

(3) The Administrator shall be notified at least 30 days prior to an emission test, so that he may at his option observe the test.

(4) Samples shall be taken over such a period or periods as are necessary to determine accurately the maximum emissions which will occur in a 24-hour period. No changes shall be made in the operation which would potentially increase emissions above the level determined by the most recent stack test, until the new emission level has been estimated by calculation and the results reported to the Administrator.

(5) All samples shall be analyzed, and mercury emissions shall be determined within 30 days after the stack test. Each determination shall be reported to the Administrator by a registered letter dispatched before the close of the next business day following such determination.

(6) Records of emission test results and other data needed to determine total emissions shall be retained at the source and shall be made available, for inspection by the Administrator, for a minimum of 2 years.

14. Sections 61.54 and 61.55 are added as follows:

§ 61.54 Sludge sampling.

(a) As an alternative means for demonstrating compliance with § 61.52 (b), an owner or operator may use Method 105 of Appendix B and the procedures specified in this section.

(1) A sludge test shall be conducted within 90 days of the effective date of these regulations in the case of an existing source or a new source which has an initial startup date preceding the effective date; or

(2) A sludge test shall be conducted within 90 days of startup in the case of a new source which did not have an initial startup date preceding the effective date.

(b) The Administrator shall be notified at least 30 days prior to a sludge sampling test, so that he may at his option observe the test.

(c) Sludge shall be sampled according to paragraph (c)(1) of this section, sludge charging rate for the plant shall be determined according to paragraph (c)(2) of this section, and the sludge analysis shall be performed according to paragraph (c)(3) of this section.

(1) The sludge shall be sampled after dewatering and before incineration or drying, at a location that provides a representative sample of the sludge that is charged to the incinerator or dryer. Eight consecutive grab samples shall be obtained at intervals of between 45 and 60 minutes and thoroughly mixed into one sample. Each of the eight grab samples shall have a volume of at least 200 ml but not more than 400 ml. A total of three composite samples shall be obtained within an operating period of 24 hours. When the 24-hour operating period is not continuous, the total sampling period shall not exceed 72 hours after the first grab sample is obtained. Samples shall not be exposed to any condition that may result in mercury contamination or loss.

(2) The maximum 24-hour period sludge incineration or drying rate shall be determined by use of a flow rate measurement device that can measure the mass rate of sludge charged to the incinerator or dryer with an accuracy of ± 5 percent over its operating range. Other methods of measuring sludge mass charging rates may be used if they have received prior approval by the Administrator.

(3) The handling, preparation, and analysis of sludge samples shall be accomplished according to Method 105 in Appendix B of this part.

(d) The mercury emissions shall be determined by use of the following equation:

$$E_{Hg} = 1 \times 10^{-6} \text{ cQ}$$

where

E_{Hg} —Mercury emissions, g/day.

c —Mercury concentration of sludge on a dry solids basis, $\mu\text{g/g}$ (ppm).

Q —Sludge charging rate, kg/day.

(e) No changes in the operation of a plant shall be made after a sludge test has been conducted which would potentially increase emissions above the level determined by the most recent sludge test, until the new emission level has been estimated by calculation and the results reported to the Administrator.

(f) All sludge samples shall be analyzed for mercury content within 30 days after the sludge sample is collected. Each determination shall be reported to the Administrator by a registered letter dispatched before the close of the next business day following such determination.

(g) Records of sludge sampling, charging rate determination and other data needed to determine mercury content of wastewater treatment plant sludges shall be retained at the source and made available, for inspection by the Administrator, for a minimum of 2 years.

§ 61.55 Emission monitoring.

(a) Wastewater treatment plant sludge incineration and drying plants. All such sources for which mercury emissions exceed 1600 g/day, demonstrated either by stack sampling according to § 61.53 or sludge sampling according to § 61.54, shall monitor mercury emissions at intervals of at least once per year by use of Method 105 of Appendix B, or the procedures specified in § 61.54(c) and (d). The results of monitoring shall be reported and retained according to § 61.53(d) (5) and (6), or § 61.54(f) and (g).

