, each split into three parts, yielded a standard deviation of 2.63 percent for a sample with a mean of 2.09 ppm, 4.16 percent for a sample with a mean of 1.66 ppm, and 5.29 percent for a sample with a mean of 62.66 ppm.

#### 14.0 Pollution Prevention[Reserved]

#### 15.0 Waste Management[Reserved]

#### 16.0 References

1. B.F. Goodrich, Residual Vinyl Chloride Monomer Content of Polyvinyl Chloride Resins, Latex, Wet Cake, Slurry and Water Samples. B.F. Goodrich Chemical Group Standard Test Procedure No. 1005-E. B.F. Goodrich Technical Center, Avon Lake, Ohio. October 8, 1979.

## **APPENDIX D**

40 CFR Part 60, Subpart Kb

Standards of Performance for Volatile Organic Liquid Storage Vessels (Including Petroleum Liquid Storage Vessels) for Which Construction, Reconstruction, or Modification Commenced After July 23, 1984 .

# e-CFR Data is current as of October 6, 2009

# Title 40: Protection of Environment

PART 60-STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES

Browse Previous | Browse Next

### Subpart Kb—Standards of Performance for Volatile Organic Liquid Storage Vessels (Including Petroleum Liquid Storage Vessels) for Which Construction, Reconstruction, or Modification Commenced After July 23, 1984

Source: 52 FR 11429, Apr. 8, 1987, unless otherwise noted.

### § 60.110b Applicability and designation of affected facility.

(a) Except as provided in paragraph (b) of this section, the affected facility to which this subpart applies is each storage vessel with a capacity greater than or equal to 75 cubic meters (m<sup>3</sup>) that is used to store volatile organic liquids (VOL) for which construction, reconstruction, or modification is commenced after July 23, 1984.

(b) This subpart does not apply to storage vessels with a capacity greater than or equal to  $151 \text{ m}^3$  storing a liquid with a maximum true vapor pressure less than 3.5 kilopascals (kPa) or with a capacity greater than or equal to 75 m<sup>3</sup> but less than 151 m<sup>3</sup> storing a liquid with a maximum true vapor pressure less than 15.0 kPa.

(c) [Reserved]

- (d) This subpart does not apply to the following:
- (1) Vessels at coke oven by-product plants.

(2) Pressure vessels designed to operate in excess of 204.9 kPa and without emissions to the atmosphere.

(3) Vessels permanently attached to mobile vehicles such as trucks, railcars, barges, or ships.

(4) Vessels with a design capacity less than or equal to 1,589.874 m<sup>3</sup> used for petroleum or condensate stored, processed, or treated prior to custody transfer.

- (5) Vessels located at bulk gasoline plants.
- (6) Storage vessels located at gasoline service stations.
- (7) Vessels used to store beverage alcohol.
- (8) Vessels subject to subpart GGGG of 40 CFR part 63.

(e) Alternative means of compliance —(1) Option to comply with part 65. Owners or operators may choose to comply with 40 CFR part 65, subpart C, to satisfy the requirements of §§60.112b through 60.117b for storage vessels that are subject to this subpart that meet the specifications in paragraphs (e)(1)(i) and (ii) of this section. When choosing to comply with 40 CFR part 65, subpart C, the monitoring requirements of §60.116b(c), (e), (f)(1), and (g) still apply. Other provisions applying to owners or operators who choose to comply with 40 CFR part 65 are provided in 40 CFR 65.1.

(i) A storage vessel with a design capacity greater than or equal to 151 m<sup>3</sup> containing a VOL that, as stored, has a maximum true vapor pressure equal to or greater than 5.2 kPa; or

(ii) A storage vessel with a design capacity greater than 75 m<sup>3</sup> but less than 151 m<sup>3</sup> containing a VOL that, as stored, has a maximum true vapor pressure equal to or greater than 27.6 kPa.

(2) Part 60, subpart A. Owners or operators who choose to comply with 40 CFR part 65, subpart C, must also comply with §§60.1, 60.2, 60.5, 60.6, 60.7(a)(1) and (4), 60.14, 60.15, and 60.16 for those storage vessels. All sections and

paragraphs of subpart A of this part that are not mentioned in this paragraph (e)(2) do not apply to owners or operators of storage vessels complying with 40 CFR part 65, subpart C, except that provisions required to be met prior to implementing 40 CFR part 65 still apply. Owners and operators who choose to comply with 40 CFR part 65, subpart C, must comply with 40 CFR part 65, subpart A.

(3) Internal floating roof report. If an owner or operator installs an internal floating roof and, at initial startup, chooses to comply with 40 CFR part 65, subpart C, a report shall be furnished to the Administrator stating that the control equipment meets the specifications of 40 CFR 65.43. This report shall be an attachment to the notification required by 40 CFR 65.5(b).

(4) External floating roof report. If an owner or operator installs an external floating roof and, at initial startup, chooses to comply with 40 CFR part 65, subpart C, a report shall be furnished to the Administrator stating that the control equipment meets the specifications of 40 CFR 65.44. This report shall be an attachment to the notification required by 40 CFR 65.5(b).

