For the reasons set out in the preamble, title 40, Chapter I of the Code of Federal Regulations is amended as follows:

PART 63--NATIONAL EMISSION STANDARDS FOR HAZARDOUS AIR POLLUTANTS FOR SOURCE CATEGORIES

1. The authority citation for part 63 continues to read as follows:

Authority: 42 U.S.C. 7401, et seq.

2. Part 63 is amended by adding subpart S to read as follows:

Subpart S--National Emission Standards for Hazardous Air Pollutants from the Pulp and Paper Industry

Sec.

Subpart S--National Emission Standards for Hazardous Air Pollutants from the Pulp and Paper Industry

§ 63.440 Applicability.

(a) The provisions of this subpart apply to the owner or operator of processes that produce pulp, paper, or paperboard; that are located at a plant site that is a major source as defined in § 63.2 of subpart A of this part; and that use the following processes and materials:

(1) Kraft, soda, sulfite, or semi-chemical pulping processes using wood; or

(2) Mechanical pulping processes using wood; or

(3) Any process using secondary or non-wood fibers.

(b) The affected source to which the existing source provisions of this subpart apply is as follows:

(1) For the processes specified in paragraph (a)(1) of this section, the affected source is the total of all HAP emission points in the pulping and bleaching systems; or

(2) For the processes specified in paragraphs (a)(2) or (a)(3) of this section, the affected source is the total of all HAP emission points in the bleaching system.

(c) The new source provisions of this subpart apply to the total of all HAP emission points at new or existing sources as follows:

(1) Each affected source defined in paragraph (b)(1) of this section that commences construction or reconstruction after December 17, 1993;

(2) Each pulping system or bleaching system for the processes specified in paragraph (a)(1) of this section that commences construction or reconstruction after December 17, 1993;

(3) Each additional pulping or bleaching line at the processes specified in paragraph (a)(1) of this section, that commences construction after December 17, 1993;

(4) Each affected source defined in paragraph (b)(2) of this section that commences construction or reconstruction after March 8, 1996; or

(5) Each additional bleaching line at the processes specified in paragraphs (a)(2) or (a)(3) of this section, that commences construction after March 8, 1996.

(d) Each existing source shall achieve compliance no later than [insert date 3 years after date published in the FEDERAL REGISTER], except as provided in paragraphs (d)(1) through (d)(3) of this section.

(1) Each kraft pulping system shall achieve compliance with the pulping system provisions of § 63.443 for the equipment listed in § 63.443(a)(1)(ii) through (a)(1)(v) as expeditiously as practicable, but in no event later than [insert date 8 years after date published in the FEDERAL REGISTER] and the owners and operators shall establish dates, update dates, and report the dates for the milestones specified in § 63.455(b).

(2) Each dissolving-grade bleaching system at either kraft or sulfite pulping mills shall achieve compliance with the bleach plant provisions of § 63.445 of this subpart as expeditiously as practicable, but in no event later than 3 years after the promulgation of the revised effluent limitation guidelines and standards under 40 CFR 430.14 through 430.17 and 40 CFR 430.44 through 430.47.

(3) Each bleaching system complying with the Voluntary Advanced Technology Incentives Program for Effluent

Limitation Guidelines in 40 CFR 430.24, shall comply with the requirements specified in either paragraph (d)(3)(i) or (d)(3)(ii) of this section for the effluent limitation guidelines and standards in 40 CFR 430.24.

(i) Comply with the bleach plant provisions of § 63.445 of this subpart as expeditiously as practicable, but in no event later than <u>[insert date 3 years after date published in the FEDERAL REGISTER</u>].

(ii) Comply with all of the following:

(A) The owner or operator of a bleaching system shall comply with the bleach plant provisions of § 63.445 of this subpart as expeditiously as practicable, but in no event later than <u>[insert date 6 years after date published in the FEDERAL REGISTER</u>].

(B) The owner or operator of a bleaching system shall not increase the application rate of chlorine or hypochlorite in kg of bleaching agent per megagram of ODP, in the bleaching system above the average daily rates used over the three months prior to [<u>insert date 60 days after publication in the FEDERAL REGISTER</u>] until the requirements of paragraph (d)(3)(ii)(A) of this section are met and record application rates as specified in § 63.454(c).

(C) Owners and operators shall establish dates, update dates, and report the dates for the milestones specified in § 63.455(b).

(e) Each new source, specified as the total of all HAP emission points for the sources specified in paragraph (c) of this section, shall achieve compliance upon start-up or [insert date 60 days after published in the FEDERAL REGISTER], whichever is later, as provided in §63.6(b) of subpart A of this part.

(f) Each owner or operator of an affected source with affected process equipment shared by more than one type of pulping process, shall comply with the applicable requirement in this subpart that achieves the maximum degree of reduction in HAP emissions.

(g) Each owner or operator of an affected source specified in paragraphs (a) through (c) of this section must comply with the requirements of subpart A - General Provisions of this part, as indicated in table 1.

§ 63.441 Definitions.

All terms used in this subpart shall have the meaning given them in the CAA, in subpart A of this part, and in this section as follows:

Acid condensate storage tank means any storage tank containing cooking acid following the sulfur dioxide gas fortification process.

Black liquor means spent cooking liquor that has been separated from the pulp produced by the kraft, soda, or semi-chemical pulping process.

Bleaching means brightening of pulp by the addition of oxidizing chemicals or reducing chemicals.

Bleaching line means a group of bleaching stages arranged in series such that bleaching of the pulp progresses as the pulp moves from one stage to the next.

Bleaching stage means all process equipment associated with a discrete step of chemical application and removal in the bleaching process including chemical and steam mixers, bleaching towers, washers, seal (filtrate) tanks, vacuum pumps, and any other equipment serving the same function as those previously listed.

Bleaching system means all process equipment after high-density pulp storage prior to the first application of oxidizing chemicals or reducing chemicals following the pulping system, up to and including the final bleaching stage.

<u>Boiler</u> means any enclosed combustion device that extracts useful energy in the form of steam. A boiler is not considered a thermal oxidizer.

<u>Chip steamer</u> means a vessel used for the purpose of preheating or pretreating wood chips prior to the digester, using flash steam from the digester or live steam.

<u>Closed-vent system</u> 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 point to a control device.

<u>Combustion device</u> means an individual unit of equipment, including but not limited to, a thermal oxidizer, lime kiln, recovery furnace, process heater, or boiler, used for the thermal oxidation of organic hazardous air pollutant vapors.

<u>Decker system</u> means all equipment used to thicken the pulp slurry or reduce its liquid content after the pulp washing system and prior to high-density pulp storage. The decker system includes decker vents, filtrate tanks, associated vacuum pumps, and any other equipment serving the same function as those previously listed.

<u>Digester system</u> means each continuous digester or each batch digester used for the chemical treatment of wood or

non-wood fibers. The digester system equipment includes associated flash tank(s), blow tank(s), chip steamer(s) not using fresh steam, blow heat recovery accumulator(s), relief gas condenser(s), prehydrolysis unit(s) preceding the pulp washing system, and any other equipment serving the same function as those previously listed. The digester system includes any of the liquid streams or condensates associated with batch or continuous digester relief, blow, or flash steam processes.

<u>Emission point</u> means any part of a stationary source that emits hazardous air pollutants regulated under this subpart, including emissions from individual process vents, stacks, open pieces of process equipment, equipment leaks, wastewater and condensate collection and treatment system units, and those emissions that could reasonably be conveyed through a stack, chimney, or duct where such emissions first reach the environment.

<u>Evaporator system</u> means all equipment associated with increasing the solids content and/or concentrating spent cooking liquor from the pulp washing system including pre-evaporators, multi-effect evaporators, concentrators, and vacuum systems, as well as associated condensers, hotwells, and condensate streams, and any other equipment serving the same function as those previously listed.

Flow indicator means any device that indicates gas or liquid flow in an enclosed system.

HAP means a hazardous air pollutant as defined in § 63.2 of subpart A of this part.

High volume, low concentration or HVLC collection system means the gas collection and transport system used to convey gases from the HVLC system to a control device.

High volume, low concentration or HVLC system means the collection of equipment including the pulp washing, knotter, screen, decker, and oxygen delignification systems, weak liquor storage tanks, and any other equipment serving the same function as those previously listed.

Knotter system means equipment where knots, oversized material, or pieces of uncooked wood are removed from the pulp slurry after the digester system and prior to the pulp washing system. The knotter system equipment includes the knotter, knot drainer tanks, ancillary tanks, and any other equipment serving the same function as those previously listed.

Kraft pulping means a chemical pulping process that uses a mixture of sodium hydroxide and sodium sulfide as the cooking liquor.

Lime kiln means an enclosed combustion device used to calcine lime mud, which consists primarily of calcium carbonate, into calcium oxide.

Low volume, high concentration or LVHC collection system means the gas collection and transport system used to convey gases from the LVHC system to a control device.

Low volume, high concentration or LVHC system means the collection of equipment including the digester, turpentine recovery, evaporator, steam stripper systems, and any other equipment serving the same function as those previously listed.

Mechanical pulping means a pulping process that only uses mechanical and thermo-mechanical processes to reduce wood to a fibrous mass. The mechanical pulping processes include, but are not limited to, stone groundwood, pressurized groundwood, refiner mechanical, thermal refiner mechanical, thermo-mechanical, and tandem thermo-mechanical.

Non-wood pulping means the production of pulp from fiber sources other than trees. The non-wood fiber sources include, but are not limited to, bagasse, cereal straw, cotton, flax straw, hemp, jute, kenaf, and leaf fibers.

Oven-dried pulp or ODP means a pulp sample at zero percent moisture content by weight. Pulp samples for applicability or compliance determinations for both the pulping and bleaching systems shall be unbleached pulp. For purposes of complying with mass emission limits in this subpart, megagram of ODP shall be measured to represent the amount of pulp entering and processed by the equipment system under the specified mass limit. For equipment that does not process pulp, megagram of ODP shall be measured to represent the amount of pulp that was processed to produce the gas and liquid streams.

Oxygen delignification system means the equipment that uses oxygen to remove lignin from pulp after high-density stock storage and prior to the bleaching system. The oxygen delignification system equipment includes the blow tank, washers, filtrate tanks, any interstage pulp storage tanks, and any other equipment serving the same function as those previously listed.

Primary fuel means the fuel that provides the principal heat input to the combustion device. To be considered primary, the fuel must be able to sustain operation of the combustion device without the addition of other fuels.

Process wastewater treatment system means a collection of equipment, a process, or specific technique that removes or destroys the HAP's in a process wastewater stream. Examples include, but are not limited to, a steam stripping unit, wastewater thermal oxidizer, or biological treatment unit.

Pulp washing system means all equipment used to wash pulp and separate spent cooking chemicals following the digester system and prior to the bleaching system, oxygen delignification system, or paper machine system (at unbleached mills). The pulp washing system equipment includes vacuum drum washers, diffusion washers, rotary pressure washers, horizontal belt filters, intermediate stock chests, and their associated vacuum pumps, filtrate tanks, foam breakers or tanks, and any other equipment serving the same function as those previously listed. The pulp washing system does not include deckers, screens, knotters, stock chests, or pulp storage tanks following the last stage of pulp washing.

Pulping line means a group of equipment arranged in series such that the wood chips are digested and the resulting pulp progresses through a sequence of steps that may include knotting, refining, washing, thickening, blending, storing, oxygen delignification, and any other equipment serving the same function as those previously listed.

Pulping process condensates means any HAP-containing liquid that results from contact of water with organic compounds in the pulping process. Examples of process condensates include digester system condensates, turpentine recovery system condensates, evaporator system condensates, LVHC system condensates, HVLC system condensates, and any other condensates from equipment serving the same function as those previously listed. Liquid streams that are intended for byproduct recovery are not considered process condensate streams.

Pulping system means all process equipment, beginning with the digester system, and up to and including the last piece of pulp conditioning equipment prior to the bleaching system, including treatment with ozone, oxygen, or peroxide before the first application of a chemical bleaching agent intended to brighten pulp. The pulping system includes pulping process condensates and can include multiple pulping lines.

Recovery furnace means an enclosed combustion device where concentrated spent liquor is burned to recover sodium and sulfur, produce steam, and dispose of unwanted dissolved wood components in the liquor.

