

each House of the Congress and to the Comptroller General of the United States. This action is not a "major rule" as defined by 5 U.S.C. 804(2).

#### List of Subjects in 40 CFR Part 63

Environmental protection, Administrative practice and procedures, Air pollution control, Hazardous substances, Incorporation by reference, National Emission Standards for Hazardous Air Pollutants: Surface Coating of Wood Building Products Residual Risk and Technology Review, Intergovernmental relations, Reporting and recordkeeping requirements.

Dated: December 20, 2018.

Andrew R. Wheeler,  
Acting Administrator.

For the reasons set out in the preamble, title 40, chapter I, part 63 of the Code of Federal Regulations is amended as follows:

#### PART 63—[AMENDED]

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

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

#### Subpart A—[Amended]

- 2. Section 63.14 is amended:
- a. In paragraph (a), by removing—  
"http://www.archives.gov/federal\_register/code\_of\_federal\_regulations/ibr\_locations.html" and adding  
"www.archives.gov/federal-register/cfr/ibr-locations.html" in its place;
- b. By redesignating the paragraphs in the Old Paragraph column as the paragraphs in the New Paragraph column as follows:

Old paragraph	New paragraph
(c) .....	(f)
(d) .....	(g)
(e) through (g) .....	(c) through (e)
(h) through (s) .....	(m) through (t);

- c. In paragraph (h)—
- i. In the introductory text, by removing "American Society for Testing and Materials (ASTM)" and adding "ASTM International" in its place;
- ii. By redesignating the paragraphs in the Old Paragraph column as the paragraphs in the New Paragraph column as follows:

Old paragraph	New paragraph
(h)(13) through (h)(19) ....	(h)(14) through (h)(20)
(h)(20) through (h)(23) ....	(h)(22) through (h)(25)
(h)(24) through (h)(26) ....	(h)(27) through (h)(29)
(h)(27) through (h)(59) ....	(h)(31) through (h)(63)
(h)(60) through (h)(73) ....	(h)(65) through (h)(78)
(h)(74) through (h)(105) ..	(h)(80) through (h)(111);

- iii. By adding new paragraphs (h)(13), (21), (26), (30), (64), and (79); and

- iv. By revising newly redesignated paragraph (h)(84).
- d. By adding new paragraph (l); and
- e. By revising newly designated paragraph (p)(5).

The revisions and additions read as follows:

#### § 63.14 Incorporations by reference.

\* \* \* \* \*

(h) \* \* \*

(13) ASTM D1475–13, Standard Test Method for Density of Liquid Coatings, Inks, and Related Products, approved November 1, 2013, IBR approved for §§ 63.4741(b) and (c) and 63.4751(c).

\* \* \* \* \*

(21) ASTM D2111–10 (Reapproved 2015), Standard Test Methods for Specific Gravity and Density of Halogenated Organic Solvents and Their Admixtures, approved June 1, 2015, IBR approved for § 63.4741(a).

\* \* \* \* \*

(26) ASTM D2369–10 (Reapproved 2015)\*, Standard Test Method for Volatile Content of Coatings, approved June 1, 2015, IBR approved for § 63.4741(a).

\* \* \* \* \*

(30) ASTM D2697–03 (Reapproved 2014), Standard Test Method for Volume Nonvolatile Matter in Clear or Pigmented Coatings, approved July 1, 2014, IBR approved for § 63.4741(a) and (b).

\* \* \* \* \*

(64) ASTM D4840–99 (Reapproved 2018)\*, Standard Guide for Sampling Chain-of-Custody Procedures, approved August 15, 2018, IBR approved for appendix A to part 63.

\* \* \* \* \*

(79) ASTM D6093–97 (Reapproved 2016), Standard Test Method for Percent Volume Nonvolatile Matter in Clear or Pigmented Coatings Using a Helium Gas Pycnometer, Approved December 1, 2016, IBR approved for § 63.4741(a) and (b).

\* \* \* \* \*

(84) ASTM D6348–03 (Reapproved 2010), Standard Test Method for Determination of Gaseous Compounds by Extractive Direct Interface Fourier Transform Infrared (FTIR) Spectroscopy, including Annexes A1 through A8, Approved October 1, 2010, IBR approved for §§ 63.1571(a), 63.4751(i), 63.4752(e), 63.4766(b), tables 4 and 5 to subpart JJJJJ, tables 4 and 6 to subpart KKKKK, tables 1, 2, and 5 to subpart UUUUU and appendix B to subpart UUUUU.

\* \* \* \* \*

(l) Composite Panel Association, 19465 Deerfield Avenue, Suite 306,

Leesburg, VA 20176, Telephone (703)724–1128, and  
www.compositepanel.org.

(1) ANSI A135.4–2012, Basic Hardboard, approved June 8, 2012, IBR approved for § 63.4781.

(2) [Reserved]

\* \* \* \* \*

(p) \* \* \*

(5) NCASI Method ISS/FP A105.01, Impinger Source Sampling Method for Selected Aldehydes, Ketones, and Polar Compounds, December 2005, Methods Manual, IBR approved for table 4 to subpart DDDD and §§ 63.4751(i) and 63.4752(e).

\* \* \* \* \*

#### Subpart QQQQ—[Amended]

- 4. Section 63.4681 is amended by revising paragraph (c)(1) introductory text to read as follows:

#### § 63.4681 Am I subject to this subpart?

\* \* \* \* \*

(c) \* \* \*

(1) Surface coating in the processes identified in paragraphs (c)(1)(i) through (xi) of this section that are part of plywood and composite wood product manufacturing and subject to subpart DDDD of this part including:

\* \* \* \* \*

- 5. Section 63.4683 is amended by revising paragraphs (a) and (b) to read as follows:

#### § 63.4683 When do I have to comply with this subpart?

\* \* \* \* \*

(a) For a new or reconstructed affected source, the compliance date is the applicable date in paragraph (a)(1) or (2) of this section:

(1) If the initial startup of your new or reconstructed affected source is before May 28, 2003, the compliance date is May 28, 2003; except that the compliance date for the revised requirements promulgated at §§ 63.4700, 63.4710, 63.4720, 63.4730, 63.4741, 63.4751, 63.4752, 63.4761, 63.4763, 63.4764, 63.4766, 63.4781, table 4 of this subpart QQQQ, and appendix A to 40 CFR part 63 is September 3, 2019.

(2) If the initial startup of your new or reconstructed affected source occurs after May 28, 2003, the compliance date is March 4, 2019 or the date of initial startup of your affected source, whichever is later; except that if you commenced construction or reconstruction of your new or reconstructed affected source after May 28, 2003, but on or before May 16, 2018, the compliance date for the revised requirements promulgated at

§§ 63.4700, 63.4710, 63.4720, 63.4730, 63.4741, 63.4751, 63.4752, 63.4761, 63.4763, 63.4764, 63.4766, 63.4781, table 4 of this subpart QQQQ, and appendix A to 40 CFR part 63 is September 3, 2019.

(b) For an existing affected source, the compliance date is the date 3 years after May 28, 2003, except that the compliance date for the revised requirements promulgated at §§ 63.4700, 63.4710, 63.4720, 63.4730, 63.4741, 63.4751, 63.4752, 63.4761, 63.4763, 63.4764, 63.4766, 63.4781, table 4 of this subpart QQQQ of part 63, and appendix A to 40 CFR part 63 is September 3, 2019.

\* \* \* \* \*

■ 6. Section 63.4700 is amended by:

- a. Revising paragraph (a)(2) introductory text and paragraphs (a)(2)(i) and (ii);
- b. Adding paragraph (a)(3); and
- c. Revising paragraphs (b) and (d).

The revisions and addition read as follows:

**§ 63.4700 What are my general requirements for complying with this subpart?**

(a) \* \* \*

(2) Any coating operation(s) at existing sources for which you use the emission rate with add-on controls option, as specified in § 63.4691(c), must be in compliance with the applicable emission limitations as specified in paragraphs (a)(2)(i) through (iii) of this section.

(i) Before September 3, 2019, the coating operation(s) must be in compliance with the applicable emission limit in § 63.4690 at all times, except during periods of startup, shutdown, and malfunction (SSM). On and after September 3, 2019, the coating operation(s) must be in compliance with the applicable emission limit in § 63.4690 at all times.

(ii) Before September 3, 2019, the coating operation(s) must be in compliance with the applicable operating limits for emission capture systems and add-on control devices required by § 63.4692 at all times, except during periods of SSM, and except for solvent recovery systems for which you conduct liquid-liquid material balances according to § 63.4761(j). On and after September 3, 2019, the coating operation(s) must be in compliance with the operating limits for emission capture systems and add-on control devices required by § 63.4692 at all times, except for solvent recovery systems for which you conduct liquid-liquid material balances according to § 63.4761(j).

\* \* \* \* \*

(3) For new or reconstructed sources with initial startup after May 16, 2018, any coating operation(s) for which you use the emission rate with add-on controls option, as specified in § 63.4691(c), must be in compliance with the applicable emission limitations and work practice standards as specified in paragraphs (a)(3)(i) through (iii) of this section.

(i) The coating operation(s) must be in compliance with the applicable emission limit in § 63.4690 at all times.

(ii) The coating operation(s) must be in compliance with the operating limits for emission capture systems and add-on control devices required by § 63.4692 at all times, except for solvent recovery systems for which you conduct liquid-liquid material balances according to § 63.4761(j).