15. Appendix A is revised to a new reporting format, and sections (I) (C) and (I) (D) are added as follows:

APPENDIX A

National Emission Standards for Hazardous Air Pollutants

Compliance Status Information

1. SOURCE REPORT

INSTRUCTIONS: Owners or operators of sources of hazardous pollutants subject to the National Emission Standards for Hazardous Air Pollutants are required to submit the information contained in Section I to the appropriate U.S. Environmental Protection Agency Regional Office prior to 90 days after the effective date of any standards or amendments which require the submission of such information.

A list of regional offices is provided in 861.04.

A. SOURCE INFORMATION

1. Identification/Location - Indicate the name and address of each source.

1	2	3	4	5	8	9	13	0 0 0	0 0	1
Region	State	County	Source Number	17	16	17	18	19		
20	22	23	26	27	Source Name					46
AQCR #		City Code								
47 Street Address (Location of Plant) 66 80										
Dup 1-18 19										
20 City Name 34 State 35 39										
40 State Regis. Number 54 55 58 NEDS X Ref.										
59 SIC 62 FF 8 77 79 Staff 80										
64 65										
Dup 1-18 5 19 25 31 30 49 80										

2. Contact - Indicate the name and telephone number of the owner or operator or other responsible official whom EPA may contact concerning this report.

RULES AND REGULATIONS

Dup 1-18 4 1 19 20 21 Name 43

44 46
Area Code 47 Number 54 80

3. Source Description - Briefly state the nature of the source (e.g., "Chlor-alkali Plant" or "Machine Shop").

Dup 1-18 4 2 19 20 21 Description 60

61 Continued 79 80

4. Alternative Mailing Address - Indicate an alternative mailing address if correspondence is to be directed to a location different than that specified above.

Dup 1-18 4 3 19 20 21 Number Street or Box Number 45 80

Dup 1-18 4 4 19 20 21 City 35 State 37 38 41 Zip 44 80

5. Compliance Status - The emissions from this source can cannot meet the emission limitations contained in the National Emission Standards on or prior to 90 days after the effective date of any standards or amendments which require the submission of such information.

Signature of Owner, Operator or Other Responsible Official

NOTE: If the emissions from the source will exceed those limits set by the National Emission Standards for Hazardous Air Pollutants, the source will be in violation and subject to Federal enforcement actions unless granted a waiver of compliance by the Administrator of the U.S. Environmental Protection Agency. The information needed for such waivers is listed in Section II of this form.

- B. PROCESS INFORMATION. Part B should be completed separately for each point of emission for each hazardous pollutant. [Sources subject to 61.22(1) may omit number 4. below.]

Dup 1-13 0 0 5
14 16 17 18 19 20 SCC 27 28 29 30 31
NEDS X Ref LS SIP

RULES AND REGULATIONS

48305

1. **Pollutant Emitted** - Indicate the type of hazardous pollutant emitted by the process. Indicate "AB" for asbestos, "BE" for beryllium, or "Hg" for mercury.

32	33	Regulation		48	49
Pollutant	34				EC

2. **Process Description** - Provide a brief description of each process (e.g., "hydrogen end box" in a mercury chlor-alkali plant, "grinding machine" in a beryllium machine shop). Use additional sheets if necessary.

50	Process Description	74	80
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Dup 1-18	6 1	19	20	21	50
51				79	80
Dup 1-18	6 2	19	20	21	50
51				79	80

3. **Amount of Pollutant** - Indicate the average weight of the hazardous material named in Item 1 which enters the process in pounds per month (based on the previous twelve months of operation).