Screen system means equipment in which oversized particles are removed from the pulp slurry prior to the bleaching or papermaking system washed stock storage.

<u>Secondary fiber pulping</u> means a pulping process that converts a fibrous material, that has previously undergone a manufacturing process, into pulp stock through the addition of water and mechanical energy. The mill then uses that pulp as the raw material in another manufactured product. These mills may also utilize chemical, heat, and mechanical processes to remove ink particles from the fiber stock.

<u>Semi-chemical pulping</u> means a pulping process that combines both chemical and mechanical pulping processes. The semi-chemical pulping process produces intermediate yields ranging from 55 to 90 percent.

<u>Soda pulping</u> means a chemical pulping process that uses sodium hydroxide as the active chemical in the cooking liquor.

<u>Spent liquor</u> means process liquid generated from the separation of cooking liquor from pulp by the pulp washing system containing dissolved organic wood materials and residual cooking compounds.

<u>Steam stripper system</u> means a column (including associated stripper feed tanks, condensers, or heat exchangers) used to remove compounds from wastewater or condensates using steam. The steam stripper system also contains all equipment associated with a methanol rectification process including rectifiers, condensers,

decanters, storage tanks, and any other equipment serving the same function as those previously listed.

Strong liquor storage tanks means all storage tanks containing liquor that has been concentrated in preparation for combustion or oxidation in the recovery process.

Sulfite pulping means a chemical pulping process that uses a mixture of sulfurous acid and bisulfite ion as the cooking liquor.

Temperature monitoring device means a piece of equipment used to monitor temperature and having an accuracy of ±1.0 percent of the temperature being monitored expressed in degrees Celsius or ±0.5 degrees Celsius (^{O}C), whichever is greater.

Thermal oxidizer means an enclosed device that destroys organic compounds by thermal oxidation.

Turpentine recovery system means all equipment associated with recovering turpentine from digester system gases including condensers, decanters, storage tanks, and any other equipment serving the same function as those previously listed. The turpentine recovery system includes any liquid streams associated with the turpentine recovery process such as turpentine decanter underflow. Liquid streams that are intended for byproduct recovery are not considered turpentine recovery system condensate streams.

Weak liquor storage tank means any storage tank except washer filtrate tanks containing spent liquor recovered from the pulping process and prior to the evaporator system.

§ 63.442 [Reserved]

§ 63.443 Standards for the pulping system at kraft, soda, and semi-chemical processes.

(a) The owner or operator of each pulping system using the kraft process subject to the requirements of this subpart shall control the total HAP emissions from the following equipment systems, as specified in paragraphs (c) and (d) of this section.

(1) At existing affected sources, the total HAP emissions from the following equipment systems shall be controlled:

(i) Each LVHC system;

(ii) Each knotter or screen system with total HAP mass emission rates greater than or equal to the rates specified in paragraphs (a)(1)(ii)(A) or (ii)(B) or the combined rate specified in paragraph (a)(1)(ii)(C) of this section.

(A) Each knotter system with emissions of 0.05 kilograms or more of total HAP per megagram of ODP (0.1 pounds per ton).

(B) Each screen system with emissions of 0.10 kilograms or more of total HAP per megagram of ODP (0.2 pounds per ton).

(C) Each knotter and screen system with emissions of 0.15 kilograms or more of total HAP per megagram of ODP (0.3 pounds per ton).

(iii) Each pulp washing system;

(iv) Each decker system that:

(A) Uses any process water other than fresh water or paper machine white water; or

(B) Uses any process water with a total HAP concentration greater than 400 parts per million by weight; and

(v) Each oxygen delignification system.

(2) At new affected sources, the total HAP emissions from the equipment systems listed in paragraphs (a)(1)(i), (a)(1)(iii), and (a)(1)(v) of this section and the following equipment systems shall be controlled:

(i) Each knotter system;

(ii) Each screen system;

(iii) Each decker system; and

(iv) Each weak liquor storage tank.

(b) The owner or operator of each pulping system using a semi-chemical or soda process subject to the requirements of this subpart shall control the total HAP emissions from the following equipment systems as specified in paragraphs (c) and (d) of this section.

(1) At each existing affected sources, the total HAP emissions from each LVHC system shall be controlled.

(2) At each new affected source, the total HAP emissions from each LVHC system and each pulp washing system shall be controlled.

(c) Equipment systems listed in paragraphs (a) and (b) of this section shall be enclosed and vented into a closed-vent system and routed to a control device that meets the requirements specified in paragraph (d) of this section. The enclosures and closed-vent system shall meet the requirements specified in § 63.450.

(d) The control device used to reduce total HAP emissions from each equipment system listed in paragraphs (a) and (b) of this section shall:

(1) Reduce total HAP emissions by 98 percent or more by weight; or

(2) Reduce the total HAP concentration at the outlet of the thermal oxidizer to 20 parts per million or less by volume, corrected to 10 percent oxygen on a dry basis; or

(3) Reduce total HAP emissions using a thermal oxidizer designed and operated at a minimum temperature of 871 ^{o}C (1600 ^{o}F) and a minimum residence time of 0.75 seconds; or

(4) Reduce total HAP emissions using a boiler, lime kiln, or recovery furnace by introducing the HAP emission stream with the primary fuel or into the flame zone.

(e) Periods of excess emissions reported under § 63.455 shall not be a violation of § 63.443(c) and (d) provided that the time of excess emissions (excluding periods of startup, shutdown, or malfunction) divided by the total process operating time in a semi-annual reporting period does not exceed the following levels:

(1) One percent for control devices used to reduce the total HAP emissions from the LVHC system; and

(2) Four percent for control devices used to reduce the total HAP emissions from the HVLC system; and

(2) Four percent for control devices used to reduce the total HAP emissions from both the LVHC and HVLC systems.

§ 63.444 Standards for the pulping system at sulfite processes.

(a) The owner or operator of each sulfite process subject to the requirements of this subpart shall control the total HAP emissions from the following equipment systems as specified in paragraphs (b) and (c) of this section.

(1) At existing sulfite affected sources, the total HAP emissions from the following equipment systems shall be controlled:

(i) Each digester system vent;

(ii) Each evaporator system vent; and

(iii) Each pulp washing system.

(2) At new affected sources, the total HAP emissions from the equipment systems listed in paragraph (a)(1) of this section and the following equipment shall be controlled:

(i) Each weak liquor storage tank;

(ii) Each strong liquor storage tank; and

(iii) Each acid condensate storage tank.

(b) Equipment listed in paragraph (a) of this section shall be enclosed and vented into a closed-vent system and routed to a control device that meets the requirements specified in paragraph (c) of this section. The enclosures and closed-vent system shall meet the requirements specified in § 63.450. Emissions from equipment listed in paragraph (a) of this section that is not necessary to be reduced to meet paragraph (c) of this section is not required to be routed to a control device.

(c) The total HAP emissions from both the equipment systems listed in paragraph (a) of this section and the vents, wastewater, and condensate streams from the control device used to reduce HAP emissions, shall be controlled as follows.

(1) Each calcium-based or sodium-based sulfite pulping process shall:

(i) Emit no more than 0.44 kilograms of total HAP or methanol per megagram (0.89 pounds per ton) of ODP; or

(ii) Remove 92 percent or more by weight of the total HAP or methanol.

(2) Each magnesium-based or ammonium-based sulfite pulping process shall:

(i) Emit no more than 1.1 kilograms of total HAP or methanol per megagram (2.2 pounds per ton) of ODP; or

(ii) Remove 87 percent or more by weight of the total HAP or methanol.

§ 63.445 Standards for the bleaching system.

(a) Each bleaching system that does not use any chlorine or chlorinated compounds for bleaching is exempt from the requirements of this section. Owners or operators of the following bleaching systems shall meet all the provisions of this section:

(1) Bleaching systems that use chlorine;

(2) Bleaching systems bleaching pulp from kraft, sulfite, or soda pulping processes that uses any chlorinated compounds; or

(3) Bleaching systems bleaching pulp from mechanical pulping processes using wood or from any process using secondary or non-wood fibers, that use chlorine dioxide.

(b) The equipment at each bleaching stage, of the bleaching systems listed in paragraph (a) of this section, where chlorinated compounds are introduced shall be enclosed and vented into a closed-vent system and routed to a control device that meets the requirements specified in paragraph (c) of this section. The enclosures and closed-vent system shall meet the requirements specified in § 63.450.

(c) The control device used to reduce chlorinated HAP emissions (not including chloroform) from the equipment specified in paragraph (b) of this section shall:

(1) Reduce the total chlorinated HAP mass in the vent stream entering the control device by 99 percent or more by weight;

(2) Achieve a treatment device outlet concentration of 10 parts per million or less by volume of total chlorinated HAP; or

(3) Achieve a treatment device outlet mass emission rate of 0.001 kg of total chlorinated HAP mass per megagram (0.002 pounds per ton) of ODP.

(d) The owner or operator of each bleaching system subject to paragraph (a)(2) of this section shall comply with paragraph (d)(1) or (d)(2) of this section to reduce chloroform air emissions to the atmosphere, except the owner or operator of each bleaching system complying with extended compliance under § 63.440(d)(3)(ii) shall comply with paragraph (d)(1) of this section.

(1) Comply with the following applicable effluent limitation guidelines and standards specified in 40 CFR part 430:

(i) Dissolving-grade kraft bleaching systems and lines §§ 430.14 through 430.17;

(ii) Paper-grade kraft and soda bleaching systems and lines, §§ 430.24(a)(1) and (e), and §§ 430.26 (a) and (c);

(iii) Dissolving-grade sulfite bleaching systems and lines, §§ 430.44 through 430.47; or

(iv) Paper-grade sulfite bleaching systems and lines, §§ 430.54 (a) and (c), and 430.56 (a) and (c).

(2) Use no hypochlorite or chlorine for bleaching in the bleaching system or line.

§ 63.446 Standards for kraft pulping process condensates.

(a) The requirements of this section apply to owners or operators of kraft processes subject to the requirements of this subpart.

(b) The pulping process condensates from the following equipment systems shall be treated to meet the requirements specified in paragraphs (c), (d), and (e) of this section:

(1) Each digester system;

(2) Each turpentine recovery system;

(3) Each evaporator stage where weak liquor is introduced (feed stages) in the evaporator system;

(4) Each HVLC collection system; and

(5) Each LVHC collection system.

(c) One of the following combinations of HAP-containing pulping process condensates generated, produced, or associated with the equipment systems listed in paragraph (b) of this section shall be subject to the requirements of paragraphs (d) and (e) of this section:

(1) All pulping process condensates from the equipment systems specified in paragraphs (b)(1) through (b)(5) of this section.

(2) The combined pulping process condensates from the equipment systems specified in paragraphs (b)(4) and (b)(5) of this section, plus pulping process condensate stream(s)

that in total contain at least 65 percent of the total HAP mass from the pulping process condensates from equipment systems listed in paragraphs (b)(1) through (b)(3) of this section.

(3) The pulping process condensates from equipment systems listed in paragraphs (b)(1) through (b)(5) of this section that in total contain a total HAP mass of 3.6 kilograms or more of total HAP per megagram (7.2 pounds per ton) of ODP for mills that do not perform bleaching or 5.5 kilograms or more of total HAP per megagram (11.1 pounds per ton) of ODP for mills that perform bleaching.

(d) The pulping process condensates from the equipment systems listed in paragraph (b) of this section shall be conveyed in a closed collection system that is designed and operated to meet the requirements specified in paragraphs (d)(1) and (d)(2) of this section.

(1) Each closed collection system shall meet the individual drain system requirements specified in § 63.960, 63.961, and 63.962 of subpart RR of this part, except for closed vent systems and control devices shall be designed and operated in accordance with § 63.443(d) and 63.450, instead of in accordance with § 63.693 as specified in § 63.962 (a)(3)(ii), (b)(3)(ii)(A), and (b)(3)(ii)(B)(5)(iii); and

(2) If a condensate tank is used in the closed collection system, the tank shall meet the following requirements:

(i) The fixed roof and all openings (e.g., access hatches, sampling ports, gauge wells) shall be designed and operated with no detectable leaks as indicated by an instrument reading of less than 500 parts per million above background, and vented into a closed-vent system that meets the requirements in § 63.450 and routed to a control device that meets the requirements in § 63.443(d); and

(ii) 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 the tank contains pulping process condensates or any HAP removed from a pulping process condensate stream except when it is necessary to use the opening for sampling, removal, or for equipment inspection, maintenance, or repair.