[52 FR 11429, Apr. 8, 1987, as amended at 54 FR 32973, Aug. 11, 1989; 65 FR 78275, Dec. 14, 2000; 68 FR 59332, Oct. 15, 2003]

#### § 60.111b Definitions.

Terms used in this subpart are defined in the Act, in subpart A of this part, or in this subpart as follows:

Bulk gasoline plant means any gasoline distribution facility that has a gasoline throughput less than or equal to 75,700 liters per day. Gasoline throughput shall be the maximum calculated design throughput as may be limited by compliance with an enforceable condition under Federal requirement or Federal, State or local law, and discoverable by the Administrator and any other person.

Condensate means hydrocarbon liquid separated from natural gas that condenses due to changes in the temperature or pressure, or both, and remains liquid at standard conditions.

Custody transfer means the transfer of produced petroleum and/or condensate, after processing and/or treatment in the producing operations, from storage vessels or automatic transfer facilities to pipelines or any other forms of transportation.

Fill means the introduction of VOL into a storage vessel but not necessarily to complete capacity.

Gasoline service station means any site where gasoline is dispensed to motor vehicle fuel tanks from stationary storage tanks.

Maximum true vapor pressure means the equilibrium partial pressure exerted by the volatile organic compounds (as defined in 40 CFR 51.100) in the stored VOL at the temperature equal to the highest calendar-month average of the VOL storage temperature for VOL's stored above or below the ambient temperature or at the local maximum monthly average temperature as reported by the National Weather Service for VOL's stored at the ambient temperature, as determined:

(1) In accordance with methods described in American Petroleum institute Bulletin 2517, Evaporation Loss From External Floating Roof Tanks, (incorporated by reference—see §60.17); or

(2) As obtained from standard reference texts; or

(3) As determined by ASTM D2879-83, 96, or 97 (incorporated by reference—see §60.17);

(4) Any other method approved by the Administrator.

Petroleum means the crude oil removed from the earth and the oils derived from tar sands, shale, and coal.

Petroleum liquids means petroleum, condensate, and any finished or intermediate products manufactured in a petroleum refinery.

*Process tank* means a tank that is used within a process (including a solvent or raw material recovery process) to collect material discharged from a feedstock storage vessel or equipment within the process before the material is transferred to other equipment within the process, to a product or by-product storage vessel, or to a vessel used to store recovered solvent or raw material. In many process tanks, unit operations such as reactions and blending are conducted. Other process tanks, such as surge control vessels and bottoms receivers, however, may not involve unit operations.

*Reid vapor pressure* means the absolute vapor pressure of volatile crude oil and volatile nonviscous petroleum liquids except liquified petroleum gases, as determined by ASTM D323–82 or 94 (incorporated by reference—see §60.17).

Storage vessel means each tank, reservoir, or container used for the storage of volatile organic liquids but does not include:

(1) Frames, housing, auxiliary supports, or other components that are not directly involved in the containment of liquids or vapors;

(2) Subsurface caverns or porous rock reservoirs; or

(3) Process tanks.

Volatile organic liquid (VOL) means any organic liquid which can emit volatile organic compounds (as defined in 40 CFR 51.100) into the atmosphere.

Waste means any liquid resulting from industrial, commercial, mining or agricultural operations, or from community activities that is discarded or is being accumulated, stored, or physically, chemically, or biologically treated prior to being discarded or recycled.

[52 FR 11429, Apr. 8, 1987, as amended at 54 FR 32973, Aug. 11, 1989; 65 FR 61756, Oct. 17, 2000; 68 FR 59333, Oct. 15, 2003]

#### § 60.112b Standard for volatile organic compounds (VOC).

(a) The owner or operator of each storage vessel either with a design capacity greater than or equal to  $151 \text{ m}^3$  containing a VOL that, as stored, has a maximum true vapor pressure equal to or greater than 5.2 kPa but less than 76.6 kPa or with a design capacity greater than or equal to  $75 \text{ m}^3$  but less than 151 m<sup>3</sup> containing a VOL that, as stored, has a maximum true vapor pressure equal to or greater than 27.6 kPa but less than 76.6 kPa, shall equip each storage vessel with one of the following:

(1) A fixed roof in combination with an internal floating roof meeting the following specifications:

(i) The internal floating roof shall rest or float on the liquid surface (but not necessarily in complete contact with it) inside a storage vessel that has a fixed roof. The internal floating roof shall be floating on the liquid surface at all times, except during initial fill and during those intervals when the storage vessel is completely emptied or subsequently emptied and refilled. When the roof is resting on the leg supports, the process of filling, emptying, or refilling shall be continuous and shall be accomplished as rapidly as possible.

(ii) Each internal floating roof shall be equipped with one of the following closure devices between the wall of the storage vessel and the edge of the internal floating roof:

(A) A foam- or liquid-filled seal mounted in contact with the liquid (liquid-mounted seal). A liquid-mounted seal means a foam- or liquid-filled seal mounted in contact with the liquid between the wall of the storage vessel and the floating roof continuously around the circumference of the tank.