(iii) The coating operation(s) must be in compliance with the work practice standards in § 63.4693 at all times.

(b) For existing sources as of March 4, 2019, before September 3, 2019, you must always operate and maintain your affected source, including all air pollution control and monitoring equipment you use for purposes of complying with this subpart, according to the provisions in § 63.6(e)(1)(i). On and after September 3, 2019 for such existing sources and after March 4, 2019 for new or reconstructed sources, you must always operate and maintain your affected source, including associated air pollution control equipment and monitoring equipment, in a manner consistent with safety and good air pollution control practices for minimizing emissions. The general duty to minimize emissions does not require you to make any further efforts to reduce emissions if levels required by the applicable standard have been achieved. Determination of whether a source is operating in compliance with operation and maintenance requirements will be based on information available to the Administrator which may include, but is not limited to, monitoring results, review of operation and maintenance procedures, review of operation and maintenance records, and inspection of the source.

\* \* \* \* \*

(d) For existing sources, before September 3, 2019, if your affected source uses an emission capture system and add-on control device, you must develop a written startup, shutdown, and malfunction plan (SSMP) according to the provisions in § 63.6(e)(3). The SSMP must address startup, shutdown, and corrective actions in the event of a malfunction of the emission capture

system or the add-on control device. The SSMP must also address any coating operation equipment that may cause increased emissions or that would affect capture efficiency if the process equipment malfunctions, such as conveyors that move parts among enclosures.

■ 7. Section 63.4710 is amended by revising paragraph (c)(8)(ii) to read as follows:

**§ 63.4710 What notifications must I submit?**

\* \* \* \* \*

(c) \* \* \*

(8) \* \* \*

(ii) For the emission rate without add-on controls option, provide the calculation of the total mass of organic HAP emissions for each month; the calculation of the total volume of coating solids used each month; and the calculation of the 12-month organic HAP emission rate, using Equations 1 and 1A (or 1A-alt) through 1C, 2, and 3, respectively, of § 63.4751.

\* \* \* \* \*

■ 8. Section 63.4720 is amended by:

- a. Revising paragraph (a)(6)(ii) and paragraph (a)(7) introductory text;
- b. Redesignating paragraphs (a)(7)(i) through (xiv) as paragraphs (a)(7)(i)(A) through (N);
- c. Adding paragraph (a)(7)(i) introductory text and paragraph (a)(7)(ii);
- d. Revising paragraph (c) introductory text; and
- e. Adding paragraph (d).

The revisions and additions read as follows:

**§ 63.4720 What reports must I submit?**

(a) \* \* \*

(6) \* \* \*

(ii) The calculations used to determine the 12-month organic HAP emission rate for the compliance period in which the deviation occurred. You must provide the calculations for Equations 1, 1A (or 1A-alt) through 1C, 2, and 3 in § 63.4751; and if applicable, the calculation used to determine mass of organic HAP in waste materials according to § 63.4751(e)(4). You do not need to submit background data supporting these calculations (e.g., information provided by materials suppliers or manufacturers, or test reports).

\* \* \* \* \*

(7) *Deviations: Emission rate with add-on controls option.* You must be in compliance with the emission limitations in this subpart as specified in paragraphs (a)(7)(i) and (ii) of this section.

(i) For existing sources, before September 3, 2019, if you used the emission rate with add-on controls option and there was a deviation from an emission limitation (including any periods when emissions bypassed the add-on control device and were diverted to the atmosphere), the semiannual compliance report must contain the information in paragraphs (a)(7)(i)(A) through (N) of this section. This includes periods of SSM during which deviations occurred.

\* \* \* \* \*

(ii) After March 4, 2019 for new and reconstructed sources, and on and after September 3, 2019 for existing sources, if you used the emission rate with add-on controls option and there was a deviation from an emission limitation (including any periods when emissions bypassed the add-on control device and were diverted to the atmosphere), the semiannual compliance report must contain the information in paragraphs (a)(7)(ii)(A) through (M) of this section.

(A) The beginning and ending dates of each compliance period during which the 12-month organic HAP emission rate exceeded the applicable emission limit in § 63.4690.

(B) The calculations used to determine the 12-month organic HAP emission rate for each compliance period in which a deviation occurred. You must provide the calculation of the total mass of organic HAP emissions for the coatings, thinners, and cleaning materials used each month, using Equations 1 and 1A through 1C of § 63.4751; and, if applicable, the calculation used to determine mass of organic HAP in waste materials according to § 63.4751(e)(4); the calculation of the total volume of coating solids used each month, using Equation 2 of § 63.4751; the calculation of the mass of organic HAP emission reduction each month by emission capture systems and add-on control devices, using Equations 1 and 1A through 1D of § 63.4761, and Equations 2, 3, and 3A through 3C of § 63.4761, as applicable; the calculation of the total mass of organic HAP emissions each month, using Equation 4 of § 63.4761; and the calculation of the 12-month organic HAP emission rate, using Equation 5 of § 63.4761. You do not need to submit the background data supporting these calculations (e.g., information provided by materials suppliers or manufacturers, or test reports).

(C) A brief description of the CPMS.

(D) The date of the latest CPMS certification or audit.

(E) The date and time that each CPMS was inoperative, except for zero (low-level) and high-level checks.

(F) The date, time, and duration that each CPMS was out-of-control, including the information in § 63.8(c)(8).

(G) The date and time period of each deviation from an operating limit in Table 3 to this subpart, date and time period of any bypass of the add-on control device.

(H) A summary of the total duration of each deviation from an operating limit in Table 3 to this subpart, each bypass of the add-on control device during the semiannual reporting period, and the total duration as a percent of the total source operating time during that semiannual reporting period.

(I) A breakdown of the total duration of the deviations from the operating limits in Table 3 to this subpart and bypasses of the add-on control device during the semiannual reporting period by identifying deviations due to control equipment problems, process problems, other known causes, and other unknown causes; a list of the affected source or equipment, an estimate of the quantity of each regulated pollutant emitted over any emission limit, and a description of the method used to estimate the emissions.

(J) A summary of the total duration of CPMS downtime during the semiannual reporting period and the total duration of CPMS downtime as a percent of the total source operating time during that semiannual reporting period.

(K) A description of any changes in the CPMS, coating operation, emission capture system, or add-on control device since the last semiannual reporting period.

(L) For each deviation from the standard, including work practice standards, a description of the deviation, the date and time period of the deviation, and the actions you took to correct the deviation.

(M) A statement of the cause of each deviation.

\* \* \* \* \*

(c) *SSM reports.* For existing sources, before September 3, 2019, if you used the emission rate with add-on controls option and you had an SSM during the semiannual reporting period, you must submit the reports specified in paragraphs (c)(1) and (2) of this section.

\* \* \* \* \*

(d) *Electronic reporting.* (1) Within 60 days after the date of completing each performance test required by this subpart, you must submit the results of the performance test following the procedures specified in paragraphs (d)(1)(i) through (iii) of this section.

(i) *Data collected using test methods supported by EPA's Electronic Reporting Tool (ERT) as listed on EPA's ERT website (<https://www.epa.gov/electronic-reporting-air-emissions/electronic-reporting-tool-ert>) at the time of the test.* Submit the results of the performance test to the EPA via the Compliance and Emissions Data Reporting Interface (CEDRI), which can be accessed through EPA's Central Data Exchange (CDX) (<https://cdx.epa.gov/>). The data must be submitted in a file format generated through the use of EPA's ERT. Alternatively, you may submit an electronic file consistent with the extensible markup language (XML) schema listed on EPA's ERT website.

(ii) *Data collected using test methods that are not supported by EPA's ERT as listed on EPA's ERT website at the time of the test.* The results of the performance test must be included as an attachment in the ERT or an alternate electronic file consistent with the XML schema listed on EPA's ERT website. Submit the ERT generated package or alternative file to the EPA via CEDRI.

(iii) *Confidential business information (CBI).* If you claim some of the information submitted under paragraph (a)(1) of this section is CBI, you must submit a complete file, including information claimed to be CBI, to the EPA. The file must be generated through the use of EPA's ERT or an alternate electronic file consistent with the XML schema listed on EPA's ERT website. Submit the file on a compact disc, flash drive, or other commonly used electronic storage medium and clearly mark the medium as CBI. Mail the electronic medium to U.S. EPA/OAQPS/CORE CBI Office, Attention: Group Leader, Measurement Policy Group, MD C404-02, 4930 Old Page Rd., Durham, NC 27703. The same file with the CBI omitted must be submitted to the EPA via EPA's CDX as described in paragraph (d)(1)(i) of this section.

(2) You must submit the Notification of Compliance Status required in § 63.4710(c) and the semiannual compliance reports required in paragraph (a) of this section to the EPA via the CEDRI. (CEDRI can be accessed through the EPA's CDX (<https://cdx.epa.gov/>)). For semiannual compliance reports, you must use the appropriate electronic report in CEDRI for this subpart or an alternative electronic file format consistent with the XML schema listed on the CEDRI website (<https://www.epa.gov/electronic-reporting-air-emissions/compliance-and-emissions-data-reporting-interface-cedri>). If the reporting form specific to this subpart is not available in CEDRI at the time that

the report is due, you must submit the report to the Administrator at all the appropriate addresses listed in § 63.13. Once the reporting template has been available in CEDRI for 1 year, you must begin submitting all subsequent reports via CEDRI. For the Notification of Compliance Status, you must submit a file in portable document format (PDF) to CEDRI. The reports must be submitted by the deadlines specified in this subpart, regardless of the method in which the reports are submitted.