(e) Each pulping process condensate from the equipment systems listed in paragraph (b) of this section shall be treated according to one of the following options:

(1) Recycle the pulping process condensate to an equipment system specified in § 63.443(a) meeting the requirements specified in § 63.443(c) and (d); or

(2) Discharge the pulping process condensate below the liquid surface of a biological treatment system meeting the requirement specified in paragraph (e)(3) of this section; or

(3) Treat the pulping process condensates to reduce or destroy the total HAP's by at least 92 percent or more by weight; or

(4) At mills that do not perform bleaching, treat the pulping process condensates to remove 3.3 kilograms or more of total HAP per megagram (6.6 pounds per ton) of ODP, or achieve a total HAP concentration of 210 parts per million or less by weight at the outlet of the control device; or

(5) At mills that perform bleaching, treat the pulping process condensates to remove 5.1 kilograms or more of total HAP per megagram (10.2 pounds per ton) of ODP, or achieve a total HAP concentration of 330 parts per million or less by weight at the outlet of the control device.

(f) Each HAP removed from a pulping process condensate stream during treatment and handling under paragraphs (d) or (e) of this section, except for those treated according to paragraph (e)(2) of this section, shall be controlled as specified in § 63.443(c) and (d).

(g) For each steam stripper system used to comply with the requirements specified in paragraph (e)(3) of this section, periods of excess emissions reported under § 63.455 shall not be a violation paragraphs (d), (e), and (f) of this section provided that the time of excess emissions (including periods of startup, shutdown, or malfunction) divided by the total process operating time in a semi-annual reporting period does not exceed 10 percent.

(h) Each owner or operator of a new or existing affected source subject to the requirements of this section shall evaluate all new or modified pulping process condensates or changes in the annual bleached or non-bleached ODP used to comply with paragraph (i) of this section, to determine if they meet the applicable requirements of this section.

(i) For the purposes of meeting the requirements in paragraphs (c)(2), (e)(4), or (e)(5) of this section at mills producing both bleached and unbleached pulp products, owners and operators may meet a prorated mass standard that is calculated by prorating the applicable mass standards (kilograms of total HAP per megagram of ODP) for bleached and unbleached specified in paragraphs (c)(2), (e)(4), or (e)(5) of this section by the ratio of annual megagrams of bleached and unbleached ODP.

§ 63.447 Clean condensate alternative.

As an alternative to the requirements specified in § 63.443(a)(1)(ii) through (a)(1)(v) for the control of HAP emissions from pulping systems using the kraft process, an owner or operator must demonstrate to the satisfaction of the Administrator, by meeting all the requirements below, that the total HAP emissions reductions achieved by this clean condensate alternative technology are equal to or greater than the total HAP emission reductions that would have been achieved by compliance with § 63.443(a)(1)(ii) through (a)(1)(v).

(a) For the purposes of this section only the following additional definitions apply.

(1) Clean condensate alternative affected source means the total of all HAP emission points in the pulping, bleaching, causticizing, and papermaking systems (exclusive of HAP emissions attributable to additives to paper machines and HAP emission points in the LVHC system).

(2) Causticizing system means all equipment associated with converting sodium carbonate into active sodium hydroxide. The equipment includes smelt dissolving tanks, lime mud washers and storage tanks, white and mud liquor clarifiers and storage tanks, slakers, slaker grit washers, lime kilns, green liquor clarifiers and storage tanks, and dreg washers ending with the white liquor storage tanks

prior to the digester system, and any other equipment serving the same function as those previously listed.

(3) Papermaking system means all equipment used to convert pulp into paper, paperboard, or market pulp, including the stock storage and preparation systems, the paper or paperboard machines, and the paper machine white water system, broke recovery systems, and the systems involved in calendering, drying, on-machine coating, slitting, winding, and cutting.

(b) Each owner or operator shall install and operate a clean condensate alternative technology with a continuous monitoring system to reduce total HAP emissions by treating and reducing HAP concentrations in the pulping process water used within the clean condensate alternative affected source.

(c) Each owner or operator shall calculate HAP emissions on a kilogram per megagram of ODP basis and measure HAP emissions according to the appropriate procedures contained in § 63.457.

(d) Each owner or operator shall determine the baseline HAP emissions for each equipment system and the total of all equipment systems in the clean condensate alternative affected source based on the following:

(1) Process and air pollution control equipment installed and operating on or after December 17, 1993, and

(2) Compliance with the following requirements that affect the level of HAP emissions from the clean condensate alternative affected source:

(i) The pulping process condensates requirements in § 63.446;

(ii) The applicable effluent limitation guidelines and standards in 40 CFR part 430, subparts A, B, D, and E; and

(iii) All other applicable requirements of local, State, or Federal agencies or statutes.

(e) Each owner or operator shall determine the following HAP emission reductions from the baseline HAP emissions determined in paragraph (d) of this section for each equipment system and the total of all equipment systems in the clean condensate alternative affected source:

(1) The HAP emission reduction occurring by complying with the requirements of § 63.443(a)(1)(ii) through (a)(1)(v), and

(2) The HAP emissions reduction that occurring by complying with the clean condensate alternative technology.

(f) For the purposes of all requirements in this section, each owner or operator may use as an alternative, individual equipment systems (instead of total of all equipment systems) within the clean condensate alternative affected source to determine emissions and reductions to demonstrate equal or greater than the reductions that would have been achieved by compliance with § 63.443(a)(1)(ii) through (a)(1)(v).

(g) The initial and updates to the control strategy report specified in § 63.455(b) shall include to the extent possible the following information:

(1) A detailed description of:

(i) The equipment systems and emission points that comprise the clean condensate alternative affected source;

(ii) The air pollution control technologies that would be used to meet the requirements of § 63.443(a)(1)(ii) through (a)(1)(v);

(iii) The clean condensate alternative technology to be used.

(2) Estimates and basis for the estimates of total HAP emissions and emissions reductions to fulfill the requirements paragraphs (d), (e), and (f) of this section.

(h) Each owner or operator shall report to the Administrator by the applicable compliance date specified in § 63.440(d) or (e) the rationale, calculations, test procedures, and data documentation used to demonstrate compliance with all the requirements of this section.

§63.448-449 [Reserved]

§63.450 Standards for enclosures and closed-vent systems.

(a) Each enclosure and closed-vent system specified in §§ 63.443(c), 63.444(b), and 63.445(b) for capturing and transporting vent streams that contain HAP shall meet the requirements specified in paragraphs (b) through (d) of this section.

(b) Each enclosure shall maintain negative pressure at each enclosure or hood opening as demonstrated by the procedures specified § 63.457(e). Each enclosure or hood opening closed during the initial performance test specified in § 63.457(a) shall be maintained in the same closed and sealed position as during the performance test at all times except when necessary to use the opening for sampling, inspection, maintenance, or repairs.

(c) Each component of the closed-vent system used to comply with § 63.443(c), § 63.444(b), and § 63.445(b) that is operated at positive pressure and located prior to a control device shall be designed for and operated with no detectable leaks as indicated by an instrument reading of less than 500 parts per million by volume above background, as measured by the procedures specified in § 63.457(d).

(d) Each bypass line in the closed-vent system that could divert vent streams containing HAP to the atmosphere without meeting the emission limitations in §§ 63.443, 63.444, or 63.445 shall comply with either of the following requirements:

(1) On each bypass line, the owner or operator shall install, calibrate, maintain, and operate according to manufacturer's specifications a flow indicator that provides a record of the presence of gas stream flow in the bypass line at least once every 15 minutes. The flow indicator shall be installed in the bypass line in such a way as to indicate flow in the bypass line; or

(2) For bypass line valves that are not computer controlled, the owner or operator shall maintain the bypass line valve in the closed position with a car seal or a seal placed on the valve or closure mechanism in such a way that valve or closure mechanism cannot be opened without breaking the seal.

§ 63.451-452 [Reserved]

§ 63.453 Monitoring Requirements.

(a) Each owner or operator subject to the standards specified in §§ 63.443(c) and (d), 63.444(b) and (c), 63.445(b) and (c), 63.446(c), (d), and (e), 63.447(b) or 63.450(d), shall install, calibrate, certify, operate, and maintain according to the manufacturer's specifications, a continuous monitoring system (CMS, as defined in § 63.2 of this part) as specified in paragraphs (b) through (m) of this section, except as allowed in paragraph (m) of this section. The CMS shall include a continuous recorder.

(b) A CMS shall be operated to measure the temperature in the firebox or in the ductwork immediately downstream of the firebox and before any substantial heat exchange occurs for each thermal oxidizer used to comply with the requirements of § 63.443(d)(1) through (d)(3). Owners and operators complying with the requirements in § 63.443(d)(2) or (d)(3) shall monitor the parameter specified and for the temperature and concentration limits specified.

(c) A CMS shall be operated to measure the following parameters for each gas scrubber used to comply with the bleaching system requirements of § 63.445(c) or the sulfite pulping system requirements of § 63.444(c).

(1) The pH or the oxidation/reduction potential of the gas scrubber effluent;

(2) The gas scrubber vent gas inlet flow rate; and

(3) The gas scrubber liquid influent flow rate.

(d) As an option to the requirements specified in paragraph (c) of this section, a CMS shall be operated to measure the chlorine outlet concentration of each gas scrubber used to comply with the bleaching system outlet concentration requirement specified in § 63.445(c)(2).

(e) The owner or operator of a bleaching system complying with 40 CFR 430.24, subpart B, shall monitor the chlorine and hypochlorite application rates, in kg of bleaching agent per megagram of ODP, of the bleaching system during the extended compliance period specified in § 63.440(d)(3).

(f) A CMS shall be operated to measure the gas scrubber parameters specified in paragraphs (c)(1) through (c)(3) of this section or those site specific parameters determined according to the procedures specified in paragraph (n) of this section to comply with the sulfite pulping system requirements specified in § 63.444(c).

(g) A CMS shall be operated to measure the following parameters for each steam stripper used to comply with the treatment requirements in § 63.446(e)(3), (4), or (5):

(1) The process wastewater feed rate;

(2) The steam feed rate; and

(3) The process wastewater column feed temperature.

(h) As an option to the requirements specified in paragraph (g) of this section, a CMS shall be operated to measure the methanol outlet concentration to comply with the steam stripper outlet concentration requirement specified in § 63.446(e)(4) or (e)(5).

(i) A CMS shall be operated to measure the appropriate parameters determined according to the procedures specified in paragraph (n) of this section to comply with the condensate applicability requirements specified in § 63.446(c).

(j) Each owner or operator using a biological treatment system to comply with § 63.446(e)(2) shall perform the following monitoring procedures.

(1) On a daily basis, monitor the following parameters for each biological treatment unit:

(i) Composite daily sample of outlet soluble BOD_5 concentration to monitor for maximum daily and maximum monthly average;

(ii) Mixed liquor volatile suspended solids;

(iii) Horsepower of aerator unit(s);

(iv) Inlet liquid flow; and

(v) Liquid temperature.

(2) Obtain daily inlet and outlet liquid grab samples from each biological treatment unit to have HAP data available to perform quarterly percent reduction tests specified in paragraph (j)(2)(ii) of this section and the compliance percent reduction tests specified in paragraph (p)(1)(i) of this section. Perform the following procedures with the liquid samples:

(i) Store the samples for 5 days as specified in § 63.457(n). The 5 day storage requirement is required since the soluble BOD5 test requires 5 days to obtain results. If the results of the soluble BOD5 test are outside of the range established during the initial performance test, then the archive sample shall be used to perform the percent reduction test specified in § 63.457(1).

(ii) Perform the percent reduction test procedures specified in § 63.457(l) within 45 days after the beginning of each quarter as follows.

(A) The percent reduction test performed in the first quarter (annually) shall be performed for total HAP and the percent reduction obtained from the test shall be at least as great as the total HAP reduction specified in § 63.446(e)(2).