(B) Two seals mounted one above the other so that each forms a continuous closure that completely covers the space between the wall of the storage vessel and the edge of the internal floating roof. The lower seal may be vapor-mounted, but both must be continuous.

(C) A mechanical shoe seal. A mechanical shoe seal is a metal sheet held vertically against the wall of the storage vessel by springs or weighted levers and is connected by braces to the floating roof. A flexible coated fabric (envelope) spans the annular space between the metal sheet and the floating roof.

(iii) Each opening in a noncontact internal floating roof except for automatic bleeder vents (vacuum breaker vents) and the rim space vents is to provide a projection below the liquid surface.

(iv) Each opening in the internal floating roof except for leg sleeves, automatic bleeder vents, rim space vents, column wells, ladder wells, sample wells, and stub drains is to be equipped with a cover or lid which is to be maintained in a closed position at all times (i.e., no visible gap) except when the device is in actual use. The cover or lid shall be equipped with a gasket. Covers on each access hatch and automatic gauge float well shall be bolted except when they are in use.

(v) Automatic bleeder vents shall be equipped with a gasket and are to be closed at all times when the roof is floating except when the roof is being floated off or is being landed on the roof leg supports.

(vi) Rim space vents shall be equipped with a gasket and are to be set to open only when the internal floating roof is not floating or at the manufacturer's recommended setting.

(vii) Each penetration of the internal floating roof for the purpose of sampling shall be a sample well. The sample well shall have a slit fabric cover that covers at least 90 percent of the opening.

(viii) Each penetration of the internal floating roof that allows for passage of a column supporting the fixed roof shall have a flexible fabric sleeve seal or a gasketed sliding cover.

(ix) Each penetration of the internal floating roof that allows for passage of a ladder shall have a gasketed sliding cover.

(2) An external floating roof. An external floating roof means a pontoon-type or double-deck type cover that rests on the liquid surface in a vessel with no fixed roof. Each external floating roof must meet the following specifications:

(i) Each external floating roof shall be equipped with a closure device between the wall of the storage vessel and the roof edge. The closure device is to consist of two seals, one above the other. The lower seal is referred to as the primary seal, and the upper seal is referred to as the secondary seal.

(A) The primary seal shall be either a mechanical shoe seal or a liquid-mounted seal. Except as provided in §60.113b(b)(4), the seal shall completely cover the annular space between the edge of the floating roof and tank wall.

(B) The secondary seal shall completely cover the annular space between the external floating roof and the wall of the storage vessel in a continuous fashion except as allowed in §60.113b(b)(4).

(ii) Except for automatic bleeder vents and rim space vents, each opening in a noncontact external floating roof shall provide a projection below the liquid surface. Except for automatic bleeder vents, rim space vents, roof drains, and leg sleeves, each opening in the roof is to be equipped with a gasketed cover, seal, or lid that is to be maintained in a closed position at all times (i.e., no visible gap) except when the device is in actual use. Automatic bleeder vents are to be closed at all times when the roof is floating except when the roof is being floated off or is being landed on the roof leg supports. Rim vents are to be set to open when the roof is being floated off the roof legs supports or at the manufacturer's recommended setting. Automatic bleeder vents and rim space vents are to be gasketed. Each emergency roof drain is to be provided with a slotted membrane fabric cover that covers at least 90 percent of the area of the opening.

(iii) The roof shall be floating on the liquid at all times (i.e., off the roof leg supports) except during initial fill until the roof is lifted off leg supports and when the tank is completely emptied and subsequently refilled. The process of filling, emptying, or refilling when the roof is resting on the leg supports shall be continuous and shall be accomplished as rapidly as possible.

(3) A closed vent system and control device meeting the following specifications:

(i) The closed vent system shall be designed to collect all VOC vapors and gases discharged from the storage vessel and operated with no detectable emissions as indicated by an instrument reading of less than 500 ppm above background and visual inspections, as determined in part 60, subpart VV, §60.485(b).

(ii) The control device shall be designed and operated to reduce inlet VOC emissions by 95 percent or greater. If a flare is used as the control device, it shall meet the specifications described in the general control device requirements (§60.18) of the General Provisions.

(4) A system equivalent to those described in paragraphs (a)(1), (a)(2), or (a)(3) of this section as provided in §60.114b of this subpart.

(b) The owner or operator of each storage vessel with a design capacity greater than or equal to 75 m<sup>3</sup> which contains a VOL that, as stored, has a maximum true vapor pressure greater than or equal to 76.6 kPa shall equip each storage vessel with one of the following:

(1) A closed vent system and control device as specified in §60.112b(a)(3).

(2) A system equivalent to that described in paragraph (b)(1) as provided in §60.114b of this subpart.

(c) Site-specific standard for Merck & Co., Inc.'s Stonewall Plant in Elkton, Virginia. This paragraph applies only to the pharmaceutical manufacturing facility, commonly referred to as the Stonewall Plant, located at Route 340 South, in Elkton, Virginia ("site").