(3) If you are required to electronically submit a report through CEDRI in EPA's CDX, you may assert a claim of EPA system outage for failure to timely comply with the reporting requirement. To assert a claim of EPA system outage, you must meet the requirements outlined in paragraphs (d)(3)(i) through (vii) of this section.

(i) You must have been or will be precluded from accessing CEDRI and submitting a required report within the time prescribed due to an outage of either EPA's CEDRI or CDX systems.

(ii) The outage must have occurred within the period of time beginning five business days prior to the date that the submission is due.

(iii) The outage may be planned or unplanned.

(iv) You must submit notification to the Administrator in writing as soon as possible following the date you first knew, or through due diligence should have known, that the event may cause or has caused a delay in reporting.

(v) You must provide to the Administrator a written description identifying:

(A) The date(s) and time(s) when CDX or CEDRI was accessed and the system was unavailable;

(B) A rationale for attributing the delay in reporting beyond the regulatory deadline to EPA system outage;

(C) Measures taken or to be taken to minimize the delay in reporting; and

(D) The date by which you propose to report, or if you have already met the reporting requirement at the time of the notification, the date you reported.

(vi) The decision to accept the claim of EPA system outage and allow an extension to the reporting deadline is solely within the discretion of the Administrator.

(vii) In any circumstance, the report must be submitted electronically as soon as possible after the outage is resolved.

(4) If you are required to electronically submit a report through CEDRI in EPA's CDX, you may assert a claim of force majeure for failure to timely comply with the reporting requirement. To assert a claim of force

majeure, you must meet the requirements outlined in paragraphs (d)(4)(i) through (v) of this section.

(i) You may submit a claim if a force majeure event is about to occur, occurs, or has occurred or there are lingering effects from such an event within the period of time beginning five business days prior to the date the submission is due. For the purposes of this section, a force majeure event is defined as an event that will be or has been caused by circumstances beyond the control of the affected facility, its contractors, or any entity controlled by the affected facility that prevents you from complying with the requirement to submit a report electronically within the time period prescribed. Examples of such events are acts of nature (e.g., hurricanes, earthquakes, or floods), acts of war or terrorism, or equipment failure or safety hazard beyond the control of the affected facility (e.g., large scale power outage).

(ii) You must submit the notification to the Administrator in writing as soon as possible following the date you first knew, or through due diligence should have known, that the event may cause or has caused a delay in reporting.

(iii) You must provide to the Administrator:

(A) A written description of the force majeure event;

(B) A rationale for attributing the delay in reporting beyond the regulatory deadline to the force majeure event;

(C) Measures taken or to be taken to minimize the delay in reporting; and

(D) The date by which you propose to report, or if you have already met the reporting requirement at the time of the notification, the date you reported.

(iv) The decision to accept the claim of force majeure and allow an extension to the reporting deadline is solely within the discretion of the Administrator.

(v) In any circumstance, the reporting must occur as soon as possible after the force majeure event occurs.

■ 9. Section 63.4730 is amended by:

■ a. Revising paragraph (c)(3) and paragraph (k) introductory text;

■ b. Redesignating paragraphs (k)(1) through (4) as paragraphs (k)(1)(i) through (iv);

■ c. Adding paragraph (k)(1) introductory text and paragraph (k)(2);

■ d. Redesignating paragraphs (k)(5)(i) through (iii) as paragraphs (k)(1)(v)(A) through (C);

■ e. Redesignating paragraph (k)(5) introductory text as paragraph (k)(1)(v) introductory text and revising it;

■ f. Redesignating paragraphs (k)(6)(i) and (ii) as paragraphs (k)(1)(vi)(A) and (B);

■ g. Redesignating paragraph (k)(6) introductory text as paragraph (k)(1)(vi) introductory text and revising it; and

■ h. Redesignating paragraphs (k)(7) and (8) as paragraphs (k)(1)(vii) and (viii).

The revisions and additions read as follows:

#### § 63.4730 What records must I keep?

\* \* \* \* \*

(c) \* \* \*

(3) For the emission rate without add-on controls option, a record of the calculation of the total mass of organic HAP emissions for the coatings, thinners, and cleaning materials used each month, using Equations 1, 1A (or 1A-alt) through 1C, and 2 of § 63.4751; and, if applicable, the calculation used to determine mass of organic HAP in waste materials according to § 63.4751(e)(4); the calculation of the total volume of coating solids used each month, using Equation 2 of § 63.4751; and the calculation of each 12-month organic HAP emission rate, using Equation 3 of § 63.4751.

\* \* \* \* \*

(k) If you use the emission rate with add-on controls option, you must keep the records specified in paragraphs (k)(1) through (2) of this section.

(1) For existing sources, before September 3, 2019:

\* \* \* \* \*

(v) For each capture system that is not a PTE, the data and documentation you used to determine capture efficiency according to the requirements specified in §§ 63.4764 and 63.4765(b) through (e), including the records specified in paragraphs (k)(1)(v)(A) through (C) of this section that apply to you.

\* \* \* \* \*

(vi) The records specified in paragraphs (k)(1)(vi)(A) and (B) of this section for each add-on control device organic HAP destruction or removal efficiency determination as specified in § 63.4766.

\* \* \* \* \*

(2) After March 4, 2019 for new and reconstructed sources, and on and after September 3, 2019 for existing sources:

(i) The records required to show continuous compliance with each operating limit specified in Table 3 to this subpart that applies to you.

(ii) For each capture system that is a PTE, the data and documentation you used to support a determination that the capture system meets the criteria in Method 204 of appendix M to 40 CFR part 51 for a PTE and has a capture efficiency of 100 percent, as specified in § 63.4765(a).

(iii) For each capture system that is not a PTE, the data and documentation

you used to determine capture efficiency according to the requirements specified in §§ 63.4764 and 63.4765(b) through (e), including the records specified in paragraphs (k)(2)(iii)(A) through (C) of this section that apply to you.

(A) *Records for a liquid-to-uncaptured-gas protocol using a temporary total enclosure or building enclosure.* Records of the mass of total volatile hydrocarbon (TVH) as measured by Method 204A or F of appendix M to 40 CFR part 51 for each material used in the coating operation, and the total TVH for all materials used during each capture efficiency test run, including a copy of the test report. Records of the mass of TVH emissions not captured by the capture system that exited the temporary total enclosure or building enclosure during each capture efficiency test run as measured by Method 204D or E of appendix M to 40 CFR part 51, including a copy of the test report. Records documenting that the enclosure used for the capture efficiency test met the criteria in Method 204 of appendix M to 40 CFR part 51 for either a temporary total enclosure or a building enclosure.

(B) *Records for a gas-to-gas protocol using a temporary total enclosure or a building enclosure.* Records of the mass of TVH emissions captured by the emission capture system as measured by Method 204B or C of appendix M to 40 CFR part 51 at the inlet to the add-on control device, including a copy of the test report. Records of the mass of TVH emissions not captured by the capture system that exited the temporary total enclosure or building enclosure during each capture efficiency test run as measured by Method 204D or E of appendix M to 40 CFR part 51, including a copy of the test report. Records documenting that the enclosure used for the capture efficiency test met the criteria in Method 204 of appendix M to 40 CFR part 51 for either a temporary total enclosure or a building enclosure.

(C) *Records for an alternative protocol.* Records needed to document a capture efficiency determination using an alternative method or protocol as specified in § 63.4765(e), if applicable. (iv) The records specified in paragraphs (k)(2)(iv)(A) and (B) of this section for each add-on control device organic HAP destruction or removal efficiency determination as specified in § 63.4766.

(A) Records of each add-on control device performance test conducted according to §§ 63.4764 and 63.4766.

(B) Records of the coating operation conditions during the add-on control

device performance test showing that the performance test was conducted under representative operating conditions.

(v) Records of the data and calculations you used to establish the emission capture and add-on control device operating limits as specified in § 63.4767 and to document compliance with the operating limits as specified in Table 3 to this subpart.

(vi) A record of the work practice plan required by § 63.4693, and documentation that you are implementing the plan on a continuous basis.

■ 10. Section 63.4741 is amended by revising:

- a. Paragraph (a)(2);
- b. The subject heading and first sentence of paragraph (b)(1);
- c. The defined terms " $m_{\text{volatiles}}$ " and " $D_{\text{avg}}$ " in Equation 1 in paragraph (b)(3) introductory text; and
- d. Paragraph (c).

The revisions read as follows:

**§ 63.4741 How do I demonstrate initial compliance with the emission limitations?**

\* \* \* \* \*

(a) \* \* \*

(2) *Method 24 (appendix A-7 to 40 CFR part 60).* For coatings, you may use Method 24 to determine the mass fraction of nonaqueous volatile matter and use that value as a substitute for mass fraction of organic HAP. (Note: Method 24 is not appropriate for those coatings with a water content that would result in an effective detection limit greater than the applicable emission limit.) One of the voluntary consensus standards in paragraphs (a)(2)(i) through (iv) may be used as an alternative to using Method 24.