(B) The remaining quarterly percent reduction tests shall be performed for methanol and the percent reduction obtained from the test shall be at least as great as the methanol reduction determined in the previous first-quarter test specified in paragraph (j)(2)(ii)(A) of this section.

(C) The parameter values used to calculate the percent reductions required in paragraphs (j)(2)(ii)(A) and (j)(2)(ii)(B) of this section shall be parameter values measured and samples taken in paragraph (j)(1) of this section.

(k) Each enclosure and closed-vent system used to comply with § 63.450(a) shall comply with the requirements specified in paragraphs (k)(1) through (k)(6) of this section.

(1) For each enclosure opening, a visual inspection of the closure mechanism specified in § 63.450(b) shall be performed at least once every 30 days to ensure the opening is maintained in the closed position and sealed.

(2) Each closed-vent system required by § 63.450(a) shall be visually inspected every 30 days and at other times as requested by the Administrator. The visual inspection shall include inspection of ductwork, piping, enclosures, and connections to covers for visible evidence of defects.

(3) For positive pressure closed-vent systems or portions of closed-vent systems, demonstrate no detectable leaks as specified in § 63.450(c) measured initially and annually by the procedures in § 63.457(d).

(4) Demonstrate initially and annually that each enclosure opening is maintained at negative pressure as specified in § 63.457(e).

(5) The valve or closure mechanism specified in § 63.450(d)(2) shall be inspected at least once every 30

days to ensure that the valve is maintained in the closed position and the emission point gas stream is not diverted through the bypass line.

(6) If an inspection required by paragraphs (k)(1) through (k)(5) of this section identifies visible defects in ductwork, piping, enclosures or connections to covers required by § 63.450, or if an instrument reading of 500 parts per million by volume or greater above background is measured, or if enclosure openings are not maintained at negative pressure, then the following corrective actions shall be taken as soon as practicable.

(i) A first effort to repair or correct the closed-vent system shall be made as soon as practicable but no later than 5 calendar days after the problem is identified.

(ii) The repair or corrective action shall be completed no later than 15 calendar days after the problem is identified.

(l) Each pulping process condensate closed collection system used to comply with § 63.446(d) shall be visually inspected every 30 days and shall comply with the inspection and monitoring requirements specified in § 63.964 of subpart RR of this part, except for the closed-vent system and control device inspection and monitoring requirements specified in § 63.964(a)(2) of subpart RR of this part, the closed-vent system and the control device shall meet the requirements specified in paragraphs (a) and (k) of this section.

(m) Each owner or operator using a control device, technique or an alternative parameter other than those specified in paragraphs (b) through (l) of this section shall install a CMS and establish appropriate operating parameters to be monitored that demonstrate, to the Administrator's satisfaction, continuous compliance with the applicable control requirements.

(n) To establish or reestablish, the value for each operating parameter required to be monitored under paragraphs (b) through (j), (l), and (m) of this section or to establish appropriate parameters for paragraphs (f), (i), and (m) of this section, each owner or operator shall use the following procedures:

(1) During the initial performance test required in § 63.457(a) or any subsequent performance test, continuously record the operating parameter;

(2) Determinations shall be based on the control performance and parameter data monitored during the performance test, supplemented if necessary by engineering assessments and the manufacturer's recommendations;

(3) The owner or operator shall provide for the Administrator’s approval the rationale for selecting the monitoring parameters necessary to comply with paragraphs (f), (i), and (m) of this section; and

(4) Provide for the Administrator's approval the rationale for the selected operating parameter value, and monitoring frequency, and averaging time. Include all data and calculations used to develop the value and a description of why the value, monitoring frequency, and averaging time demonstrate continuous compliance with the applicable emission standard.

(o) Each owner or operator of a control device subject to the monitoring provisions of this section shall operate the control device in a manner consistent with the minimum or maximum (as appropriate) operating parameter value or procedure required to be monitored under paragraphs (a) through (n) of this section and established under this subpart. Except as provided in paragraph (p) of this section, §§ 63.443 (e), or 63.446 (g), operation of the control device below minimum operating parameter values or above maximum operating parameter values established under this subpart or failure to perform procedures required by this subpart shall constitute a violation of the applicable emission standard of this subpart and be reported as a period of excess emissions.

(p) Each owner or operator of a biological treatment system complying with paragraph (j) of this section shall perform all the following requirements when the monitoring parameters specified in paragraphs (j)(1)(i) through (j)(1)(iii) of this section are below minimum operating parameter values or above maximum operating parameter values established in paragraph (n) of this section.

(1) The following shall occur and be recorded as soon as practical:

(i) Determine compliance with § 63.446(e)(2) using the percent reduction test procedures specified in § 63.457(l) and the monitoring data specified in paragraph (j)(1) of this section that coincide with the time period of the parameter excursion;

(ii) Steps shall be taken to repair or adjust the operation of the process to end the parameter excursion period; and

(iii) Steps shall be taken to minimize total HAP emissions to the atmosphere during the parameter excursion period.

(2) A parameter excursion is not a violation of the applicable emission standard if the percent reduction test

specified in paragraph (p)(1)(i) of this section demonstrates compliance with § 63.446(e)(2), and no maintenance or changes have been made to the process or control device after the beginning of a parameter excursion that would influence the results of the determination.

§ 63.454 Recordkeeping Requirements.

(a) The owner or operator of each affected source subject to the requirements of this subpart shall comply with the recordkeeping requirements of § 63.10 of subpart A of this part, as shown in table 1, and the requirements specified in paragraphs (b) through (d) of this section for the monitoring parameters specified in § 63.453.

(b) For each applicable enclosure opening, closed-vent system, and closed collection system, the owner or operator shall prepare and maintain a site-specific inspection plan including a drawing or schematic of the components of applicable affected equipment and shall record the following information for each inspection:

(1) Date of inspection;

(2) The equipment type and identification;

(3) Results of negative pressure tests for enclosures;

(4) Results of leak detection tests;

(5) The nature of the defect or leak and the method of detection (i.e., visual inspection or instrument detection);

(6) The date the defect or leak was detected and the date of each attempt to repair the defect or leak;

(7) Repair methods applied in each attempt to repair the defect or leak;

(8) The reason for the delay if the defect or leak is not repaired within 15 days after discovery;

(9) The expected date of successful repair of the defect or leak if the repair is not completed within 15 days;

(10) The date of successful repair of the defect or leak;

(11) The position and duration of opening of bypass line valves and the condition of any valve seals; and

(12) The duration of the use of bypass valves on computer controlled valves.

(c) The owner or operator of a bleaching system complying paragraph § 63.440(d)(3)(ii)(B) shall record the daily average chlorine and hypochlorite application rates, in kg of bleaching agent per megagram of ODP, of the bleaching system until the requirements specified in § 63.440(d)(3)(ii)(A) are met.

(d) The owner or operator shall record the CMS parameters specified in § 63.453 and meet the requirements specified in paragraph (a) of this section for any new affected process equipment or pulping process condensate stream that becomes subject to the standards in this subpart due to a process change or modification.

§ 63.455 Reporting Requirements.

(a) Each owner or operator of a source subject to this subpart shall comply with the reporting requirements of subpart A of this part as specified in table 1 and all the following requirements in this section. The initial notification report specified under § 63.9(b)(2) of subpart A of this part shall be submitted by [insert date 1 year after date published in the FEDERAL REGISTER].

(b) Each owner or operator of a kraft pulping system specified in § 63.440(d)(1) or a bleaching system specified in § 63.440(d)(3)(ii) shall submit, with the initial notification report specified under § 63.9(b)(2) of subpart A of this part and paragraph (a) of this section and update every two years thereafter, a non-binding control strategy report containing, at a minimum, the information specified in paragraphs (b)(1) through (b)(3) of this section in addition to the information required in § 63.9(b)(2) of subpart A of this part.

(1) A description of the emission controls or process modifications selected for compliance with the control requirements in this standard.

(2) A compliance schedule, including the dates by which each step toward compliance will be reached for each emission point or sets of emission points. At a minimum, the list of dates shall include:

(i) The date by which the major study(s) for determining the compliance strategy will be completed;

(ii) The date by which contracts for emission controls or process modifications will be awarded, or the date by which orders will be issued for the purchase of major components to accomplish emission controls or process changes;

(iii) The date by which on-site construction, installation of emission control equipment, or a process change is to be initiated;

(iv) The date by which on-site construction, installation of emissions control equipment, or a process change is to be completed;

(v) The date by which final compliance is to be achieved;

(vi) For compliance with paragraph § 63.440(d)(3)(ii), the tentative dates by which compliance with effluent limitation guidelines and standards intermediate pollutant load effluent reductions and as available, all the dates for the best available technology’s milestones reported in the

National Pollutant Discharge Elimination System authorized under section 402 of the Clean Water Act and for the best professional milestones in the Voluntary Advanced Technology Incentives Program under 40 CFR 430.24 (b)(2); and

(vii) The date by which the final compliance tests will be performed.

(3) Until compliance is achieved, revisions or updates shall be made to the control strategy report required by paragraph (b) of this section indicating the progress made towards completing the installation of the emission controls or process modifications during the 2-year period.

(c) The owner or operator of each bleaching system complying with § 63.440(d)(3)(ii)(B) shall certify in the report specified under § 63.10(e)(3) of subpart A of this part that the daily application rates of chlorine and hypochlorite for that bleaching system have not increased as specified in § 63.440(d)(3)(ii)(B) until the requirements of § 63.440(d)(3)(ii)(A) are met.

(d) The owner or operator shall meet the requirements specified in paragraph (a) of this section upon startup of any new affected process equipment or pulping process condensate stream that becomes subject to the standards of this subpart due to a process change or modification.

§ 63.456 [Reserved]

§ 63.457 Test methods and procedures.

(a) Initial performance test. An initial performance test is required for all emission sources subject to the limitations in §§ 63.443, 63.444, 63.445, 63.446, and 63.447, except those controlled by a combustion device that is designed and operated as specified in § 63.443(d)(3) or (d)(4).

(b) Vent sampling port locations and gas stream properties. For purposes of selecting vent sampling port locations and determining vent gas stream properties, required in §§ 63.443, 63.444, 63.445, and 63.447, each owner or operator shall comply with the applicable procedures in paragraphs (b)(1) through (b)(6) of this section.

(1) Method 1 or 1A of part 60, appendix A, as appropriate, shall be used for selection of the sampling site as follows:

(i) To sample for vent gas concentrations and volumetric flow rates, the sampling site shall be located prior to dilution of the vent gas stream and prior to release to the atmosphere;

(ii) For determining compliance with percent reduction requirements, sampling sites shall be located prior to the inlet of the control device and at the outlet of the control device; measurements shall be performed simultaneously at the two sampling sites; and

(iii) For determining compliance with concentration limits or mass emission rate limits, the sampling site shall be located at the outlet of the control device.

(2) No traverse site selection method is needed for vents smaller than 0.10 meter (4.0 inches) in diameter.

(3) The vent gas volumetric flow rate shall be determined using Method 2, 2A, 2C, or 2D of part 60, appendix A, as appropriate.

(4) The moisture content of the vent gas shall be measured using Method 4 of part 60, appendix A.

(5) To determine vent gas concentrations, the owner or operator shall collect a minimum of three samples that are representative of normal conditions and average the resulting pollutant concentrations using the following procedures.

(i) Method 308 in Appendix A of this part shall be used to determine the methanol concentration.

(ii) Except for the modifications specified in paragraphs (b)(5)(ii)(A) through (b)(5)(ii)(K) of this section, Method 26A of part 60, appendix A shall be used to determine chlorine concentration in the vent stream.

(A) Probe/Sampling Line. A separate probe is not required. The sampling line shall be an appropriate length of 0.64 cm (0.25 in) OD Teflon® tubing. The sample inlet end of the sampling line shall be inserted into the stack in such a way as to not entrain liquid condensation from the vent gases. The other end shall be connected to the impingers. The length of the tubing may vary from one sampling site to another, but shall be as short as possible in each situation. If sampling is conducted in sunlight, opaque tubing shall be used. Alternatively, if transparent tubing is used, it shall be covered with opaque tape.