(1) For any storage vessel that otherwise would be subject to the control technology requirements of paragraphs (a) or (b) of this section, the site shall have the option of either complying directly with the requirements of this subpart, or reducing the site-wide total criteria pollutant emissions cap (total emissions cap) in accordance with the procedures set forth in a permit issued pursuant to 40 CFR 52.2454. If the site chooses the option of reducing the total emissions cap in accordance with the procedures set forth in such permit, the requirements of such permit shall apply in lieu of the otherwise applicable requirements of this subpart for such storage vessel.

(2) For any storage vessel at the site not subject to the requirements of 40 CFR 60.112b (a) or (b), the requirements of 40 CFR 60.116b (b) and (c) and the General Provisions (subpart A of this part) shall not apply.

[52 FR 11429, Apr. 8, 1987, as amended at 62 FR 52641, Oct. 8, 1997]

### § 60.113b Testing and procedures.

The owner or operator of each storage vessel as specified in §60.112b(a) shall meet the requirements of paragraph (a), (b), or (c) of this section. The applicable paragraph for a particular storage vessel depends on the control equipment installed to meet the requirements of §60.112b.

(a) After installing the control equipment required to meet §60.112b(a)(1) (permanently affixed roof and internal floating roof), each owner or operator shall:

(1) Visually inspect the internal floating roof, the primary seal, and the secondary seal (if one is in service), prior to filling the storage vessel with VOL. If there are holes, tears, or other openings in the primary seal, the secondary seal, or the seal fabric or defects in the internal floating roof, or both, the owner or operator shall repair the items before filling the storage vessel.

(2) For Vessels equipped with a liquid-mounted or mechanical shoe primary seal, visually inspect the internal floating roof and the primary seal or the secondary seal (if one is in service) through manholes and roof hatches on the fixed roof at least once every 12 months after initial fill. If the internal floating roof is not resting on the surface of the VOL inside the storage vessel, or there is liquid accumulated on the roof, or the seal is detached, or there are holes or tears in the seal fabric, the owner or operator shall repair the items or empty and remove the storage vessel from service within 45 days. If a failure that is detected during inspections required in this paragraph cannot be repaired within 45 days and if the vessel cannot be emptied within 45 days, a 30-day extension may be requested from the Administrator in the inspection report required in §60.115b(a)(3). Such a request for an extension must document that alternate storage capacity is unavailable and specify a schedule of actions the company will take that will assure that the control equipment will be repaired or the vessel will be emptied as soon as possible.

(3) For vessels equipped with a double-seal system as specified in §60.112b(a)(1)(ii)(B):

(i) Visually inspect the vessel as specified in paragraph (a)(4) of this section at least every 5 years; or

(ii) Visually inspect the vessel as specified in paragraph (a)(2) of this section.

(4) Visually inspect the internal floating roof, the primary seal, the secondary seal (if one is in service), gaskets, slotted membranes and sleeve seals (if any) each time the storage vessel is emptied and degassed. If the internal floating roof has defects, the primary seal has holes, tears, or other openings in the seal or the seal fabric, or the secondary seal has holes, tears, or other openings in the seal or the seal fabric, or the secondary seal has holes, tears, or other openings in the seal or the seal fabric, or the secondary seal has holes, tears, or other openings in the seal or the seal fabric, or the gaskets no longer close off the liquid surfaces from the atmosphere, or the slotted membrane has more than 10 percent open area, the owner or operator shall repair the items as necessary so that none of the conditions specified in this paragraph exist before refilling the storage vessel with VOL. In no event shall inspections conducted in accordance with this provision occur at intervals greater than 10 years in the case of vessels conducting the annual visual inspection as specified in paragraphs (a)(2) and (a)(3)(ii) of this section and at intervals no greater than 5 years in the case of vessels specified in paragraph (a)(3)(i) of this section.

(5) Notify the Administrator in writing at least 30 days prior to the filling or refilling of each storage vessel for which an inspection is required by paragraphs (a)(1) and (a)(4) of this section to afford the Administrator the opportunity to have an observer present. If the inspection required by paragraph (a)(4) of this section is not planned and the owner or operator could not have known about the inspection 30 days in advance or refilling the tank, the owner or operator shall notify the Administrator at least 7 days prior to the refilling of the storage vessel. Notification shall be made by telephone immediately followed by written documentation demonstrating why the inspection was unplanned. Alternatively, this notification including the written documentation may be made in writing and sent by express mail so that it is received by the Administrator at least 7 days prior to the refilling.

(b) After installing the control equipment required to meet §60.112b(a)(2) (external floating roof), the owner or operator shall:

(1) Determine the gap areas and maximum gap widths, between the primary seal and the wall of the storage vessel and between the secondary seal and the wall of the storage vessel according to the following frequency.

(i) Measurements of gaps between the tank wall and the primary seal (seal gaps) shall be performed during the hydrostatic testing of the vessel or within 60 days of the initial fill with VOL and at least once every 5 years thereafter.