(i) ASTM Method D2111-10 (Reapproved 2015), "Standard Test Methods for Specific Gravity and Density of Halogenated Organic Solvents and Their Admixtures," (incorporated by reference, see § 63.14);

(ii) ASTM Method D2369-10 (Reapproved 2015)<sup>a</sup>, "Standard Test Method for Volatile Content of Coatings," (incorporated by reference, see § 63.14);

(iii) ASTM Method D2697-03 (Reapproved 2014), "Standard Test Method for Volume Nonvolatile Matter in Clear or Pigmented Coatings," (incorporated by reference, see § 63.14); and

(iv) ASTM Method D6093-97 (Reapproved 2016), "Standard Test Method for Percent Volume Nonvolatile Matter in Clear or Pigmented Coatings Using a Helium Gas Pycnometer," (incorporated by reference, see § 63.14).

\* \* \* \* \*

(b) \* \* \*

(1) *ASTM Method D2697-03 (Reapproved 2014) or D6093-97 (Reapproved 2016).* You may use ASTM Method D2697-03 (Reapproved 2014), "Standard Test Method for Volume Nonvolatile Matter in Clear or Pigmented Coatings" (incorporated by reference, see § 63.14), or D6093-97 (Reapproved 2016), "Standard Test Method for Percent Volume Nonvolatile Matter in Clear or Pigmented Coatings Using a Helium Gas Pycnometer" (incorporated by reference, see § 63.14), to determine the volume fraction of coating solids for each coating. \* \* \*

\* \* \* \* \*

(3) \* \* \*

$m_{\text{volatiles}}$  = Total volatile matter content of the coating, including HAP, volatile organic compounds (VOC), water, and exempt compounds, determined according to Method 24 in appendix A-7 of 40 CFR part 60, grams volatile matter per liter coating.

$D_{\text{avg}}$  = Average density of volatile matter in the coating, grams volatile matter per liter volatile matter, determined from test results using ASTM Method D1475-13, "Standard Test Method for Density of Liquid Coatings, Inks, and Related Products," (incorporated by reference, see § 63.14), information from the supplier or manufacturer of the material, or reference sources providing density or specific gravity data for pure materials. If there is disagreement between ASTM Method D1475-13 test results and other information sources, the test results will take precedence.

(c) *Determine the density of each coating.* Determine the density of each coating used during the compliance period from test results using ASTM Method D1475-13, "Standard Test Method for Density of Liquid Coatings, Inks, and Related Products," (incorporated by reference, see § 63.14), or information from the supplier or manufacturer of the material. If there is disagreement between ASTM Method D1475-13 test results and the supplier's or manufacturer's information, the test results will take precedence.

\* \* \* \* \*

■ 11. Section 63.4751 is amended by:

- a. Revising paragraph (c);
- b. Revising the defined term "A" in Equation 1 in of paragraph (e) introductory text; and
- c. Adding paragraph (i).

The revisions and addition read as follows:

**§ 63.4751 How do I demonstrate initial compliance with the emission limitations?**

\* \* \* \* \*

(c) *Determine the density of each material.* Determine the density of each coating, thinner, and cleaning material



used during each month from test results using ASTM Method D1475–13 (incorporated by reference, see § 63.14), information from the supplier or manufacturer of the material, or reference sources providing density or specific gravity data for pure materials. If there is disagreement between ASTM Method D1475–13 test results and such other information sources, the test results will take precedence.

\* \* \* \*

(e) \* \* \*

A = Total mass of organic HAP in the coatings used during the month, grams, as calculated in Equation 1A (or 1A-alt) of this section.

\* \* \* \*

(i) *Alternative compliance demonstration.* As an alternative to paragraph (h) of this section, you may demonstrate initial compliance by identifying each organic HAP component in the coating(s) and conducting a performance test using Method 320 of appendix A to 40 CFR part 63 or NCASI Method ISS/FP A105.01 (incorporated by reference in

§ 63.14) (for formaldehyde) or Method 326 of appendix A to 40 CFR part 63 (for isocyanates) to obtain an organic HAP emission factor (EF). The voluntary consensus standard ASTM D6348–03 (Reapproved 2010) (incorporated by reference, see § 63.14) may be used as an alternative to using Method 320 under the conditions specified in paragraphs (i)(4)(i) and (ii) of this section.

(1) You must also calculate the mass of organic HAP emitted from the coatings used during the month using Equation 1A-alt of this section:

$$A = \sum_{i=1}^m (Vol_{c,i})(D_{c,i})(W_{c,i})(EF_{c,i})$$

(Eq. 1A – alt)

Where:

A = Total mass of organic HAP in the coatings used during the month, grams.

Vol<sub>c,i</sub> = Total volume of coating, i, used during the month, liters.

D<sub>c,i</sub> = Density of coating, i, grams coating per liter of coatings.

W<sub>c,i</sub> = Mass fraction of organic HAP in coating, i, grams organic HAP per gram coating.

EF<sub>c,i</sub> = Organic HAP emission factor (three-run average from performance testing, evaluated as proportion of mass organic HAP emitted to mass of organic HAP in the coatings used during the performance test).

m = Number of different coatings used during the month.

(2) Calculate the organic HAP emission rate for the 12-month compliance period, grams organic HAP per liter coating solids used, using Equation 3 of this section.

(3) The organic HAP emission rate for the initial 12-month compliance period, calculated using Equation 3 of this section, must be less than or equal to the applicable emission limit in § 63.4690. You must keep all records as required by §§ 63.4730 and 63.4731. As part of the Notification of Compliance Status required by § 63.4710, you must identify the coating operation(s) for which you used the emission rate without add-on controls option and submit a statement that the coating operation(s) was (were) in compliance with the emission limitations during the initial compliance period because the organic HAP emission rate was less than or equal to the applicable emission limit in § 63.4690, determined according to this section.

(4) If ASTM D6348–03 (Reapproved 2010) is used, the conditions specified in paragraphs (i)(4)(i) and (ii) must be met.

(i) Test plan preparation and implementation in the Annexes to

ASTM D6348–03 (Reapproved 2010), sections A1 through A8 are mandatory.

(ii) In ASTM D6348–03 (Reapproved 2010) Annex A5 (Analyte Spiking Technique), the percent (%) R must be determined for each target analyte (Equation A5.5 of ASTM D6348–03). In order for the test data to be acceptable for a compound, %R must be between 70 and 130 percent. If the %R value does not meet this criterion for a target compound, the test data are not acceptable for that compound, and the test must be repeated for that analyte following adjustment of the sampling and/or analytical procedure before the retest. The %R value for each compound must be reported in the test report, and all field measurements must be corrected with the calculated %R value for that compound using the following equation: Reported Result = (Measured Concentration in the Stack × 100)/%R.

■ 12. Section 63.4752 is amended by adding paragraph (e) to read as follows:

**§ 63.4752 How do I demonstrate continuous compliance with the emission limitations?**

\* \* \* \*

(e) If you use the alternative compliance demonstration described in § 63.4751(i), you must identify each organic HAP component in the coating(s) and conduct a performance test every 5 years to obtain an organic HAP emission factor (EF). You must use the following methods, as appropriate: Method 320 of appendix A to 40 CFR part 63 or NCASI Method ISS/FP A105.01 (incorporated by reference, see § 63.14) (for formaldehyde) or Method 326 of appendix A to 40 CFR part 63 (for isocyanates). The voluntary consensus standard ASTM D6348–03 (Reapproved 2010) (incorporated by reference, see § 63.14) may be used as an alternative to

using Method 320 under the conditions specified in § 63.4751(i)(4)(i) and (ii).

■ 13. Section 63.4761 is amended by revising paragraph (j)(3) to read as follows:

**§ 63.4761 How do I demonstrate initial compliance?**

\* \* \* \*

(j) \* \* \*

(3) Determine the mass fraction of volatile organic matter for each coating, thinner, and cleaning material used in the coating operation controlled by the solvent recovery system during the month, grams volatile organic matter per gram coating. You may determine the volatile organic matter mass fraction using Method 24 of 40 CFR part 60, appendix A–7, one of the voluntary consensus standards specified in § 63.4741(a)(2)(i) through (iv), or an EPA approved alternative method, or you may use information provided by the manufacturer or supplier of the coating. In the event of any inconsistency between information provided by the manufacturer or supplier and the results of Method 24 of 40 CFR part 60, appendix A–7, or an approved alternative method, the test method results will take precedence unless after consultation, a regulated source could demonstrate to the satisfaction of the enforcement agency that the formulation data were correct.

\* \* \* \*

■ 14. Section 63.4763 is amended by revising paragraph (h) to read as follows:

**§ 63.4763 How do I demonstrate continuous compliance with the emission limitations?**

\* \* \* \*

(h) For existing sources, before September 3, 2019, consistent with §§ 63.6(e) and 63.7(e)(1), deviations that occur during a period of SSM of the

emission capture system, add-on control device, or coating operation that may affect emission capture or control device efficiency are not violations if you demonstrate to the Administrator's satisfaction that you were operating in accordance with § 63.6(e)(1). The Administrator will determine whether deviations that occur during a period you identify as an SSM are violations, according to the provisions in § 63.6(e).