(B) Impinger Train. Three 30 milliliter (ml) capacity midget impingers shall be connected in series to the sampling line. The impingers shall have regular tapered stems. Silica gel shall be placed in the third impinger as a desiccant. All impinger train connectors shall be glass and/or Teflon®.

(C) Critical Orifice. The critical orifice shall have a flow rate of 200 to 250 ml/min and shall be followed by a vacuum pump capable of providing a vacuum of 640 millimeters of mercury (mm Hg). A 45 millimeter diameter in-line Teflon® 0.8 micrometer filter shall follow the impingers to project the critical orifice and vacuum pump.

(D) The following are necessary for the analysis apparatus:

(1) Wash bottle filled with deionized water;

(2) 25 or 50 ml graduated burette and stand;

(3) Magnetic stirring apparatus and stir bar;

(4) Calibrated pH Meter;

(5) 150-250 ml beaker or flask; and

(6) A 5 ml pipette.

(E) The procedures listed in paragraphs (b)(5)(ii)(E)(1) through (b)(5)(ii)(E)(7) of this section shall be used to prepare the reagents.

(1) To prepare the 1 molarity (M) potassium dihydrogen phosphate solution, dissolve 13.61 grams (g) of potassium dihydrogen phosphate in water and dilute to 100 ml.

(2) To prepare the 1 M sodium hydroxide solution (NaOH), dissolve 4.0 g of sodium hydroxide in water and dilute to 100 ml.

(3) To prepare the buffered 2 percent potassium iodide solution, dissolve 20 g of potassium iodide in 900 ml water. Add 50 ml of the 1 M potassium dihydrogen phosphate solution and 30 ml of the 1 M sodium hydroxide solution. While stirring solution, measure the pH of solution electrometrically and add the 1 M sodium hydroxide solution to bring pH to between 6.95 and 7.05.

(4) To prepare the 0.1 normality (N) sodium thiosulfate solution, dissolve 25 g of sodium thiosulfate, pentahydrate, in 800 ml of freshly boiled and cooled distilled water in a 1-liter volumetric flask. Dilute to volume. To prepare the 0.01 N sodium thiosulfate solution, add 10.0 ml standardized 0.1 N sodium thiosulfate solution to a 100 ml volumetric flask, and dilute to volume with water.

(5) To standardize the 0.1 N sodium thiosulfate solution, dissolve 3.249 g of anhydrous potassium bi-iodate, primary standard quality, or 3.567 g potassium iodate dried at 103 +/- 2 degrees Centigrade for 1 hour, in distilled water and dilute to 1000 ml to yield a 0.1000 N solution. Store in a glass-stoppered bottle. To 80 ml distilled water, add, with constant stirring, 1 ml concentrated sulfuric acid, 10.00 ml 0.1000 N anhydrous potassium bi-iodate, and 1 g potassium iodide. Titrate immediately with 0.1 n sodium thiosulfate titrant until the yellow color of the liberated iodine is almost discharged. Add 1 ml starch indicator solution and continue titrating until the blue color disappears. The normality of the sodium thiosulfate solution is inversely proportional to the ml of sodium thiosulfate solution consumed:

$$\text{Normality of Sodium Thiosulfate} = \frac{1}{\text{ml Sodium Thiosulfate Consumed}}$$

(<u>6</u>) To prepare the starch indicator solution, add a small amount of cold water to 5 g starch and grind in a mortar to obtain a thin paste. Pour paste into 1 L of boiling distilled water, stir, and let settle overnight. Use clear supernate for starch indicator solution.

(<u>7</u>) To prepare the 10 percent sulfuric acid solution, add 10 ml of concentrated sulfuric acid to 80 ml water in an 100 ml volumetric flask. Dilute to volume.

(F) The procedures specified in paragraphs (b)(5)(ii)(F)(<u>1</u>) through (b)(5)(ii)(F)(<u>5</u>) of this section shall be used to perform the sampling.

(<u>1</u>) Preparation of Collection Train. Measure 20 ml buffered potassium iodide solution into each of the first two impingers and connect probe, impingers, filter, critical orifice, and pump. The sampling line and the impingers shall be shielded from sunlight.

(<u>2</u>) Leak and Flow Check Procedure. Plug sampling line inlet tip and turn on pump. If a flow of bubbles is visible in either of the liquid impingers, tighten fittings and adjust connections and impingers. A leakage rate not in excess of 2 percent of the sampling rate is acceptable.

Carefully remove the plug from the end of the probe. Check the flow rate at the probe inlet with a bubble tube flow meter. The flow should be comparable or slightly less than the flow rate of the critical orifice with the impingers off-line. Record the flow and turn off the pump.

(3) Sample Collection. Insert the sampling line into the stack and secure it with the tip slightly lower than the port height. Start the pump, recording the time. End the sampling after 60 minutes, or after yellow color is observed in the second in-line impinger. Record time and remove the tubing from the vent. Recheck flow rate at sampling line inlet and turn off pump. If the flow rate has changed significantly, redo sampling with fresh capture solution. A slight variation (less than 5 percent) in flow may be averaged. With the inlet end of the line elevated above the impingers, add about 5 ml water into the inlet tip to rinse the line into the first impinger.

(4) Sample Analysis. Fill the burette with 0.01 N sodium thiosulfate solution to the zero mark. Combine the contents of the impingers in the beaker or flask. Stir the solution and titrate with thiosulfate until the solution is colorless. Record the volume of the first endpoint (TN, ml). Add 5 ml of the 10 percent sulfuric acid solution, and continue the titration until the contents of the flask are

again colorless. Record the total volume of titrant required to go through the first and to the second endpoint (T_A, ml). If the volume of neutral titer is less than 0.5 ml, repeat the testing for a longer period of time. It is important that sufficient lighting be present to clearly see the endpoints, which are determined when the solution turns from pale yellow to colorless. A lighted stirring plate and a white background are useful for this purpose.

(5) Interferences. Known interfering agents of this method are sulfur dioxide and hydrogen peroxide. Sulfur dioxide, which is used to reduce oxidant residuals in some bleaching systems, reduces formed iodine to iodide in the capture solution. It is therefore a negative interference for chlorine, and in some cases could result in erroneous negative chlorine concentrations. Any agent capable of reducing iodine to iodide could interfere in this manner. A chromium trioxide impregnated filter will capture sulfur dioxide and pass chlorine and chlorine dioxide. Hydrogen peroxide, which is commonly used as a bleaching agent in modern bleaching systems, reacts with iodide to form iodine and thus can cause a positive interference in the chlorine measurement. Due to the chemistry involved, the precision of the chlorine analysis will decrease as the ratio of chlorine dioxide to chlorine increases. Slightly negative

calculated concentrations of chlorine may occur when sampling a vent gas with high concentrations of chlorine dioxide and very low concentrations of chlorine.

(G) The following calculation shall be performed to determine the corrected sampling flow rate:

$$S_C = S_U \left(\frac{BP - PW}{760} \right) \left(\frac{293}{273 + t} \right)$$

where:

S_C = Corrected (dry standard) sampling flow rate, liters per minute;

S_U = Uncorrected sampling flow rate, L/min;

BP = Barometric pressure at time of sampling;

PW = Saturated partial pressure of water vapor, mm Hg at temperature; and

t = Ambient temperature, °C.

(H) The following calculation shall be performed to determine the moles of chlorine in the sample:

$$Cl_2 \text{ Moles} = 1/8000\ (5\ T_N - T_A) \times N_{Thio}$$

where:

T_N = Volume neutral titer, ml;

T_A = Volume acid titer (total), ml; and

N_{Thio} = Normality of sodium thiosulfate titrant.

(I) The following calculation shall be performed to determine the concentration of chlorine in the sample:

$$Cl_2 \text{ ppmv} = \frac{3005\ (5\ T_N - T_A) \times N_{Thio}}{S_C \times t_S}$$

where:

S_C = Corrected (dry standard) sampling flow rate, liters per minute;

t_S = Time sampled, minutes;

T_N = Volume neutral titer, ml;

T_A = Volume acid titer (total), ml; and

N_{Thio} = Normality of sodium thiosulfate titrant.

(J) The following calculation shall be performed to determine the moles of chlorine dioxide in the sample:

$$ClO_2 \text{ Moles} = 1/4000(T_A - T_N) \times N_{Thio}$$

where:

T_A = Volume acid titer (total), ml;

T_N = Volume neutral titer, ml; and

N_{Thio} = Normality of sodium thiosulfate titrant.

(K) The following calculation shall be performed to determine the concentration of chlorine dioxide in the sample:

$$ClO_2 \text{ ppmv} = \frac{6010\ (T_A - T_N) \times N_{Thio}}{S_C \times t_S}$$

where:

S_C = Corrected (dry standard) sampling flow rate, liters per minute;

t_S = Time sampled, minutes;

T_A = Volume acid titer (total), ml;

T_N = Volume neutral titer, ml; and

N_{Thio} = Normality of sodium thiosulfate titrant.

(iii) Any other method that measures the total HAP or methanol concentration that has been demonstrated to the Administrator's satisfaction.

(6) The minimum sampling time for each of the three runs per method shall be 1 hour in which either an integrated sample or four grab samples shall be taken. If grab sampling is used, then the samples shall be taken at approximately equal intervals in time, such as 15 minute intervals during the run.

(c) Liquid sampling locations and properties. For purposes of selecting liquid sampling locations and for determining properties of liquid streams such as wastewaters, process waters, and condensates required in §§ 63.444, 63.446, and 63.447, the owner or operator shall comply with the following procedures:

(1) Samples shall be collected using the sampling procedures specified in Method 305 of part 60, appendix A;

(i) Where feasible, samples shall be taken from an enclosed pipe prior to the liquid stream being exposed to the atmosphere; and

(ii) When sampling from an enclosed pipe is not feasible, samples shall be collected in a manner to minimize exposure of the sample to the atmosphere and loss of HAP compounds prior to sampling.

(2) The volumetric flow rate of the entering and exiting liquid streams shall be determined using the inlet and outlet flow meters or other methods demonstrated to the Administrator's satisfaction. The volumetric flow rate measurements to determine actual mass removal shall be taken at the same time as the concentration measurements;

(3) To determine liquid stream total HAP or methanol concentrations, the owner or operator shall collect a minimum of three samples that are representative of normal conditions and average the resulting pollutant concentrations using one of the following:

(i) Method 305 in Appendix A of this part, adjusted using the following equation:

$$\overline{C} = \sum_{i=1}^{n} C_i/fm_i$$

where:

$\overline{C}$ = Pollutant concentration for the liquid stream, parts per million by weight.

C_i = Measured concentration of pollutant i in the liquid stream sample determined using Method 305, parts per million by weight.

fm_i = Pollutant-specific constant that adjusts concentration measured by Method 305 to actual liquid concentration; the fm for methanol is 0.85. Additional pollutant fm values can be found in table 34, subpart G of this part.

n = Number of individual pollutants, i, summed to calculate total HAP.

(ii) Any other method that measures total HAP concentration that has been demonstrated to the Administrator's satisfaction.

(4) To determine soluble BOD_5 in the effluent stream from a biological treatment unit used to comply with

§ 63.446(e)(2) and § 63.453(j), the owner or operator shall use Method 405.1, of part 136, with the following modifications:

(i) Filter the sample through the filter paper, into Erlenmeyer flask by applying a vacuum to the flask sidearm. Minimize the time for which vacuum is applied to prevent stripping of volatile organics from the sample. Replace filter paper as often as needed in order to maintain filter times of less than approximately 30 seconds per filter paper. No rinsing of sample container or filter bowl into the Erlenmeyer flask is allowed.

(ii) Perform Method 405.1 on the filtrate obtained in paragraph (c)(4) of this section. Dilution water shall be seeded with 1 milliliter of final effluent per liter of dilution water. Dilution ratios may require adjustment to reflect the lower oxygen demand of the filtered sample in comparison to the total BOD_5. Three BOD bottles and different dilutions shall be used for each sample.

(d) Detectable leak procedures. To measure detectable leaks for closed-vent systems as specified in § 63.450 or for pulping process wastewater collection systems as specified in § 63.446(d)(2)(i), the owner or operator shall comply with the following:

(1) Method 21, of part 60, appendix A; and

(2) The instrument specified in Method 21 shall be calibrated before use according to the procedures specified in Method 21 on each day that leak checks are performed. The following calibration gases shall be used:

(i) Zero air (less than 10 parts per million by volume 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 parts per million by volume methane or n-hexane.