(ii) Measurements of gaps between the tank wall and the secondary seal shall be performed within 60 days of the initial fill with VOL and at least once per year thereafter.

(iii) If any source ceases to store VOL for a period of 1 year or more, subsequent introduction of VOL into the vessel shall be considered an initial fill for the purposes of paragraphs (b)(1)(i) and (b)(1)(i) of this section.

(2) Determine gap widths and areas in the primary and secondary seals individually by the following procedures:

(i) Measure seal gaps, if any, at one or more floating roof levels when the roof is floating off the roof leg supports.

(ii) Measure seal gaps around the entire circumference of the tank in each place where a 0.32-cm diameter uniform probe passes freely (without forcing or binding against seal) between the seal and the wall of the storage vessel and measure the circumferential distance of each such location.

(iii) The total surface area of each gap described in paragraph (b)(2)(ii) of this section shall be determined by using probes of various widths to measure accurately the actual distance from the tank wall to the seal and multiplying each such width by its respective circumferential distance.

(3) Add the gap surface area of each gap location for the primary seal and the secondary seal individually and divide the sum for each seal by the nominal diameter of the tank and compare each ratio to the respective standards in paragraph (b)(4) of this section.

(4) Make necessary repairs or empty the storage vessel within 45 days of identification in any inspection for seals not meeting the requirements listed in (b)(4) (i) and (ii) of this section:

(i) The accumulated area of gaps between the tank wall and the mechanical shoe or liquid-mounted primary seal shall not exceed 212 Cm<sup>2</sup> per meter of tank diameter, and the width of any portion of any gap shall not exceed 3.81 cm.

(A) One end of the mechanical shoe is to extend into the stored liquid, and the other end is to extend a minimum vertical distance of 61 cm above the stored liquid surface.

(B) There are to be no holes, tears, or other openings in the shoe, seal fabric, or seal envelope.

(ii) The secondary seal is to meet the following requirements:

(A) The secondary seal is to be installed above the primary seal so that it completely covers the space between the roof edge and the tank wall except as provided in paragraph (b)(2)(iii) of this section.

(B) The accumulated area of gaps between the tank wall and the secondary seal shall not exceed 21.2 cm<sup>2</sup> per meter of tank diameter, and the width of any portion of any gap shall not exceed 1.27 cm.

(C) There are to be no holes, tears, or other openings in the seal or seal fabric.

(iii) If a failure that is detected during inspections required in paragraph (b)(1) of §60.113b(b) cannot be repaired within 45 days and if the vessel cannot be emptied within 45 days, a 30-day extension may be requested from the Administrator in the inspection report required in §60.115b(b)(4). Such extension request must include a demonstration of unavailability of alternate storage capacity and a specification of a schedule that will assure that the control equipment will be repaired or the vessel will be emptied as soon as possible.

(5) Notify the Administrator 30 days in advance of any gap measurements required by paragraph (b)(1) of this section to afford the Administrator the opportunity to have an observer present.

(6) Visually inspect the external floating roof, the primary seal, secondary seal, and fittings each time the vessel is emptied and degassed.

(i) If the external floating roof has defects, the primary seal has holes, tears, or other openings in the seal or the seal fabric, or the secondary seal has holes, tears, or other openings in the seal or the seal fabric, the owner or operator shall repair the items as necessary so that none of the conditions specified in this paragraph exist before filling or refilling the storage vessel with VOL.

(ii) For all the inspections required by paragraph (b)(6) of this section, the owner or operator shall notify the Administrator in writing at least 30 days prior to the filling or refilling of each storage vessel to afford the Administrator the opportunity to inspect the storage vessel prior to refilling. If the inspection required by paragraph (b)(6) of this section is not planned and the owner or operator could not have known about the inspection 30 days in advance of refilling the tank, the owner or operator shall notify the Administrator at least 7 days prior to the refilling of the storage vessel. Notification shall be made by telephone immediately followed by written documentation demonstrating why the inspection was unplanned. Alternatively, this notification including the written documentation may be made in writing and sent by express mail so that it is received by the Administrator at least 7 days prior to the refilling.

(c) The owner or operator of each source that is equipped with a closed vent system and control device as required in §60.112b (a)(3) or (b)(2) (other than a flare) is exempt from §60.8 of the General Provisions and shall meet the following requirements.

(1) Submit for approval by the Administrator as an attachment to the notification required by 60.7(a)(1) or, if the facility is exempt from 60.7(a)(1), as an attachment to the notification required by 60.7(a)(2), an operating plan containing the information listed below.

(i) Documentation demonstrating that the control device will achieve the required control efficiency during maximum loading conditions. This documentation is to include a description of the gas stream which enters the control device, including flow and VOC content under varying liquid level conditions (dynamic and static) and manufacturer's design specifications for the control device. If the control device or the closed vent capture system receives vapors, gases, or liquids other than fuels from sources that are not designated sources under this subpart, the efficiency demonstration is to include consideration of all vapors, gases, and liquids received by the closed vent capture system and control device. If an enclosed combustion device with a minimum residence time of 0.75 seconds and a minimum temperature of 816 °C is used to meet the 95 percent requirement, documentation that those conditions will exist is sufficient to meet the requirements of this paragraph.