■ 15. Section 63.4764 is amended by revising paragraphs (a)(1) and (2) to read as follows:

**§ 63.4764 What are the general requirements for performance tests?**

(a) \* \* \*

(1) *Representative coating operation operating conditions.* You must conduct the performance test under representative operating conditions for the coating operation. Operations during periods of startup, shutdown, and nonoperation do not constitute representative conditions. You may not conduct performance tests during periods of malfunction. You must record the process information that is necessary to document operating conditions during the test and explain why the conditions represent normal operation. Upon request, you shall make available to the Administrator such records as may be necessary to determine the conditions of performance tests.

(2) *Representative emission capture system and add-on control device operating conditions.* You must conduct the performance test when the emission capture system and add-on control device are operating at a representative flow rate, and the add-on control device is operating at a representative inlet concentration. Representative conditions exclude periods of startup and shutdown. You may not conduct performance tests during periods of malfunction. You must record information that is necessary to

document emission capture system and add-on control device operating conditions during the test and explain why the conditions represent normal operation.

\* \* \* \* \*

■ 16. Section 63.4766 is amended by revising paragraphs (a)(1) through (4), (b), (d), and (f) to read as follows:

**§ 63.4766 How do I determine the add-on control device emission destruction or removal efficiency?**

\* \* \* \* \*

(a) \* \* \*

(1) Use Method 1 or 1A of appendix A-1 to 40 CFR part 60, as appropriate, to select sampling sites and velocity traverse points.

(2) Use Method 2, 2A, 2C, 2D, or 2F of appendix A-1 to 40 CFR part 60, or Method 2G of appendix A-2 to 40 CFR part 60, as appropriate, to measure gas volumetric flow rate.

(3) Use Method 3, 3A, or 3B of appendix A-2 to 40 CFR part 60, as appropriate, for gas analysis to determine dry molecular weight. You may also use as an alternative to Method 3B, the manual method for measuring the oxygen, carbon dioxide, and carbon monoxide content of exhaust gas in ANSI/ASME PTC 19.10-1981, "Flue and Exhaust Gas Analyses [Part 10, Instruments and Apparatus]" (incorporated by reference, see § 63.14).

(4) Use Method 4 of appendix A-3 to 40 CFR part 60 to determine stack gas moisture.

\* \* \* \* \*

(b) Measure total gaseous organic mass emissions as carbon at the inlet and outlet of the add-on control device simultaneously, using Method 25 or 25A of appendix A-7 to 40 CFR part 60, and Method 320 or 326 of appendix A to 40 CFR part 63, as specified in paragraphs (b)(1) through (5) of this section. The voluntary consensus standard ASTM D6348-03 (Reapproved 2010) (incorporated by reference in § 63.14) may be used as an alternative to

using Method 320 if the conditions specified in § 63.4751(i)(4)(i) and (ii) are met. You must use the same method for both the inlet and outlet measurements.

(1) Use Method 25 of appendix A-7 to 40 CFR part 60 if the add-on control device is an oxidizer, and you expect the total gaseous organic concentration as carbon to be more than 50 parts per million (ppm) at the control device outlet.

(2) Use Method 25A of appendix A-7 to 40 CFR part 60 if the add-on control device is an oxidizer, and you expect the total gaseous organic concentration as carbon to be 50 ppm or less at the control device outlet.

(3) Use Method 25A of appendix A-7 to 40 CFR part 60 if the add-on control device is not an oxidizer.

(4) If Method 25A is used, and if formaldehyde is a major organic HAP component of the surface coating exhaust stream, use Method 320 of appendix A to 40 CFR part 63 or NCASI Method ISS/FP A105.01 (incorporated by reference in § 63.14) or ASTM D6348-03 (Reapproved 2010) (incorporated by reference in § 63.14) to determine formaldehyde concentration.

(5) In addition to Method 25 or 25A, use Method 326 of appendix A to 40 CFR part 63 if isocyanate is a major organic HAP component of the surface coating exhaust stream.

\* \* \* \* \*

(d) For each test run, determine the total gaseous organic emissions mass flow rates for the inlet and the outlet of the add-on control device, using Equation 1 of this section. If there is more than one inlet or outlet to the add-on control device, you must calculate the total gaseous organic mass flow rate using Equation 1 of this section for each inlet and each outlet and then total all of the inlet emissions and total all of the outlet emissions. The mass emission rates for formaldehyde and individual isocyanate must be determined separately.

$$M_f = Q_{sd} C_c MW (41.6) (10^{-6})$$

(Eq. 1)

Where:

$M_f$  = Total gaseous organic emissions mass flow rate, grams per hour (h).

$MW$  = Molecular weight of analyte of interest (12 for Method 25 and 25A results).

$C_c$  = Concentration of organic compounds in the vent gas (as carbon if determined by Method 25 or Method 25A), parts per million by volume (ppmv), dry basis.

$Q_{sd}$  = Volumetric flow rate of gases entering or exiting the add-on control device, as determined by Method 2, 2A, 2C, 2D, 2F,

or 2G, dry standard cubic meters/hour (dscm/h).

41.6 = Conversion factor for molar volume, gram-moles per cubic meter (mol/m<sup>3</sup>) (@ 293 Kelvin (K) and 760 millimeters of mercury (mmHg)).

\* \* \* \* \*

(f) Determine the emission destruction or removal efficiency of the add-on control device as the average of the efficiencies determined in the three test runs and calculated in Equation 2 of this

section. Destruction and removal efficiency must be determined independently for formaldehyde and isocyanates.

■ 17. Section 63.4781 is amended by revising paragraph (3) under the definition of "deviation" and revising the definition of "tileboard" to read as follows:

**§ 63.4781 What definitions apply to this subpart?**

\* \* \* \* \*

*Deviation \* \* \**

(3) On and after September 3, 2019, fails to meet any emission limit, or operating limit, or work practice standard in this subpart during SSM.

\* \* \* \* \*

*Tileboard* means hardboard that meets the specifications for Class I given by

the standard ANSI A135.4–2012 (incorporated by reference, see § 63.14) as approved by the American National Standards Institute. The standard specifies requirements and test methods for water absorption, thickness swelling, modulus of rupture, tensile strength, surface finish, dimensions, squareness, edge straightness, and moisture content for five classes of hardboard. Tileboard

is also known as Class I hardboard or tempered hardboard.

\* \* \* \* \*

■ 18. Table 4 to Subpart QQQQ is revised to read as follows:

Table 4 to Subpart QQQQ of Part 63—Applicability of General Provisions to Subpart QQQQ of Part 63

You must comply with the applicable General Provisions requirements according to the following table:

Citation	Subject	Applicable to subpart QQQQ	Explanation
§ 63.1(a)(1)–(14)	General Applicability	Yes.	Applicability to subpart QQQQ is also specified in § 63.4681.
§ 63.1(b)(1)–(3)	Initial Applicability Determination	Yes	
§ 63.1(c)(1)	Applicability After Standard Established	Yes.	Area sources are not subject to subpart QQQQ.
§ 63.1(c)(2)	Applicability of Permit Program for Area Sources	No	
§ 63.1(c)(3)	[Reserved]	No.	
§ 63.1(c)(4)–(5)	Extensions and Notifications	Yes.	
§ 63.1(d)	[Reserved]	No.	
§ 63.1(e)	Applicability of Permit Program Before Relevant Standard is Set.	Yes.	Additional definitions are specified in § 63.4781.
§ 63.2	Definitions	Yes	
§ 63.3(a)–(c)	Units and Abbreviations	Yes.	
§ 63.4(a)(1)–(5)	Prohibited Activities	Yes.	
§ 63.4(b)–(c)	Circumvention/Severability	Yes.	
§ 63.5(a)	Construction/Reconstruction	Yes.	
§ 63.5(b)(1)–(6)	Requirements for Existing, Newly Constructed, and Reconstructed Sources.	Yes.	
§ 63.5(c)	[Reserved]	No.	
§ 63.5(d)	Application for Approval of Construction/Reconstruction.	Yes.	
§ 63.5(e)	Approval of Construction/Reconstruction	Yes.	
§ 63.5(f)	Approval of Construction/Reconstruction Based on Prior State Review.	Yes.	
§ 63.6(a)	Compliance With Standards and Maintenance Requirements—Applicability.	Yes.	
§ 63.6(b)(1)–(7)	Compliance Dates for New and Reconstructed Sources.	Yes	
§ 63.6(c)(1)–(5)	Compliance Dates for Existing Sources	Yes	
§ 63.6(d)	[Reserved]	No.	
§ 63.6(e)(1)(i)	General Duty to Minimize Emissions	No	See § 63.4700(b) for general duty requirement.
§ 63.6(e)(1)(ii)	Requirement to Correct Malfunctions ASAP	No.	
§ 63.6(e)(1)(iii)	Operation and Maintenance Requirements Enforceable Independent of Emissions Limitations.	Yes.	Subpart QQQQ does not establish opacity standards and does not require continuous opacity monitoring systems (COMS).
§ 63.6(e)(2)	[Reserved]	No.	
§ 63.6(e)(3)	SSMP	No.	
§ 63.6(f)(1)	Compliance Except During SSM	No.	
§ 63.6(f)(2)–(3)	Methods for Determining Compliance	Yes.	
§ 63.6(g)(1)–(3)	Use of an Alternative Standard	Yes.	
§ 63.6(h)	Compliance with Opacity/Visible Emissions Standards.	No	
§ 63.6(i)(1)–(16)	Extension of Compliance	Yes.	Applies to all affected sources. Additional requirements for performance testing are specified in §§ 63.4751, 63.4752, 63.4764, 63.4765, and 63.4766.
§ 63.6(j)	Presidential Compliance Exemption	Yes.	
§ 63.7(a)(1)	Performance Test Requirements—Applicability	Yes	
§ 63.7(a)(2)	Performance Test Requirements—Dates	Yes	Applies only to performance tests for capture system and control device efficiency at sources using these to comply with the standard. § 63.4760 specifies the schedule for performance test requirements that are earlier than those specified in § 63.7(a)(2).
§ 63.7(a)(3)	Performance Tests Required By the Administrator.	Yes.	
§ 63.7(a)(4)	Notification of Delay in Performance Testing Due to Force Majeure.	Yes.	