(e) Negative pressure procedures. To demonstrate negative pressure at process equipment enclosure openings as specified in § 63.450(b), the owner or operator shall use one of the following procedures:

(1) An anemometer to demonstrate flow into the enclosure opening;

(2) Measure the static pressure across the opening;

(3) Smoke tubes to demonstrate flow into the enclosure opening; or

(4) Any other industrial ventilation test method demonstrated to the Administrator's satisfaction.

(f) HAP concentration measurements. For purposes of complying with the requirements in §§ 63.443, 63.444, and 63.447, the owner or operator shall measure the total HAP concentration as one of the following:

(1) As the sum of all individual HAP's; or

(2) As methanol.

(g) Condensate HAP concentration measurement. For purposes of complying with the kraft pulping condensate requirements in § 63.446, the owner or operator shall measure the total HAP concentration as methanol except for the purposes of complying with the initial performance test specified in § 63.457(a) for § 63.446(e)(2) and as specified in § 63.453 (j)(2)(ii).

(h) Bleaching HAP concentration measurement. For purposes of complying with the bleaching system requirements in § 63.445, the owner or operator shall measure the total HAP concentration as the sum of all individual chlorinated HAP’s or as chlorine.

(i) Vent gas stream calculations. To demonstrate compliance with the mass emission rate, mass emission rate per megagram of ODP, and percent reduction requirements for vent gas streams specified in §§ 63.443, 63.444, 63.445, and 63.447, the owner or operator shall use the following:

(1) The total HAP mass emission rate shall be calculated using the following equation:

$$E = K_2 \left[\sum_{j=1}^{n} C_j M_j \right] Q_s$$

where:

E = Mass emission rate of total HAP from the sampled vent, kilograms per hour.

K_2 = Constant, 2.494 x 10^{-6} (parts per million by volume)$^{-1}$ (gram-mole per standard cubic meter) (kilogram/gram) (minutes/hour), where standard temperature for (gram-mole per standard cubic meter) is 20 ^{o}C.

C_j = Concentration on a dry basis of pollutant j in parts per million by volume as measured by the test methods specified in paragraph (b) of this section.

M_j = Molecular weight of pollutant j, gram/gram-mole.

Q_s = Vent gas stream flow rate (dry standard cubic meter per minute) at a temperature of 20 ^{o}C as indicated in paragraph (b) of this section.

n = Number of individual pollutants, i, summed to calculate total HAP.

(2) The total HAP mass emission rate per megagram of ODP shall be calculated using the following equation:

$$F = \frac{E}{P}$$

where:

F = Mass emission rate of total HAP from the sampled vent, in kilograms per megagram of ODP.

E = Mass emission rate of total HAP from the sampled vent, in kilograms per hour determined as specified in paragraph (i)(1) of this section.

P = The production rate of pulp during the sampling period, in megagrams of ODP per hour.

(3) The total HAP percent reduction shall be calculated using the following equation:

$$R = \frac{E_i - E_o}{E_i} (100)$$

where:

R = Efficiency of control device, percent.

E_i = Inlet mass emission rate of total HAP from the sampled vent, in kilograms of pollutant per hour, determined as specified in paragraph (i)(1) of this section.

E_o = Outlet mass emission rate of total HAP from the sampled vent, in kilograms of pollutant per hour, determined as specified in paragraph (i)(1) of this section.

(j) Liquid stream calculations. To demonstrate compliance with the mass flow rate, mass per megagram of ODP, and percent reduction requirements for liquid streams specified in § 63.446, the owner or operator shall use the following:

(1) The mass flow rates of total HAP or methanol entering and exiting the treatment process shall be calculated using the following equations:

$$E_b = \frac{K}{n \times 10^6} \left(\sum_{i=1}^{n} V_{bi} C_{bi} \right)$$

$$E_a = \frac{K}{n \times 10^6} \left(\sum_{i=1}^{n} V_{ai} C_{ai} \right)$$

where:

E_b = Mass flow rate of total HAP or methanol in the liquid stream entering the treatment process, kilograms per hour.

E_a = Mass flow rate of total HAP or methanol in the liquid exiting the treatment process, kilograms per hour.

K = Density of the liquid stream, kilograms per cubic meter.

V_{bi} = Volumetric flow rate of liquid stream entering the treatment process during each run i, cubic meters per hour, determined as specified in paragraph (c) of this section.

V_{ai} = Volumetric flow rate of liquid stream exiting the treatment process during each run i, cubic meters per hour, determined as specified in paragraph (c) of this section.

C_{bi} = Concentration of total HAP or methanol in the stream entering the treatment process during each run i, parts per million by weight, determined as specified in paragraph (c) of this section.

C_{ai} = Concentration of total HAP or methanol in the stream exiting the treatment process during each run i, parts per million by weight, determined as specified in paragraph (c) of this section.

n = Number of runs.

(2) The mass of total HAP or methanol per megagram ODP shall be calculated using the following equation:

$$F = \frac{E}{P}$$

where:

F = Mass loading of total HAP or methanol in the sample, in kilograms per megagram of ODP.

E_a = Mass flow rate of total HAP or methanol in the wastewater stream in kilograms per hour as determined using the procedures in paragraph (j)(1) of this section.

P = The production rate of pulp during the sampling period in megagrams of ODP per hour.

(3) The percent reduction of total HAP across the applicable treatment process shall be calculated using the following equation:

$$R = \frac{E_b - E_a}{E_b} \times 100$$

where:

R = Control efficiency of the treatment process, percent.

E_b = Mass flow rate of total HAP in the stream entering the treatment process, kilograms per hour, as determined in paragraph (j)(1) of this section.

E_a = Mass flow rate of total HAP in the stream exiting the treatment process, kilograms per hour, as determined in paragraph (j)(1) of this section.

(4) Compounds that meet the requirements specified in paragraphs (j)(4)(i) or (4)(ii) of this section are not required to be included in the mass flow rate, mass per megagram of ODP, or the mass percent reduction determinations.

(i) Compounds with concentrations at the point of determination that are below 1 part per million by weight; or

(ii) Compounds with concentrations at the point of determination that are below the lower detection limit where the lower detection limit is greater than 1 part per million by weight.

(k) Oxygen concentration correction procedures. To demonstrate compliance with the total HAP concentration limit of 20 ppmv in § 63.443(d)(2), the concentration measured using the methods specified in paragraph (b)(5) of this section shall be corrected to 10 percent oxygen using the following procedures:

(1) The emission rate correction factor and excess air integrated sampling and analysis procedures of Methods 3A or 3B of part 60, appendix A shall be used to determine the oxygen concentration. The samples shall be taken at the same time that the HAP samples are taken.

(2) The concentration corrected to 10 percent oxygen shall be computed using the following equation:

$$C_c = C_m \left(\frac{10.9}{20.9 - \%O_{2d}} \right)$$

where:

C_c = Concentration of total HAP corrected to 10 percent oxygen, dry basis, parts per million by volume.

C_m = Concentration of total HAP dry basis, parts per million by volume, as specified in paragraph (b) of this section.

$\%O_{2d}$ = Concentration of oxygen, dry basis, percent by volume.

(l) <u>Biological treatment system percent reduction calculation</u>. To determine compliance with an open biological treatment system option specified in § 63.446(e)(2) and the monitoring requirements specified in § 63.453(j)(2), the percent reduction due to destruction in the biological treatment system shall be calculated using the following equation:

$$R = f_{bio} \times 100$$

where:

R = Destruction of total HAP or methanol in the biological treatment process, percent.

f_{bio} = The fraction of total HAP or methanol removed in the biological treatment system. The site-specific biorate constants shall be determined using the procedures specified and as limited in Appendix C of part 63.

(m) <u>Condensate segregation procedures</u>. The following procedures shall be used to demonstrate compliance with the condensate segregation requirements specified in § 63.446(c).

(1) To demonstrate compliance with the percent mass requirements specified in § 63.446(c)(1), the procedures specified in paragraphs (m)(1)(i) through (1)(iii) of this section shall be performed.

(i) Determine the total HAP mass of all condensates from each equipment system listed in § 63.446(b)(1) through (b)(3) using the procedures specified in paragraphs (c) and (j) of this section.

(ii) Multiply the total HAP mass determine in paragraph (m)(1)(i) of this section by 0.65 to determine the target HAP mass for the high-HAP fraction condensate stream or streams.

(iii) Compliance with the segregation requirements specified in § 63.446(c)(1) is demonstrated if the condensate stream or streams from each equipment system listed in § 63.446(b)(1) through (b)(3) being treated as specified in § 63.446(e) contain at least as much total HAP mass as the target total HAP mass determined in paragraph (m)(1)(ii) of this section.

(2) To demonstrate compliance with the percent mass requirements specified in § 63.446(c)(2), the procedures specified in paragraphs (m)(2)(i) through (2)(ii) of this section shall be performed.

(i) Determine the total HAP mass contained in the high-HAP fraction condensates from each equipment system listed in § 63.446(b)(1) through (b)(3) and the total condensates streams from the equipment systems listed in § 63.446(b)(4) and (b)(5), using the procedures specified in paragraphs (c) and (j) of this section.

(ii) Compliance with the segregation requirements specified in § 63.446(c)(2) is demonstrated if the total HAP mass determined in paragraph (m)(2)(i) of this section is equal to or greater than the appropriate mass requirements specified in § 63.446(c)(2).

(n) Biological treatment system monitoring sampling storage. The inlet and outlet grab samples required to be collected in § 63.453(j)(2) shall be stored at 4° C (40° F) to minimize the biodegradation of the organic compounds in the samples.

§ 63.458 Delegation of authority.

(a) In delegating implementation and enforcement authority to a State under section 112(d) of the CAA, 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:

§ 63.6(g) - Use of an alternative nonopacity emission standard;

§ 63.453(m) - Use of an alternative monitoring parameter;

§ 63.457(b)(5)(iii) - Use of an alternative test method for total HAP or methanol in vents; and

§ 63.457(c)(3)(ii) - Use of an alternative test method for total HAP or methanol in wastewater.

§ 63.459 [Reserved]

TABLE 1 TO SUBPART S. GENERAL PROVISIONS APPLICABILITY TO SUBPART S[a]

Reference	Applies to Subpart S	Comment
63.1(a)(1)- (3)	Yes	
63.1(a)(4)	Yes	Subpart S (this table) specifies applicability of each paragraph in subpart A to subpart S
63.1(a)(5)	No	Section reserved
63.1(a)(6)-(8)	Yes	
63.1(a)(9)	No	Section reserved
63.1(a)(10)	No	Subpart S and other cross-referenced subparts specify calendar or operating day
63.1(a)(11)-(14)	Yes	
63.1(b)(1)	No	Subpart S specifies its own applicability
63.1(b)(2)-(3)	Yes	
63.1(c)(1)-(2)	Yes	
63.1(c)(3)	No	Section reserved
63.1(c)(4)-(5)	Yes	
63.1(d)	No	Section reserved
63.1(e)	Yes	
63.2	Yes	
63.3	Yes	
63.4(a)(1)-63.4(a)(3)	Yes	
63.4(a)(4)	No	Section reserved
63.4(a)(5)	Yes	
63.4(b)	Yes	
63.4(c)	Yes	
63.5(a)	Yes	

TABLE 1 TO SUBPART S. GENERAL PROVISIONS APPLICABILITY TO SUBPART S[a] (CONTINUED)

Reference	Applies to Subpart S	Comment
63.5(b)(1)	Yes	
63.5(b)(2)	No	Section reserved
63.5(b)(3)	Yes	
63.5(b)(4)-(6)	Yes	
63.5(c)	No	Section reserved
63.5(d)	Yes	
63.5(e)	Yes	
63.5(f)	Yes	
63.6(a)	Yes	
63.6(b)	No	Subpart S specifies compliance dates for sources subject to subpart S
63.6(c)	No	Subpart S specifies compliance dates for sources subject to subpart S
63.6(d)	No	Section reserved
63.6(e)	Yes	
63.6(f)	Yes	
63.6(g)	Yes	
63.6(h)	No	Pertains to continuous opacity monitors that are not part of this standard
63.6(i)	Yes	
63.6(j)	Yes	
63.7	Yes	
63.8(a)(1)	Yes	
63.8(a)(2)	Yes	