(ii) A description of the parameter or parameters to be monitored to ensure that the control device will be operated in conformance with its design and an explanation of the criteria used for selection of that parameter (or parameters).

(2) Operate the closed vent system and control device and monitor the parameters of the closed vent system and control device in accordance with the operating plan submitted to the Administrator in accordance with paragraph (c)(1) of this section, unless the plan was modified by the Administrator during the review process. In this case, the modified plan applies.

(d) The owner or operator of each source that is equipped with a closed vent system and a flare to meet the requirements in (0, 1) or (b)(2) shall meet the requirements as specified in the general control device requirements, (0, 1) (e) and (f).

[52 FR 11429, Apr. 8, 1987, as amended at 54 FR 32973, Aug. 11, 1989]

#### § 60.114b Alternative means of emission limitation.

(a) If, in the Administrator's judgment, an alternative means of emission limitation will achieve a reduction in emissions at least equivalent to the reduction in emissions achieved by any requirement in §60.112b, the Administrator will publish in theFederal Registera notice permitting the use of the alternative means for purposes of compliance with that requirement.

(b) Any notice under paragraph (a) of this section will be published only after notice and an opportunity for a hearing.

(c) Any person seeking permission under this section shall submit to the Administrator a written application including:

(1) An actual emissions test that uses a full-sized or scale-model storage vessel that accurately collects and measures all VOC emissions from a given control device and that accurately simulates wind and accounts for other emission variables such as temperature and barometric pressure.

(2) An engineering evaluation that the Administrator determines is an accurate method of determining equivalence.

(d) The Administrator may condition the permission on requirements that may be necessary to ensure operation and maintenance to achieve the same emissions reduction as specified in §60.112b.

#### § 60.115b Reporting and recordkeeping requirements.

The owner or operator of each storage vessel as specified in 60.112b(a) shall keep records and furnish reports as required by paragraphs (a), (b), or (c) of this section depending upon the control equipment installed to meet the requirements of 60.112b. The owner or operator shall keep copies of all reports and records required by this section, except for the record required by (c)(1), for at least 2 years. The record required by (c)(1) will be kept for the life of the control equipment.

(a) After installing control equipment in accordance with §60.112b(a)(1) (fixed roof and internal floating roof), the owner or operator shall meet the following requirements.

(1) Furnish the Administrator with a report that describes the control equipment and certifies that the control equipment meets the specifications of (1) and (1) and (1). This report shall be an attachment to the notification required by (0, 1).

(2) Keep a record of each inspection performed as required by §60.113b (a)(1), (a)(2), (a)(3), and (a)(4). Each record shall identify the storage vessel on which the inspection was performed and shall contain the date the vessel was inspected and the observed condition of each component of the control equipment (seals, internal floating roof, and fittings).

(3) If any of the conditions described in §60.113b(a)(2) are detected during the annual visual inspection required by §60.113b(a)(2), a report shall be furnished to the Administrator within 30 days of the inspection. Each report shall identify the storage vessel, the nature of the defects, and the date the storage vessel was emptied or the nature of and date the repair was made.

(4) After each inspection required by  $\S60.113b(a)(3)$  that finds holes or tears in the seal or seal fabric, or defects in the internal floating roof, or other control equipment defects listed in  $\S60.113b(a)(3)(ii)$ , a report shall be furnished to the Administrator within 30 days of the inspection. The report shall identify the storage vessel and the reason it did not meet the specifications of  $\S61.112b(a)(1)$  or  $\S60.113b(a)(3)$  and list each repair made.

(b) After installing control equipment in accordance with §61.112b(a)(2) (external floating roof), the owner or operator shall meet the following requirements.

(1) Furnish the Administrator with a report that describes the control equipment and certifies that the control equipment meets the specifications of (0.112b(a))(2) and (0.113b(b))(2), (b)(3), and (b)(4). This report shall be an attachment to the notification required by (0.12b(a))(3).

(2) Within 60 days of performing the seal gap measurements required by §60.113b(b)(1), furnish the Administrator with a report that contains:

- (i) The date of measurement.
- (ii) The raw data obtained in the measurement.
- (iii) The calculations described in §60.113b (b)(2) and (b)(3).

(3) Keep a record of each gap measurement performed as required by §60.113b(b). Each record shall identify the storage vessel in which the measurement was performed and shall contain:

- (i) The date of measurement.
- (ii) The raw data obtained in the measurement.
- (iii) The calculations described in §60.113b (b)(2) and (b)(3).

(4) After each seal gap measurement that detects gaps exceeding the limitations specified by §60.113b(b)(4), submit a report to the Administrator within 30 days of the inspection. The report will identify the vessel and contain the information specified in paragraph (b)(2) of this section and the date the vessel was emptied or the repairs made and date of repair.