Dup 1-18	6 3	19	20	21	27	lbs./mo.	29	36	80
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4. **Control Devices**

- a. Indicate the type of pollution control devices, if any, used to reduce the emissions from the process (e.g., venturi scrubber, baghouse, wet cyclone) and the estimated percent of the pollutant which the device removes from the process gas stream.

Dup 1-18	6 4	19	20	21	PRIMARY CONTROL DEVICE:	43
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45	Primary Device Name	66	70	Percent Removal Efficiency	72	79
----	---------------------	----	----	----------------------------	----	----

80

RULES AND REGULATIONS

Dup 1-18	6 5	SECONDARY CONTROL DEVICES:		45
19	20	21		
47	Secondary Device Name		64	66
			Percent Removal Efficiency	70
			% EFFIC.	72
				79
				80

b. Asbestos Emission Control Devices Only

1. If a baghouse is specified in Item 4a, give the following information:

• The air flow permeability in cubic feet per minute per square foot of fabric area.

Air flow permeability = _____ cfm/ft²

• The pressure drop in inches water gauge across the filter at which the baghouse is operated.

Operating pressure drop = _____ inches w.g.

• If the baghouse material contains synthetic fill yarn, check whether this material is / / spun / / or not spun.

• If the baghouse utilizes a felted fabric, give the minimum thickness in inches and the density in ounces per square yard.

Thickness = _____ inches . Density = _____ oz/yd²

11. If a wet collection device is specified in Item 4a, give the designed unit contacting energy in inches water gauge.

• Unit contacting energy = _____ inches w.g.

C. DISPOSAL OF ASBESTOS-CONTAINING WASTES. Part C should be completed separately for each asbestos-containing waste generation operation arising from sources subject to §61.22(a), (c), (e), and (h).

Dup 1-13	14	16	17	18	19	20	SCC	27	28	29	30	31
			0 0		5							
									NEDS X Ref		CS	SIP
A B	32	33	34	Regulation			48	49				
Pollutant								EC				

RULES AND REGULATIONS

48307

1. Waste Generation - Provide a brief description of each process that generates asbestos-containing waste (e.g. disposal of control device wastes).

50 _____ Process Description _____ 79 80

2. Asbestos Concentration - Indicate the average percentage asbestos content of these materials.

Dup 1-18 6 1 _____ ASBESTOS CONCENTRATION: _____ 43 45 48
19 20 21

50 80

3. Amount of Wastes - Indicate the average weight of asbestos-containing wastes disposed of, measured in kg/day.

Dup 1-18 6 2 _____ kg/day _____ 34 80
19 20 21 27 29

4. Control Methods - Indicate the emission control methods used in all stages of waste disposal, from collection, processing, and packaging to transporting and deposition.

Dup 1-18 6 3 _____ Primary Control Method _____ 43
19 20 21

45 _____ 79 80

Dup 1-18 6 4 _____ 50
19 20 21

51 _____ 79 80

5. Waste Disposal - Indicate the type of disposal site (sanitary landfill, open, covered) or incineration site (municipal, private) where the waste is disposed of and who operates the site (company, private, municipal). State the name and location of the site (closest city or town, county, state).

Dup 1-18 6 5 _____ TYPE OF SITE: _____ 33 35 50
19 20 21

51 _____ 79 80

RULES AND REGULATIONS

Dup 1-18 6 6 OPERATOR: 21 29 31 50

51 79 80

Dup 1-18 6 7 LOCATION: 21 29

31 70

71 79 80

D. WASTE DISPOSAL SITES. Part D should be completed separately for each asbestos waste disposal site subject to section 61.22(1).

Dup 1-13 14 16 17 18 19 20 0 0 5 SCC 27 28 29 30 31
NEDS X Ref CS SIP

A B
32 33 34 Regulation 48 49
Pollutant EC

WASTE DISPOSAL SITE
50 68 80

1. Description - Provide a brief description of the site, including its size and configuration, and the distance to the closest city or town, closest residence, and closest primary road.