TABLE 1 TO SUBPART S. GENERAL PROVISIONS APPLICABILITY TO SUBPART S[a](CONTINUED)

Reference	Applies to Subpart S	Comment
63.8(a)(3)	No	Section reserved
63.8(a)(4)	Yes	
63.8(b)(1)	Yes	
63.8(b)(2)	No	Subpart S specifies locations to conduct monitoring
63.8(b)(3)	Yes	
63.8(c)(1)	Yes	
63.8(c)(2)	Yes	
63.8(c)(3)	Yes	
63.8(c)(4)	No	Subpart S allows site specific determination of monitoring frequency in § 63.453 (n)(4)
63.8(c)(5)	No	Pertains to continuous opacity monitors that are not part of this standard
63.8(c)(6)	Yes	
63.8(c)(7)	Yes	
63.8(c)(8)	Yes	
63.8(d)	Yes	
63.8(e)	Yes	
63.8(f)(1)-(5)	Yes	
63.8(f)(6)	No	Subpart S does not specify relative accuracy test for CEM's
63.8(g)	Yes	
63.9(a)	Yes	

TABLE 1 TO SUBPART S. GENERAL PROVISIONS APPLICABILITY TO SUBPART S[a] (CONTINUED)

Reference	Applies to Subpart S	Comment
63.9(b)	Yes	Initial notifications must be submitted within one year after the source becomes subject to the relevant standard
63.9(c)	Yes	
63.9(d)	No	Special compliance requirements are only applicable to kraft mills
63.9(e)	Yes	
63.9(f)	No	Pertains to continuous opacity monitors that are not part of this standard
63.9(g)(1)	Yes	
63.9(g)(2)	No	Pertains to continuous opacity monitors that are not part of this standard
63.9(g)(3)	No	Subpart S does not specify relative accuracy tests, therefore no notification is required for an alternative
63.9(h)	Yes	
63.9(i)	Yes	
63.9(j)	Yes	
63.10(a)	Yes	
63.10(b)	Yes	
63.10(c)	Yes	
63.10(d)(1)	Yes	
63.10(d)(2)	Yes	

TABLE 1 TO SUBPART S. GENERAL PROVISIONS APPLICABILITY TO SUBPART S[a](CONTINUED)

Reference	Applies to Subpart S	Comment
63.10(d)(3)	No	Pertains to continuous opacity monitors that are not part of this standard
63.10(d)(4)	Yes	
63.10(d)(5)	Yes	
63.10(e)(1)	Yes	
63.10(e)(2)(i)	Yes	
63.10(e)(2)(ii)	No	Pertains to continuous opacity monitors that are not part of this standard
63.10(e)(3)	Yes	
63.10(e)(4)	No	Pertains to continuous opacity monitors that are not part of this standard
63.10(f)	Yes	
63.11-63.15	Yes	

[a] Wherever subpart A specifies "postmark" dates, submittals may be sent by methods other than the U.S. Mail (e.g., by fax or courier). Submittals shall be sent by the specified dates, but a postmark is not required.

3. Appendix A of part 63 is amended by adding Method 308 in numerical order to read as follows:

Appendix A to Part 63--Test Methods

* * * * *

Method 308--Procedure for Determination of Methanol Emission from Stationary Sources

1.0 Scope and Application.

1.1 Analyte. Methanol. Chemical Abstract Service (CAS) No. 67-56-1.

1.2 Applicability. This method applies to the measurement of methanol emissions from specified stationary sources.

2.0 Summary of Method. A gas sample is extracted from the sampling point in the stack. The methanol is collected in deionized distilled water and adsorbed on silica gel. The sample is returned to the laboratory where the methanol in the water fraction is separated from other organic compounds with a gas chromatograph (GC) and is then measured by a flame ionization detector (FID). The fraction adsorbed on silica gel is extracted with an aqueous solution of n-propanol and is then separated and measured by GC/FID.

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 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 to determine the applicability of regulatory limitations before performing this test method.

5.2 Methanol Characteristics. Methanol is flammable and a dangerous fire and explosion risk. It is moderately toxic by ingestion and inhalation.

<u>6.0 Equipment and Supplies</u>.

6.1 Sample Collection. The following items are required for sample collection:

6.1.1 Sampling Train. The sampling train is shown in Figure 308-1 and component parts are discussed below.

6.1.1.1 Probe. Teflon®, approximately 6-millimeter (mm) (0.24 inch) outside diameter.

6.1.1.2 Impinger. A 30-milliliter (ml) midget impinger. The impinger must be connected with leak-free glass connectors. Silicone grease may not be used to lubricate the connectors.

6.1.1.3 Adsorbent Tube. Glass tubes packed with the required amount of the specified adsorbent.

6.1.1.4 Valve. Needle valve, to regulate sample gas flow rate.

6.1.1.5 Pump. Leak-free diaphragm pump, or equivalent, to pull gas through the sampling train. Install a small surge tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rotameter.

6.1.1.6 Rate Meter. Rotameter, or equivalent, capable of measuring flow rate to within 2 percent of the selected flow rate of up to 1000 milliliter per minute (ml/min). Alternatively, the tester may use a critical orifice to set the flow rate.

6.1.1.7 Volume Meter. Dry gas meter (DGM), sufficiently accurate to measure the sample volume to within 2 percent, calibrated at the selected flow rate and conditions actually encountered during sampling, and equipped with a temperature sensor (dial thermometer, or equivalent) capable of measuring temperature accurately to within 3 ^{o}C (5.4 ^{o}F).

6.1.1.8 Barometer. Mercury (Hg), aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm (0.1 inch) Hg. See the NOTE in Method 5 (40 CFR part 60, appendix A), section 6.1.2.

6.1.1.9 Vacuum Gauge and Rotameter. At least 760-mm (30-inch) Hg gauge and 0- to 40-ml/min rotameter, to be used for leak-check of the sampling train.

6.2 Sample Recovery. The following items are required for sample recovery:

6.2.1 Wash Bottles. Polyethylene or glass, 500-ml, two.

6.2.2 Sample Vials. Glass, 40-ml, with Teflon®-lined septa, to store impinger samples (one per sample).

6.2.3 Graduated Cylinder. 100-ml size.

6.3 Analysis. The following are required for analysis:

6.3.1 Gas Chromatograph. GC with an FID, programmable temperature control, and heated liquid injection port.

6.3.2 Pump. Capable of pumping 100 ml/min. For flushing sample loop.

6.3.3 Flow Meter. To monitor accurately sample loop flow rate of 100 ml/min.

6.3.4 Regulators. Two-stage regulators used on gas cylinders for GC and for cylinder standards.

6.3.5 Recorder. To record, integrate, and store chromatograms.

6.3.6 Syringes. 1.0- and 10-microliter (l) size, calibrated, for injecting samples.

6.3.7 Tubing Fittings. Stainless steel, to plumb GC and gas cylinders.

6.3.8 Vials. Two 5.0-ml glass vials with screw caps fitted with Teflon®-lined septa for each sample.

6.3.9 Pipettes. Volumetric type, assorted sizes for preparing calibration standards.

6.3.10 Volumetric Flasks. Assorted sizes for preparing calibration standards.

6.3.11 Vials. Glass 40-ml with Teflon®-lined septa, to store calibration standards (one per standard).

7.0 Reagents and Standards.

NOTE: 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 Sampling. The following are required for sampling:

7.1.1 Water. Deionized distilled to conform to the American Society for Testing and Materials (ASTM) Specification D 1193-77, Type 3. At the option of the analyst, the potassium permanganate ($KMnO_4$) test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

7.1.2 Silica Gel. Deactivated chromatographic grade 20/40 mesh silica gel packed in glass adsorbent tubes. The silica gel is packed in two sections. The front section contains 520 milligrams (mg) of silica gel, and the back section contains 260 mg.

7.2 Analysis. The following are required for analysis:

7.2.1 Water. Same as specified in section 7.1.1.

7.2.2 n-Propanol, 3 Percent. Mix 3 ml of n-propanol with 97 ml of water.

7.2.3 Methanol Stock Standard. Prepare a methanol stock standard by weighing 1 gram of methanol into a 100-ml volumetric flask. Dilute to 100 ml with water.

7.2.3.1 Methanol Working Standard. Prepare a methanol working standard by pipetting 1 ml of the methanol stock standard into a 100-ml volumetric flask. Dilute the solution to 100 ml with water.

7.2.3.2 Methanol Standards For Impinger Samples. Prepare a series of methanol standards by pipetting 1, 2, 5, 10, and 25 ml of methanol working standard solution respectively into five 50-ml volumetric flasks. Dilute the solutions to 50 ml with water. These standards will have 2, 4, 10, 20, and 50 μg/ml of methanol, respectively. After preparation, transfer the solutions to 40-ml glass vials capped with Teflon® septa and store the vials under refrigeration. Discard any excess solution.

7.2.3.3 Methanol Standards for Adsorbent Tube Samples. Prepare a series of methanol standards by first pipetting 10 ml of the methanol working standard into a 100-ml volumetric flask and diluting the contents to exactly 100 ml with 3 percent n-propanol solution. This standard will contain 10 μg/ml of methanol. Pipette 5, 15, and 25 ml of this standard, respectively, into four 50-ml volumetric flasks. Dilute each solution to 50 ml with 3 percent n-propanol solution. These standards will have 1, 3, and 5 μg/ml of methanol, respectively. Transfer all four standards into 40-ml glass vials capped with Teflon®-lined septa and store under refrigeration. Discard any excess solution.

7.2.4 GC Column. Capillary column, 30 meters (100 feet) long with an inside diameter (ID) of 0.53 mm (0.02 inch), coated with DB 624 to a film thickness of 3.0 micrometers, (μm) or an equivalent column. Alternatively, a 30-meter capillary column coated with polyethylene glycol to a film thickness of 1 μm such as AT-WAX or its equivalent.

7.2.5 Helium. Ultra high purity.

7.2.6 Hydrogen. Zero grade.

7.2.7 Oxygen. Zero grade.

8.0 Procedure.

8.1 Sampling. The following items are required for sampling:

8.1.1 Preparation of Collection Train. Measure 20 ml of water into the midget impinger. The adsorbent tube must contain 520 mg of silica gel in the front section and 260 mg of silica gel in the backup section. Assemble the train as shown in Figure 308-1. An optional, second impinger that is left empty may be placed in front of the water-containing impinger to act as a condensate trap. Place crushed ice and water around the impinger.

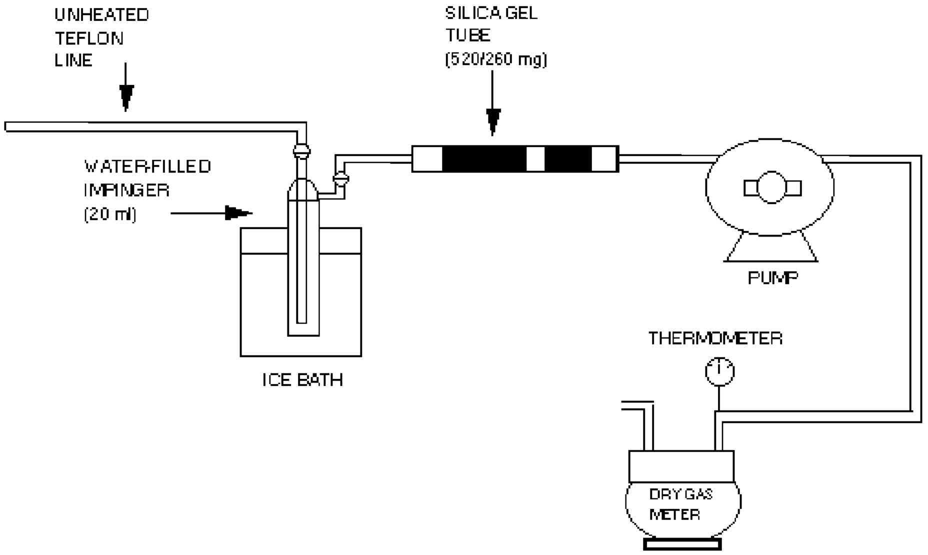

Figure 308.1. Sampling train schematic

8.1.2 Leak Check. A leak check prior to the sampling run is optional; however, a leak check after the sampling run is mandatory. The leak-check procedure is as follows:

Temporarily attach a suitable (e.g., 0- to 40-ml/min) rotameter to the outlet of the DGM, and place a vacuum gauge at or near the probe inlet. Plug the probe inlet, pull a vacuum of at least 250 mm (10 inch) Hg, and note the flow rate as indicated by the rotameter. A leakage rate not in excess of 2 percent of the average sampling rate is acceptable. NOTE: Carefully release the probe inlet plug before turning off the pump.