Citation	Subject	Applicable to subpart QQQQ	Explanation
§ 63.7(b)–(d) .....	Performance Test Requirements—Notification, Quality Assurance, Facilities Necessary for Safe Testing, Conditions During Test.	Yes .....	Applies only to performance tests for capture system and add-on control device efficiency at sources using these to comply with the standard.
§ 63.7(e)(1) .....	Performance Testing .....	Yes.	
§ 63.7(f) .....	Performance Test Requirements—Use of Alternative Test Method.	Yes .....	Applies to all test methods except those used to determine capture system efficiency.
§ 63.7(g)–(h) .....	Performance Test Requirements—Data Analysis, Recordkeeping, Reporting, Waiver of Test.	Yes .....	Applies only to performance tests for capture system and add-on control device efficiency at sources using these to comply with the standard.
§ 63.8(a)(1)–(2) .....	Monitoring Requirements—Applicability .....	Yes .....	Applies only to monitoring of capture system and add-on control device efficiency at sources using these to comply with the standard. Additional requirements for monitoring are specified in § 63.4768.
§ 63.8(a)(3) .....	{Reserved} .....	No.	
§ 63.8(a)(4) .....	Additional Monitoring Requirements .....	No .....	Subpart QQQQ does not have monitoring requirements for flares.
§ 63.8(b) .....	Conduct of Monitoring .....	Yes.	
§ 63.8(c)(1) .....	Continuous Monitoring System (CMS) Operation and Maintenance.	Yes .....	Applies only to monitoring of capture system and add-on control device efficiency at sources using these to comply with the standard. Additional requirements for CMS operations and maintenance are specified in § 63.4768.
§ 63.8(c)(1)(i) .....	General Duty to Minimize Emissions and CMS Operation.	No.	
§ 63.8(c)(1)(ii) .....	Operation and Maintenance of CMS .....	Yes.	
§ 63.8(c)(1)(iii) .....	Requirement to Develop SSM Plan for CMS .....	No.	
§ 63.8(c)(2)–(3) .....	Monitoring System Installation .....	Yes.	
§ 63.8(c)(4) .....	CMSs .....	No .....	§ 63.4768 specifies the requirements for the operation of CMS for capture systems and add-on control devices at sources using these to comply.
§ 63.8(c)(5) .....	COMS .....	No .....	Subpart QQQQ does not have opacity for visible emission standards.
§ 63.8(c)(6) .....	CMS Requirements .....	Yes .....	§ 63.4768 specifies the requirements for monitoring systems for capture systems and add-on control devices at sources using these to comply.
§ 63.8(c)(7) .....	CMS Out-of-Control Periods .....	Yes.	
§ 63.8(c)(8) .....	CMS Out-of-Control Periods Reporting .....	No .....	§ 63.4720 requires reporting of CMS out-of-control periods.
§ 63.8(d)–(e) .....	Quality Control Program and CMS Performance Evaluation.	No .....	Subpart QQQQ does not require the use of continuous emissions monitoring systems.
§ 63.8(f)(1)–(5) .....	Use of an Alternative Monitoring Method .....	Yes.	
§ 63.8(f)(6) .....	Alternative to Relative Accuracy Test .....	No .....	Subpart QQQQ does not require the use of continuous emissions monitoring systems.
§ 63.8(g)(1)–(5) .....	Data Reduction .....	No .....	§§ 63.4767 and 63.4768 specify monitoring data reduction.
§ 63.9(a)–(d) .....	Notification Requirements .....	Yes.	
§ 63.9(e) .....	Notification of Performance Test .....	Yes .....	Applies only to capture system and add-on control device performance tests at sources using these to comply with the standard.
§ 63.9(f) .....	Notification of Visible Emissions/Opacity Test .....	No .....	Subpart QQQQ does not have opacity or visible emission standards.
§ 63.9(g)(1)–(3) .....	Additional Notifications When Using CMS .....	No .....	Subpart QQQQ does not require the use of continuous emissions monitoring systems.
§ 63.9(h) .....	Notification of Compliance Status .....	Yes .....	§ 63.4710 specifies the dates for submitting the Notification of Compliance Status.
§ 63.9(i) .....	Adjustment of Submittal Deadlines .....	Yes.	
§ 63.9(j) .....	Change in Previous Information .....	Yes.	
§ 63.10(a) .....	Recordkeeping/Reporting—Applicability and General Information.	Yes.	
§ 63.10(b)(1) .....	General Recordkeeping Requirements .....	Yes .....	Additional requirements are specified in §§ 63.4730 and 63.4731.
§ 63.10(b)(2)(i)–(ii) .....	Recordkeeping of Occurrence and Duration of Startups and Shutdowns.	No.	
§ 63.10(b)(2)(iii) .....	Recordkeeping Relevant to CMS .....	Yes.	
§ 63.10(b)(2)(iv)–(v) .....	Recordkeeping Relevant to SSM .....	No.	
§ 63.10(b)(2)(vi)–(xi) .....	Recordkeeping for CMS Malfunctions .....	Yes.	
§ 63.10(b)(2)(xii) .....	Records .....	Yes.	

Citation	Subject	Applicable to subpart QQQQ	Explanation
§ 63.10(b)(2)(xiii) .....	.....	No .....	Subpart QQQQ does not require the use of continuous emissions monitoring systems.
§ 63.10(b)(2)(xiv) .....	.....	Yes.	
§ 63.10(b)(3) .....	Recordkeeping Requirements for Applicability Determinations.	Yes.	
§ 63.10(c)(1)–(6) .....	Additional Recordkeeping Requirements for Sources with CMS.	Yes.	The same records are required in § 63.4720(a)(7).
§ 63.10(c)(7)–(8) .....	.....	No .....	
§ 63.10(c)(9)–(14) .....	.....	Yes.	
§ 63.10(c)(15) .....	Use of SSM Plan .....	No.	Additional requirements are specified in § 63.4720.
§ 63.10(d)(1) .....	General Reporting Requirements .....	Yes .....	
§ 63.10(d)(2) .....	Report of Performance Test Results .....	Yes .....	
§ 63.10(d)(3) .....	Reporting Opacity or Visible Emissions Observations.	No .....	Subpart QQQQ does not require opacity or visible emissions observations.
§ 63.10(d)(4) .....	Progress Reports for Sources With Compliance Extensions.	Yes.	
§ 63.10(d)(5) .....	SSM Reports .....	No .....	
§ 63.10(e)(1)–(2) .....	Additional CMS Reports .....	No .....	Subpart QQQQ does not require the use of continuous emissions monitoring systems. § 63.4720(b) specifies the contents of periodic compliance reports.
§ 63.10(e)(3) .....	Excess Emissions/CMS Performance Reports ....	No .....	
§ 63.10(e)(4) .....	COMS Data Reports .....	No .....	
§ 63.10(f) .....	Recordkeeping/Reporting Waiver .....	Yes.	Subpart QQQQ does not specify requirements for opacity or COMS.
§ 63.11 .....	Control Device Requirements/Flares .....	No .....	
§ 63.12 .....	State Authority and Delegations .....	Yes.	
§ 63.13 .....	Addresses .....	Yes.	Test Methods ANSI A135.4–2012, ANSI/ASME PTC 19.10–1981, Part 10, ASTM D1475–13, ASTM D2111–10 (Reapproved 2015), ASTM D2369–10 (Reapproved 2015) <sup>e</sup> , ASTM D2697–03 (Reapproved 2014), ASTM D4840–99 (2018) <sup>e</sup> , ASTM D6093–97 (Reapproved 2016), ASTM D6348–03 (Reapproved 2010) and NCASI Method ISS/FP A105.01 (incorporated by reference, see § 63.14).
§ 63.14 .....	Incorporation by Reference .....	Yes .....	
§ 63.15 .....	Availability of Information/Confidentiality .....	Yes.	
§ 63.16 .....	Requirements for Performance Track Member Facilities.	Yes.	

■ 19. Appendix A to part 63 is amended by adding Method 326 in numerical order to read as follows:

#### Appendix A to Part 63—Test Methods

\* \* \* \* \*

#### Method 326—Method for Determination of Isocyanates in Stationary Source Emissions

##### 1.0 Scope and Application

This method is applicable to the collection and analysis of isocyanate compounds from the emissions associated with manufacturing processes. This method is not inclusive with respect to specifications (e.g., equipment and supplies) and sampling procedures essential to its performance. Some material is incorporated by reference from other EPA

methods. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least Method 1, Method 2, Method 3, and Method 5 found in Appendices A–1, A–2, and A–3 in Part 60 of this title.