Dup 1-18 6 1 SITE DESCRIPTION 21 37 39 50

51 79 80

Dup 1-18 6 2 DISTANCE: TOWN: K M
19 20 21 29 30 34 36 40 42 43

RESIDENCE: K M ROAD: 45 54 56 60 62 63 65 69 71 75

K M
77 78 80

2. Inactivation - After the site is inactivated, indicate the method or methods used to comply with the standard and send a list of the actions that will be undertaken to maintain the inactivated site.

Dup 1-18 6 8 19 20 21 52

METHOD/INACTIVE SITE:

54 79 80

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 emissions 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.)

3. Increments of Progress - Specify the dates by which the following increments of progress will be met.

- 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.

Dup 1-16 0 1 7
17 19 53 54 55 60 61 MD/DY/YR 66 80

- Date of initiation of on-site construction or installation of emission control equipment or process change.

Dup 1-16 0 2 7
17 19 53 54 55 60 61 MD/DY/YR 66 80

- Date by which on-site construction or installation of emission control equipment or process modification is to be completed.

Dup 1-16 0 3 7
17 19 53 54 55 60 61 MD/DY/YR 66 80

- Date by which final compliance is to be achieved.

Dup 1-16 0 4 7
17 19 53 54 55 60 61 MD/DY/YR 66 80

- B. **WAIVER OF EMISSION TESTS.** A waiver of emission testing may be granted to owners or operators of sources of beryllium or mercury pollutants 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.

Date

Signature of the owner or operator

APPENDIX B—TEST METHODS

16. Method 105 is added to Appendix B as follows:

METHOD 105. METHOD FOR DETERMINATION OF MERCURY IN WASTEWATER TREATMENT PLANT SEWAGE SLUDGES

1. **Principle and applicability.** 1.1 Principle—A weighed portion of the sewage sludge sample is digested in aqua regia for 2 minutes at 95°C, followed by oxidation with potassium permanganate. Mercury in the digested sample is then measured by the conventional spectrophotometer cold vapor technique. An alternative digestion involving the use of an autoclave is described in paragraph 4.5.2 of this method.

1.2 Applicability—This method is applicable for the determination of total organic and inorganic mercury content in sewage sludges, soils, sediments, and bottom-type materials. The normal range of this method is 0.2 to 5 µg/g. The range may be extended above or below the normal range by increasing or decreasing sample size and through instrument and recorder control.

2. **Apparatus.** 2.1 Analysis—The conventional cold-vapor technique(5) is used to analyze the sample.

2.1.1 Atomic Absorption Spectrophotometer—Any atomic absorption unit having an open sample presentation area in which to mount the absorption cell is suitable. Instrument settings recommended by the particular manufacturer should be followed.

1 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.

2.1.2 Mercury Hollow Cathode Lamp—Westinghouse WL-22847, argon filled, or equivalent.

2.1.3 Recorder—Any multirange, variable-speed recorder that is compatible with the UV detection system is suitable.

2.1.4 Absorption Cell—Standard spectrophotometer cells 10 cm long, having quartz end windows may be used. Suitable cells may be constructed from plexiglass tubing, 2.5 cm O.D. x 11.4 cm (ca. 1" O.D. x 4 1/4"). The ends are ground perpendicular to the longitudinal axis, and quartz windows [2.5 cm diameter x 0.16 cm thickness (ca. 1" diameter x 3/16" thickness)] are cemented in place. Gas inlet and outlet ports [also of plexiglass but 0.6 cm O.D. (ca. 1/4" O.D.)] are attached approximately 1.3 cm (1/2") from each end. The cell is strapped to a burner for support and aligned in the light beam to give the maximum transmittance. NOTE: Two 5.1 cm x 5.1 cm (ca. 2" x 2") cards with 2.5 cm (ca. 1") diameter holes may be placed over each end of the cell to assist in positioning the cell for maximum transmittance.