8.1.3 Sample Collection. Record the initial DGM reading and barometric pressure. To begin sampling, position the tip of the Teflon® tubing at the sampling point, connect the tubing to the impinger, and start the pump. Adjust the sample flow to a constant rate between 200 and 1000 ml/min as indicated by the rotameter. Maintain this constant rate (±10 percent) during the entire sampling run. Take readings (DGM, temperatures at DGM and at impinger outlet, and rate meter) at least every 5 minutes. Add more ice during the run to keep the temperature of the gases leaving the last impinger at 20 ^{o}C (68 ^{o}F) or less. At the conclusion of each run, turn off the pump, remove the Teflon® tubing from the stack, and record the final readings. Conduct a leak check as in section 8.1.2. (This leak check is mandatory.) If a leak is found, void the test run or use procedures acceptable to the Administrator to adjust the sample volume for the leakage.

8.2 Sample Recovery. The following items are required for sample recovery:

8.2.1 Impinger. Disconnect the impinger. Pour the contents of the midget impinger into a graduated cylinder. Rinse the midget impinger and the connecting tubes with water, and add the rinses to the graduated cylinder. Record the sample volume. Transfer the sample to a glass vial and cap with a Teflon® septum. Discard any excess sample. Place the samples in an ice chest for shipment to the laboratory.

8.2.2. Adsorbent Tubes. Seal the silica gel adsorbent tubes and place them in an ice chest for shipment to the laboratory.

9.0 Quality Control.

9.1 Miscellaneous Quality Control Measures. The following quality control measures are required:

Section	Quality control measure	Effect
8.1.2, 8.1.3, 10.1	Sampling equipment leak check and calibration	Ensures accurate measurement of sample volume

10.2	GC calibration	Ensures precision of GC analysis

9.2 Applicability. When the method is used to analyze samples to demonstrate compliance with a source emission regulation, an audit sample must be analyzed, subject to availability.

9.3 Audit Procedure. Analyze an audit sample with each set of compliance samples. Concurrently analyze the audit sample and a set of compliance samples in the same manner to evaluate the technique of the analyst and the standards preparation. The same analyst, analytical reagents, and analytical system shall be used both for the compliance samples and the EPA audit sample.

9.4 Audit Sample Availability. Audit samples will be supplied only to enforcement agencies for compliance tests. Audit samples may be obtained by writing:

Source Test Audit Coordinator (MD-77B)
Air Measurement Research Division
National Exposure Research Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

or by calling the Source Test Audit Coordinator (STAC) at (919) 541-7834. The audit sample request must be made at least 30 days prior to the scheduled compliance sample analysis.

9.5 Audit Results. Calculate the audit sample concentration according to the calculation procedure provided in the audit instructions included with the audit sample. Fill in the audit sample concentration and the analyst's name on the audit response form included with the audit instructions. Send one copy to the EPA Regional Office or the appropriate enforcement agency and a second copy to the STAC. The EPA Regional office or the appropriate enforcement agency will report the results of the audit to the laboratory being audited. Include this response with the results of the compliance samples in relevant reports to the EPA Regional Office or the appropriate enforcement agency.

10.0 Calibration and Standardization.

10.1 Metering System. The following items are required for the metering system:

10.1.1 Initial Calibration.

10.1.1.1 Before its initial use in the field, first leak-check the metering system (drying tube, needle valve, pump, rotameter, and DGM) as follows: Place a vacuum gauge at the inlet to the drying tube, and pull a vacuum of 250 mm (10 inch) Hg; plug or pinch off the outlet of the flow meter, and then turn off the pump. The vacuum shall remain stable for at least 30 seconds. Carefully release the vacuum gauge before releasing the flow meter end.

10.1.1.2 Next, remove the drying tube, and calibrate the metering system (at the sampling flow rate specified by the method) as follows: Connect an appropriately sized wet test meter (e.g., 1 liter per revolution (0.035 cubic feet per revolution)) to the inlet of the drying tube. Make three independent calibrations runs, using at least five revolutions of the DGM per run. Calculate the calibration factor, Y (wet test meter calibration volume divided by the DGM volume, both volumes adjusted to the same reference temperature and pressure), for each run, and average the results. If any Y-value deviates by more than 2 percent from the average, the metering system is unacceptable for use. Otherwise, use the average as the calibration factor for subsequent test runs.

10.1.2 Posttest Calibration Check. After each field test series, conduct a calibration check as in section 10.1.1 above, except for the following variations: (a) the leak check is not to be conducted, (b) three, or more revolutions of the DGM may be used, and (c) only two independent runs need be made. If the calibration factor does not deviate by more than 5 percent from the initial calibration factor (determined in section 10.1.1), then the

DGM volumes obtained during the test series are acceptable. If the calibration factor deviates by more than 5 percent, recalibrate the metering system as in section 10.1.1, and for the calculations, use the calibration factor (initial or recalibration) that yields the lower gas volume for each test run.

10.1.3 Temperature Sensors. Calibrate against mercury-in-glass thermometers.

10.1.4 Rotameter. The rotameter need not be calibrated, but should be cleaned and maintained according to the manufacturer's instruction.

10.1.5 Barometer. Calibrate against a mercury barometer.

10.2 Gas Chromatograph. The following procedures are required for the gas chromatograph:

10.2.1 Initial Calibration. Inject 1 μl of each of the standards prepared in sections 7.2.3.3 and 7.2.3.4 into the GC and record the response. Repeat the injections for each standard until two successive injections agree within 5 percent. Using the mean response for each calibration standard, prepare a linear least squares equation relating the response to the mass of methanol in the sample. Perform the calibration before analyzing each set of samples.

10.2.2 Continuing Calibration. At the beginning of each day, analyze the mid level calibration standard as described in section 10.5.1. The response from the daily analysis must agree with the response from the initial calibration within 10 percent. If it does not, the initial calibration must be repeated.

11.0 Analytical Procedure.

11.1 Gas Chromatograph Operating Conditions. The following operating conditions are required for the GC:

11.1.1 Injector. Configured for capillary column, splitless, 200 ^{o}C (392 ^{o}F).

11.1.2 Carrier. Helium at 10 ml/min.

11.1.3 Oven. Initially at 45 ^{o}C for 3 minutes; then raise by 10 ^{o}C to 70 ^{o}C; then raise by 70 ^{o}C/min to 200 ^{o}C.

11.2 Impinger Sample. Inject 1 μl of the stored sample into the GC. Repeat the injection and average the results. If the sample response is above that of the highest calibration standard, either dilute the sample until it is in the measurement range of the calibration line or prepare additional calibration standards. If the sample response is below that of the lowest calibration standard, prepare additional calibration standards. If additional calibration standards are prepared, there shall be at least two that bracket the response of the sample. These standards should produce approximately 50 percent and 150 percent of the response of the sample.

11.3 Silica Gel Adsorbent Sample. The following items are required for the silica gel adsorbent samples:

11.3.1 Preparation of Samples. Extract the front and backup sections of the adsorbent tube separately. With a file, score the glass adsorbent tube in front of the first section of silica gel. Break the tube open. Remove and discard the glass wool. Transfer the first section of the silica gel to a 5-ml glass vial and stopper the vial. Remove the spacer between the first and second section of the adsorbent tube and discard it. Transfer the second section of silica gel to a separate 5-ml glass vial and stopper the vial.

11.3.2 Desorption of Samples. Add 3 ml of the 10 percent n-propanol solution to each of the stoppered vials and shake or vibrate the vials for 30 minutes.

11.3.3 Inject a 1-μl aliquot of the diluted sample from each vial into the GC. Repeat the injection and average the results. If the sample response is above that of the highest calibration standard, either dilute the sample until it is in the measurement range of the calibration line or prepare additional calibration standards. If the sample response is below that of the lowest calibration standard, prepare additional calibration standards. If additional calibration standards are prepared, there shall be at least two that bracket the response of the sample. These standards should produce approximately 50 percent and 150 percent of the response of the sample.

12.0 Data Analysis and Calculations.

12.1 Nomenclature.

C_{af} = Concentration of methanol in the front of the adsorbent tube, μg/ml.

C_{ab} = Concentration of methanol in the back of the adsorbent tube, μg/ml.

C_i = Concentration of methanol in the impinger portion of the sample train, μg/ml.

E = Mass emission rate of methanol, μg/hr (lb/hr).

M_{tot} = Total mass of methanol collected in the sample train, μg.

P_{bar} = Barometric pressure at the exit orifice of the DGM, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

Q_{std} = Dry volumetric stack gas flow rate corrected to standard conditions, dscm/hr (dscf/hr).

T_m = Average DGM absolute temperature, degrees K (^oR).

T_{std} = Standard absolute temperature, 293 degrees K (528 ^oR).

V_{af} = Volume of front half adsorbent sample, ml.

V_{ab} = Volume of back half adsorbent sample, ml.

V_i = Volume of impinger sample, ml.

V_m = Dry gas volume as measured by the DGM, dry cubic meters (dcm), dry cubic feet (dcf).

$V_{m(std)}$ = Dry gas volume measured by the DGM, corrected to standard conditions, dry standard cubic meters (dscm), dry standard cubic feet (dscf).

12.2 Mass of Methanol. Calculate the total mass of methanol collected in the sampling train using Equation 308-1.

$$M_{tot} = V_i C_i + V_{af} C_{af} + V_{ab} C_{ab} \quad \text{Equation 308-1}$$

12.3 Dry Sample Gas Volume, Corrected to Standard Conditions. Calculate the volume of gas sampled at standard conditions using Equation 308-2.

$$V_m(std) = \frac{V_m \; Y \; T_{std} \; P_{bar}}{T_m \; P_{std}} \quad \text{Equation 308-2}$$

12.4 Mass Emission Rate of Methanol. Calculate the mass emission rate of methanol using Equation 308-3.

$$E = \frac{M_{tot}\ Q_{sd}}{V_{m(std)}} \qquad \text{Equation 308-3}$$

13.0 Method Performance. [Reserved]

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 Bibliography.

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3. Westlin, P.R. and R.T. Shigehara. "Procedure for Calibrating and Using Dry Gas Volume Meters as Calibration Standards." Source Evaluation Society Newsletter. 3(1):17-30. February 1978.

4. Yu, K.K. "Evaluation of Moisture Effect on Dry Gas Meter Calibration." Source Evaluation Society Newsletter. 5(1):24-28. February 1980.

5. NIOSH Manual of Analytical Methods, Volume 2. U.S. Department of Health and Human Services National Institute for Occupational Safety and Health. Center for Disease Control. 4676 Columbia Parkway, Cincinnati, OH 45226. (available from the Superintendent of Documents, Government Printing Office, Washington, DC 20402.)

6. Pinkerton, J.E. "Method for Measuring Methanol in Pulp Mill Vent Gases." National Council of the Pulp and Paper Industry for Air and Stream Improvement, Inc., New York, NY.

17.0 Tables, Diagrams, Flowcharts, and Validation Data.

[Reserved]

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PART 261--[AMENDED]

1. The authority citation of part 261 continues to read as follows:

Authority: 42 U.S.C. 6905, 6912(a), 6921, 6922, and 6938.

2. Section 261.4, is amended by adding (a)(15) to read as follows:

§ 261.4 Exclusions.

(a) *   *   *

(15) Condensates derived from the overhead gases from kraft mill steam strippers that are used to comply with 40 CFR part 63, subpart S, § 63.446(e). The exemption applies only to combustion at the mill generating the condensates.

*   *   *   *   *