1.1 Analytes. This method is designed to determine the mass emission of isocyanates being emitted from manufacturing processes. The following is a table (Table 1–1) of the isocyanates and the manufacturing process at which the method has been evaluated:

TABLE 326–1—ANALYTES

Compound's name	CAS No.	Detection limit (ng/m <sup>3</sup> ) <sup>a</sup>	Manufacturing process
2,4-Toluene Diisocyanate (TDI) .....	584–84–9	106	Flexible Foam Production.
1,6-Hexamethylene Diisocyanate (HDI) .....	822–06–0	396	Paint Spray Booth.
Methylene Diphenyl Diisocyanate (MDI) .....	101–68–8	112	Pressed Board Production.
Methyl Isocyanate (MI) .....	624–83–0	228	Not used in production.

<sup>a</sup> Estimated detection limits are based on a sample volume of 1 m<sup>3</sup> and a 10-ml sample extraction volume.

1.2 **Applicability.** Method 326 is a method designed for determining compliance with National Emission Standards for Hazardous Air Pollutants (NESHAP). Method 326 may also be specified by New Source Performance Standards (NSPS), State Implementation Plans (SIPs), and operating permits that require measurement of isocyanates in stationary source emissions, to determine compliance with an applicable emission standard or limit.

1.3 **Data Quality Objectives (DQO).** The principal objective is to ensure the accuracy of the data at the actual emissions levels and in the actual emissions matrix encountered. To meet this objective, method performance tests are required and NIST-traceable calibration standards must be used.

## 2.0 Summary of Method

2.1 **Gaseous and/or aerosol isocyanates** are withdrawn from an emission source at an isokinetic sampling rate and are collected in a multicomponent sampling train. The primary components of the train include a heated probe, three impingers containing derivatizing reagent in toluene, an empty impinger, an impinger containing charcoal, and an impinger containing silica gel.

2.2 The liquid impinger contents are recovered, concentrated to dryness under vacuum, brought to volume with acetonitrile (ACN) and analyzed with a high pressure liquid chromatograph (HPLC).

## 3.0 Definitions [Reserved]

## 4.0 Interferences

4.1 The greatest potential for interference comes from an impurity in the derivatizing reagent, 1-(2-pyridyl)piperazine (1,2-PP). This compound may interfere with the resolution of MI from the peak attributed to unreacted 1,2-PP.

4.2 Other interferences that could result in positive or negative bias are (1) alcohols that could compete with the 1,2-PP for reaction with an isocyanate and (2) other compounds that may co-elute with one or more of the derivatized isocyanates.

4.3 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware. All these materials must be routinely shown to be free from interferences under conditions of the analysis by preparing and analyzing laboratory method (or reagent) blanks.

4.3.1 Glassware must be cleaned thoroughly before using. The glassware should be washed with laboratory detergent in hot water followed by rinsing with tap water and distilled water. The glassware may be dried by baking in a glassware oven at 400 °C for at least one hour. After the glassware has cooled, it should be rinsed three times with methylene chloride and three times with acetonitrile. Volumetric glassware should not be heated to 400 °C. Instead, after washing and rinsing, volumetric glassware may be rinsed with acetonitrile followed by methylene chloride and allowed to dry in air.

4.3.2 The use of high purity reagents and solvents helps to reduce interference problems in sample analysis.

## 5.0 Safety

5.1 Organizations performing this method are responsible for maintaining a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding safe handling of the chemicals specified in this method. A reference file of material safety data sheets should also be made available to all personnel involved in performing the method. Additional references to laboratory safety are available.

## 6.0 Equipment and Supplies

6.1 **Sample Collection.** A schematic of the sampling train used in this method is shown in Figure 207-1. This sampling train configuration is adapted from Method 5 procedures, and, as such, most of the required equipment is identical to that used in Method 5 determinations. The only new component required is a condenser.

6.1.1 **Probe Nozzle.** Borosilicate or quartz glass; constructed and calibrated according to Method 5, sections 6.1.1.1 and 10.1, and coupled to the probe liner using a Teflon union; a stainless steel nut is recommended for this union. When the stack temperature exceeds 210 °C (410 °F), a one-piece glass nozzle/liner assembly must be used.

6.1.2 **Probe Liner.** Same as Method 5, section 6.1.1.2, except metal liners shall not be used. Water-cooling of the stainless steel sheath is recommended at temperatures exceeding 500 °C (932 °F). Teflon may be used in limited applications where the minimum stack temperature exceeds 120 °C (250 °F) but never exceeds the temperature where Teflon is estimated to become unstable (approximately 210 °C (410 °F)).

6.1.3 **Pitot Tube, Differential Pressure Gauge, Filter Heating System, Metering System, Barometer, Gas Density Determination Equipment.** Same as Method 5, sections 6.1.1.3, 6.1.1.4, 6.1.1.6, 6.1.1.9, 6.1.2, and 6.1.3.

6.1.4 **Impinger Train.** Glass impingers are connected in series with leak-free ground-glass joints following immediately after the heated probe. The first impinger shall be of the Greenburg-Smith design with the standard tip. The remaining five impingers shall be of the modified Greenburg-Smith design, modified by replacing the tip with a 1.3-cm (½-in.) I.D. glass tube extending about 1.3 cm (½ in.) from the bottom of the outer cylinder. A water-jacketed condenser is placed between the outlet of the first impinger and the inlet to the second impinger to reduce the evaporation of toluene from the first impinger.

6.1.5 **Moisture Measurement.** For the purpose of calculating volumetric flow rate and isokinetic sampling, you must also collect either Method 4 in Appendix A-3 to this part or other moisture measurement methods approved by the Administrator concurrent with each Method 326 test run.

## 6.2 Sample Recovery

6.2.1 **Probe and Nozzle Brushes;** Polytetrafluoroethylene (PTFE) bristle brushes with stainless steel wire or PTFE handles are required. The probe brush shall have extensions constructed of stainless steel, PTFE, or inert material at least as long as the probe. The brushes shall be properly sized and shaped to brush out the probe liner and the probe nozzle.

6.2.2 **Wash Bottles.** Three. PTFE or glass wash bottles are recommended; polyethylene wash bottles must not be used because organic contaminants may be extracted by exposure to organic solvents used for sample recovery.

6.2.3 **Glass Sample Storage Containers.** Chemically resistant, borosilicate amber glass bottles, 500-mL or 1,000-mL. Bottles should be tinted to prevent the action of light on the sample. Screw-cap liners shall be either PTFE or constructed to be leak-free and resistant to chemical attack by organic recovery solvents. Narrow-mouth glass bottles have been found to leak less frequently.

6.2.4 **Graduated Cylinder.** To measure impinger contents to the nearest 1 ml or 1 g. Graduated cylinders shall have subdivisions not >2 mL.

6.2.5 **Plastic Storage Containers.** Screw-cap polypropylene or polyethylene containers to store silica gel and charcoal.

6.2.6 **Funnel and Rubber Policeman.** To aid in transfer of silica gel or charcoal to container (not necessary if silica gel is weighed in field).

6.2.7 **Funnels.** Glass, to aid in sample recovery.

6.3 **Sample Preparation and Analysis.** The following items are required for sample analysis.

6.3.1 **Rotary Evaporator.** Buchii Model EL-130 or equivalent.

6.3.2 **1000 ml Round Bottom Flask** for use with a rotary evaporator.

6.3.3 **Separatory Funnel.** 500-ml or larger, with PTFE stopcock.

6.3.4 **Glass Funnel.** Short-stemmed or equivalent.

6.3.5 **Vials.** 15-ml capacity with PTFE lined caps.

6.3.6 **Class A Volumetric Flasks.** 10-ml for bringing samples to volume after concentration.

6.3.7 **Filter Paper.** Qualitative grade or equivalent.

6.3.8 **Buchner Funnel.** Porcelain with 100 mm ID or equivalent.

6.3.9 **Erlenmeyer Flask.** 500-ml with side arm and vacuum source.

6.3.10 **HPLC** with at least a binary pumping system capable of a programmed gradient.

6.3.11 **Column Systems** Column systems used to measure isocyanates must be capable of achieving separation of the target compounds from the nearest eluting compound or interferents with no more than 10 percent peak overlap.

6.3.12 **Detector.** UV detector at 254 nm. A fluorescence detector (FD) with an excitation of 240 nm and an emission at 370 nm may be also used to allow the detection of low concentrations of isocyanates in samples.

6.3.13 **Data system** for measuring peak areas and retention times.

## 7.0 Reagents and Standards

7.1 **Sample Collection Reagents.**

7.1.1 **Charcoal.** Activated, 6-16 mesh. Used to absorb toluene vapors and prevent them from entering the metering device. Use once with each train and discard.

7.1.2 **Silica Gel and Crushed Ice.** Same as Method 5, sections 7.1.2 and 7.1.4 respectively

7.1.3 Impinger Solution. The impinger solution is prepared by mixing a known amount of 1-(2-pyridyl) piperazine (purity 99.5+%) in toluene (HPLC grade or equivalent). The actual concentration of 1,2-PP should be approximately four times the amount needed to ensure that the capacity of the derivatizing solution is not exceeded. This amount shall be calculated from the stoichiometric relationship between 1,2-PP and the isocyanate of interest and preliminary information about the concentration of the isocyanate in the stack emissions. A concentration of 130 µg/ml of 1,2-PP in toluene can be used as a reference point. This solution shall be prepared, stored in a refrigerated area away from light, and used within ten days of preparation.