(c) After installing control equipment in accordance with §60.112b (a)(3) or (b)(1) (closed vent system and control device other than a flare), the owner or operator shall keep the following records.

(1) A copy of the operating plan.

(2) A record of the measured values of the parameters monitored in accordance with §60.113b(c)(2).

(d) After installing a closed vent system and flare to comply with §60.112b, the owner or operator shall meet the following requirements.

(1) A report containing the measurements required by §60.18(f) (1), (2), (3), (4), (5), and (6) shall be furnished to the Administrator as required by §60.8 of the General Provisions. This report shall be submitted within 6 months of the initial start-up date.

(2) Records shall be kept of all periods of operation during which the flare pilot flame is absent.

(3) Semiannual reports of all periods recorded under §60.115b(d)(2) in which the pilot flame was absent shall be furnished to the Administrator.

#### § 60.116b Monitoring of operations.

(a) The owner or operator shall keep copies of all records required by this section, except for the record required by paragraph (b) of this section, for at least 2 years. The record required by paragraph (b) of this section will be kept for the life of the source.

(b) The owner or operator of each storage vessel as specified in §60.110b(a) shall keep readily accessible records showing the dimension of the storage vessel and an analysis showing the capacity of the storage vessel.

(c) Except as provided in paragraphs (f) and (g) of this section, the owner or operator of each storage vessel either with a design capacity greater than or equal to  $151 \text{ m}^3$  storing a liquid with a maximum true vapor pressure greater than or equal to 3.5 kPa or with a design capacity greater than or equal to  $75 \text{ m}^3$  but less than  $151 \text{ m}^3$  storing a liquid with a maximum true vapor pressure greater than or equal to 15.0 kPa shall maintain a record of the VOL stored, the period of storage, and the maximum true vapor pressure of that VOL during the respective storage period.

(d) Except as provided in paragraph (g) of this section, the owner or operator of each storage vessel either with a design capacity greater than or equal to 151 m<sup>3</sup> storing a liquid with a maximum true vapor pressure that is normally less than 5.2 kPa or with a design capacity greater than or equal to 75 m<sup>3</sup> but less than 151 m<sup>3</sup> storing a liquid with a maximum true vapor pressure that is normally less than 27.6 kPa shall notify the Administrator within 30 days when the maximum true vapor pressure of the liquid exceeds the respective maximum true vapor pressure values for each volume range.

(e) Available data on the storage temperature may be used to determine the maximum true vapor pressure as determined below.

(1) For vessels operated above or below ambient temperatures, the maximum true vapor pressure is calculated based upon the highest expected calendar-month average of the storage temperature. For vessels operated at ambient temperatures, the maximum true vapor pressure is calculated based upon the maximum local monthly average ambient temperature as reported by the National Weather Service.

(2) For crude oil or refined petroleum products the vapor pressure may be obtained by the following:

(i) Available data on the Reid vapor pressure and the maximum expected storage temperature based on the highest expected calendar-month average temperature of the stored product may be used to determine the maximum true vapor pressure from nomographs contained in API Bulletin 2517 (incorporated by reference—see §60.17), unless the Administrator specifically requests that the liquid be sampled, the actual storage temperature determined, and the Reid vapor pressure determined from the sample(s).

(ii) The true vapor pressure of each type of crude oil with a Reid vapor pressure less than 13.8 kPa or with physical properties that preclude determination by the recommended method is to be determined from available data and recorded if the estimated maximum true vapor pressure is greater than 3.5 kPa.

(3) For other liquids, the vapor pressure:

(i) May be obtained from standard reference texts, or

(ii) Determined by ASTM D2879-83, 96, or 97 (incorporated by reference-see §60.17); or

(iii) Measured by an appropriate method approved by the Administrator; or

(iv) Calculated by an appropriate method approved by the Administrator.

(f) The owner or operator of each vessel storing a waste mixture of indeterminate or variable composition shall be subject to the following requirements.

(1) Prior to the initial filling of the vessel, the highest maximum true vapor pressure for the range of anticipated liquid compositions to be stored will be determined using the methods described in paragraph (e) of this section.

(2) For vessels in which the vapor pressure of the anticipated liquid composition is above the cutoff for monitoring but below the cutoff for controls as defined in §60.112b(a), an initial physical test of the vapor pressure is required; and a physical test at least once every 6 months thereafter is required as determined by the following methods:

(i) ASTM D2879-83, 96, or 97 (incorporated by reference-see §60.17); or

(ii) ASTM D323-82 or 94 (incorporated by reference-see §60.17); or

(iii) As measured by an appropriate method as approved by the Administrator.

(g) The owner or operator of each vessel equipped with a closed vent system and control device meeting the specification of §60.112b or with emissions reductions equipment as specified in 40 CFR 65.42(b)(4), (b)(5), (b)(6), or (c) is exempt from the requirements of paragraphs (c) and (d) of this section.