2.1.5 Air Pump—Any peristaltic pump capable of delivering 1 liter of air per minute may be used. A Masterflex pump with electronic speed control has been found to be satisfactory. (Regulated compressed air can be used in an open one-pass system.)

2.1.6 Flowmeter—Capable of measuring an air flow of 1 liter per minute.

2.1.7 Aeration Tubing—Tygon tubing is used for passage of the mercury vapor from the sample bottle to the absorption cell and return. Straight glass tubing terminating in a coarse porous frit is used for sparging air into the sample.

2.1.8 Drying Tube—15 cm long x 1.9 cm diameter (ca. 6" long x 3/4" diameter) tube containing 20 grams of the desiccant magnesium perchlorate. The apparatus is assem-

bled as shown in Figure 105-1. In place of the magnesium perchlorate drying tube, a small reading lamp with 60W bulb may be used to prevent condensation of moisture inside the cell. The lamp is positioned so as not to interfere with the measurement and to shine on the absorption cell maintaining the air temperature about 5°C above ambient.

3. Reagents. 3.1 Analysis.

3.1.1 Aqua Regia—Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO₃.

3.1.2 Sulfuric Acid, 0.5N—Dilute 14.0 ml of concentrated sulfuric acid to 1.0 liter.

3.1.3 Stannous Sulfate—Add 25 g stannous sulfate to 250 ml of 0.5N sulfuric acid. This mixture is a suspension and should be stirred continuously during use. Stannous chloride may be used in place of the stannous sulfate.

3.1.4 Sodium Chloride—Hydroxylamine Sulfate Solution—Dissolve 12 grams of sodium chloride and 12 grams of hydroxylamine sulfate in distilled water and dilute to 100 ml. Hydroxylamine hydrochloride may be used in place of the hydroxylamine sulfate.

3.1.5 Potassium Permanganate—5% solution, w/v. Dissolve 5 grams of potassium permanganate in 100 ml of distilled water.

3.1.6 Stock Mercury Solution—Dissolve 0.1354 grams of reagent grade mercuric chloride (Assay >95%) in 75 ml of distilled water. Add 10 ml of concentrated nitric acid and adjust the volume to 100.0 ml. 1 ml = 1 mg Hg.

3.1.7 Working Mercury Solution—Make successive dilutions of the stock mercury solution to obtain a working standard containing 0.1 µg per ml. This working standard and the dilutions of the stock mercury solution should be prepared fresh daily. Acidity of the working standard should be maintained at 0.15% nitric acid. This acid should be added to the flask as needed before the addition of the aliquot. Mercuric solutions should not be prepared in plastic containers.

4. **Procedures.** Samples for mercury analysis are subject to contamination from a variety of sources. Extreme care must be taken to prevent contamination. Certain interferences may occur during the analysis procedures. Extreme caution must be taken to avoid inhalation of mercury.

4.1 Sample Handling and Preservation.

4.1.1 Because of the extreme sensitivity of the analytical procedure and the omnipresence of mercury, care must be taken to avoid extraneous contamination. Sampling devices, sample containers, and reagents should be ascertained to be free of significant amounts of mercury; the sample should not be exposed to any condition in the laboratory that may result in contact or airborne mercury contamination. Sample containers to be used for collection and shipment of mercury samples should be properly cleaned before use. These should be rinsed with at least 20% v/v HNO₃ followed by distilled water.

4.1.2 While the sample may be analyzed without drying, it has been found to be more convenient to analyze a dry sample. Moisture may be driven off in a drying oven at a temperature of 60°C. No significant mercury losses have been observed by using this drying step. The dry sample should be pulverized and thoroughly mixed before the aliquot is weighed.

4.2 Interferences.

4.2.1 Interferences that may occur in sludge samples are sulfides, high copper, high chlorides, etc. A discussion of possible interferences and suggested preventative measures to be taken is given in Reference (6) (7).

4.2.2 Volatile materials which absorb at the 253.7 nm will cause a positive interfer-