[52 FR 11429, Apr. 8, 1987, as amended at 65 FR 61756, Oct. 17, 2000; 65 FR 78276, Dec. 14, 2000; 68 FR 59333, Oct. 15, 2003]

#### § 60.117b Delegation of authority.

(a) In delegating implementation and enforcement authority to a State under section 111(c) of the Act, the authorities contained in paragraph (b) of this section shall be retained by the Administrator and not transferred to a State.

(b) Authorities which will not be delegated to States: §§60.111b(f)(4), 60.114b, 60.116b(e)(3)(iii), 60.116b(e)(3)(iv), and 60.116b(f)(2)(iii).

[52 FR 11429, Apr. 8, 1987, as amended at 52 FR 22780, June 16, 1987]

Browse Previous | Browse Next

For questions or comments regarding e-CFR editorial content, features, or design, email ecfr@nara.gov.

For questions concerning e-CFR programming and delivery issues, email webteam@gpo.gov.

# APPENDIX E

Example of Loading Rack Emission Calculation

#### Appendix E

#### Example of Loading Rack Emission Calculations Format for Loading Racks

 VOC Emission Calculation:

 Equation
 LL = 12.46 \* S \* P \* M / T

where

- LL = S = P = loading loss, pounds per 1000 gallons of liquid loaded saturation factor from AP-42 Table 5.2-1 true vapor pressure of liquid loaded, psia
- molecular weight of vapors, pounds per pound-mole (see AP-42 Table 7.1-2) temperature of bulk liquid loaded, R (F + 460) =
- . М Т =

SN-14 Diesel, Naptha, Kerosene Loading Rack

Product	S	1997 <b>-</b> 1997 -	M	T	LL
Diesel	0.5	0.0074	130	520	0.012
Naptha	0.5	1.3	80	520	1.246
Kerosene	0.5	0.0085	130	520	0.013

#### VOC Emissions

Product	Hourly Production Rate (gallons)	Yearly Throughput (gallons)	VOC Emission Rate (lb/hr)	VOC Emission Rate (tpy)
Diesel	0	21,949,306	0.000	0.126
Naptha	0	3,010,224	0.000	1.875
Kerosene	0	300,048	0.000	0.002
		Maximum	0.000	2.004

#### HAP Emissions

	k: No. La	Diesel	Case 1.	() (Fight)	Kerosene	e a search an	s a de se la	Naphtha	and the l	olices <b>T</b> e	tal
Pollutant	HAP Wt%	b/hr	tpy	HAP Wt%	ib/hr	tpy .	HAP Wt%	lb/hr :	tpy	lb/hr	ton/yr
2,2,4-Trimethylpentane	0.284	0.000	0.006	0.327	0.000	0.007	0.355	0.000	0.007	0.000	0.019
Benzene	12.383	0.000	0.248	2.149	0.000	0.043	1.063	0.000	0.021	0.000	0.312
Cumene	0.375	0.000	0.008	0.194	0.000	0.004	0.037	0.000	0.001	0.000	0.012
Ethylbenzene	0.876	0.000	0.018	0.892	0.000	0.018	0.118	0.000	0.002	0.000	0.038
Hexane	24.761	0.000	0.496	22.823	0.000	0.457	10.266	0.000	0.206	0.000	1.159
Naphthalene	0.089	0.000	0.002	0.080	0.000	0.002	0.001	0.000	0.000	0.000	0.003
Phenol	0.013	0.000	0.000	0.014	0.000	0.000	0.000	0.000	0.000	0.000	0.001
Styrene	0.079	0.000	0.002	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.002
Toluene	4.357	0.000	0.087	6.003	0.000	0.120	1.295	0.000	0.026	0.000	0.234
Xylene	2.453	0.000	0.049	2.910	0.000	0.058	0.395	0.000	0.008	0.000	0.115
Total HAP										0.00	1.90

Product S P M T			
	roduct	The L	
Asphalt 1.45 0.002 165 960	sphalt	960 0.006	)6

#### VOC Emissions

Product	Production Rate (gallons)	Yearly Throughput (gallons)	Emission Rate (lb/hr)	Emission Rate (tpy)
Asphalt	0	14,930,110	0.000	0.046

SN-17/SN-18/SN-21	Lube Oil Loa	ding			
Product	S	P	N ST	i tin	LL
Lube Oil	1.45	0.0003	150	520	0.002

#### VOC Emissions

Product	Hourly Production Rate (gallons)	Yearly Throughput (gallons)	Emission Rate (lb/hr)	Emission Rate (tpy)
Lube Oil	0	62,595,370	0.000	0.049

#### HAP Emissions:

Pollutant	HAP Wt %	HAP Emissions (lb/hr)	HAP Emissions (tpy)
Benzene	22,755	0.00	0.01
Hexane	28,608	0.00	0.01
Toluene	7.031	0.00	0.00
Total HAP		0.00	0.03

# **CERTIFICATE OF SERVICE**

I, Cynthia Hook, hereby certify that a copy of this permit has been mailed by first class mail to Martin Operating Partnership L. P., 484 East 6th Street, Smackover, AR, 71762, on this  $\frac{2}{5}$ 

day of December, 2009.

Cynthia Hook, AAII, Air Division