#### 7.2 Sample Recovery Reagents.

7.2.1 Toluene. HPLC grade is required for sample recovery and cleanup (see Note to 7.2.2 below).

7.2.2 Acetonitrile. HPLC grade is required for sample recovery and cleanup. **Note:** Organic solvents stored in metal containers may have a high residue blank and should not be used. Sometimes suppliers transfer solvents from metal to glass bottles; thus blanks shall be run before field use and only solvents with a low blank value should be used.

7.3 Analysis Reagents. Reagent grade chemicals should be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

7.3.1 Toluene,  $C_6H_5CH_3$ . HPLC Grade or equivalent.

7.3.2 Acetonitrile,  $CH_3CN$  (ACN). HPLC Grade or equivalent.

7.3.3 Methylene Chloride,  $CH_2Cl_2$ . HPLC Grade or equivalent.

7.3.4 Hexane,  $C_6H_{14}$ . HPLC Grade or equivalent.

7.3.5 Water,  $H_2O$ . HPLC Grade or equivalent.

7.3.6 Ammonium Acetate,  $CH_3CO_2NH_4$ .

7.3.7 Acetic Acid (glacial),  $CH_3CO_2H$ .

7.3.8 1-(2-Pyridyl)piperazine, (1,2-PP), ≥99.5% or equivalent.

7.3.9 Absorption Solution. Prepare a solution of 1-(2-pyridyl)piperazine in toluene at a concentration of 40 mg/300 ml. This solution is used for method blanks and method spikes.

7.3.10 Ammonium Acetate Buffer Solution (AAB). Prepare a solution of ammonium acetate in water at a concentration of 0.1 M by transferring 7.705 g of ammonium acetate to a 1,000 ml volumetric flask and diluting to volume with HPLC Grade water. Adjust pH to 6.2 with glacial acetic acid.

#### 8.0 Sample Collection, Storage and Transport

**Note:** Because of the complexity of this method, field personnel should be trained in and experienced with the test procedures in order to obtain reliable results.

##### 8.1 Sampling

8.1.1 Preliminary Field Determinations. Same as Method 5, section 8.2.

8.1.2 Preparation of Sampling Train. Follow the general procedure given in

Method 5, section 8.3.1, except for the following variations: Place 300 ml of the impinger absorbing solution in the first impinger and 200 ml each in the second and third impingers. The fourth impinger shall remain empty. The fifth and sixth impingers shall have 400 g of charcoal and 200–300 g of silica gel, respectively. Alternatively, the charcoal and silica gel may be combined in the fifth impinger. Set-up the train as in Figure 326–1. During assembly, do not use any silicone grease on ground-glass joints.

**Note:** During preparation and assembly of the sampling train, keep all openings where contamination can occur covered with PTFE film or aluminum foil until just before assembly or until sampling is about to begin.

8.1.3 Leak-Check Procedures. Follow the leak-check procedures given in Method 5, sections 8.4.2 (Pretest Leak-Check), 8.4.3 (Leak-Checks During the Sample Run), and 8.4.4 (Post-Test Leak-Check), with the exception that the pre-test leak-check is mandatory.

8.1.4 Sampling Train Operation. Follow the general procedures given in Method 5, section 8.5. Turn on the condenser coil coolant recirculating pump and monitor the gas entry temperature. Ensure proper gas entry temperature before proceeding and again before any sampling is initiated. It is important that the gas entry temperature not exceed 50 °C (122 °F), thus reducing the loss of toluene from the first impinger. For each run, record the data required on a data sheet such as the one shown in Method 5, Figure 5–3.

8.2 Sample Recovery. Allow the probe to cool. When the probe can be handled safely, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over the tip to prevent losing or gaining particulate matter. Do not cap the probe tip tightly while the sampling train is cooling down because this will create a vacuum in the train. Before moving the sample train to the cleanup site, remove the probe from the sample train and cap the opening to the probe, being careful not to lose any condensate that might be present. Cap the impingers and transfer the probe and the impinger/condenser assembly to the cleanup area. This area should be clean and protected from the weather to reduce sample contamination or loss. Inspect the train prior to and during disassembly and record any abnormal conditions. It is not necessary to measure the volume of the impingers for the purpose of moisture determination as the method is not validated for moisture determination. Treat samples as follows:

8.2.1 Container No. 1. Probe and Impinger Numbers 1 and 2. Rinse and brush the probe/nozzle first with toluene twice and then twice again with acetonitrile and place the wash into a glass container labeled with the test run identification and "Container No. 1." When using these solvents ensure that proper ventilation is available. Quantitatively transfer the liquid from the first two impingers and the condenser into Container No. 1. Rinse the impingers and all connecting glassware twice with toluene and then twice again with acetonitrile and transfer the rinses into Container No. 1. After all components have been collected in the container, seal the

container, and mark the liquid level on the bottle.

8.2.2 Container No. 2, Impingers 3 and 4. Quantitatively transfer the liquid from each impinger into a glass container labeled with the test run identification and "Container No. 2." Rinse each impinger and all connecting glassware twice with toluene and twice again with acetonitrile and transfer the rinses into Container No. 2. After all components have been collected in the container, seal the container, and mark the liquid level on the bottle.

**Note:** The contents of the fifth and sixth impinger (silica gel) can be discarded.

8.2.3 Container No. 3, Reagent Blank. Save a portion of both washing solutions (toluene/acetonitrile) used for the cleanup as a blank. Transfer 200 ml of each solution directly from the wash bottle being used and combine in a glass sample container with the test identification and "Container No. 3." Seal the container, and mark the liquid level on the bottle and add the proper label.

8.2.4 Field Train Proof Blanks. To demonstrate the cleanliness of sampling train glassware, you must prepare a full sampling train to serve as a field train proof blank just as it would be prepared for sampling. At a minimum, one complete sampling train will be assembled in the field staging area, taken to the sampling area, and leak-checked. The probe of the blank train shall be heated during and the train will be recovered as if it were an actual test sample. No gaseous sample will be passed through the sampling train. Field blanks are recovered in the same manner as described in sections 8.2.1 and 8.2.2 and must be submitted with the field samples collected at each sampling site.

8.2.5 Field Train Spike. To demonstrate the effectiveness of the sampling train, field handling, and recovery procedures you must prepare a full sampling train to serve as a field train spike just as it would be prepared for sampling. The field spike is performed in the same manner as the field train proof blank with the additional step of adding the Field Spike Solution to the first impinger after the initial leak check. The train will be recovered as if it were an actual test sample. No gaseous sample will be passed through the sampling train. Field train spikes are recovered in the same manner as described in sections 8.2.1 and 8.2.2 and must be submitted with the samples collected for each test program.

8.3 Sample Transport Procedures. Containers must remain in an upright position at all times during shipment. Samples must also be stored at <4 °C between the time of sampling and concentration. Each sample should be extracted and concentrated within 30 days after collection and analyzed within 30 days after extraction. The extracted sample must be stored at 4 °C.

8.4 Sample Custody. Proper procedures and documentation for sample chain of custody are critical to ensuring data integrity. The chain of custody procedures in ASTM D4840–99 (Reapproved 2018) "Standard Guide for Sampling Chain-of-Custody Procedures" (incorporated by reference, see § 63.14) shall be followed for all samples (including field samples and blanks).

**9.0 Quality Control**

**9.1 Sampling, Sampling Operations.** The sampling quality control procedures and acceptance criteria are listed in Table 326-2 below; see also section 9.0 of Method 5.

**9.2 Analysis.** The analytical quality control procedures required for this method includes the analysis of the field train proof blank, field train spike, and reagent and method blanks. Analytical quality control

procedures and acceptance criteria are listed in Table 326-3 below.

**9.2.1 Check for Breakthrough.** Recover and determine the isocyanate(s) concentration of the last two impingers separately from the first two impingers.

**9.2.2 Field Train Proof Blank.** Field blanks must be submitted with the samples collected at each sampling site.

**9.2.3 Reagent Blank and Field Train Spike.** At least one reagent blank and a field

train spike must be submitted with the samples collected for each test program.

**9.2.4 Determination of Method Detection Limit.** Based on your instrument's sensitivity and linearity, determine the calibration concentrations or masses that make up a representative low level calibration range. The MDL must be determined at least annually for the analytical system using an MDL study such as that found in section 15.0 to Method 301 of appendix A to part 63 of this chapter.

**TABLE 326-2—SAMPLING QUALITY ASSURANCE AND QUALITY CONTROL**

QA/QC criteria	Acceptance criteria	Frequency	Consequence if not met
Sampling Equipment Leak Checks.	$\leq 0.00057$ m <sup>3</sup> /min (0.020 cfm) or 4% of sampling rate, whichever is less.	Prior to, during (optional) and at the completion to sampling.	Prior to: Repair and repeat calibration. During/Completion: None, testing should be considered invalid.
Dry Gas Meter Calibration—Pre-Test (individual correction factor— $Y_i$ ).	within $\pm 2\%$ of average factor (individual).	Pre-test .....	Repeat calibration point.
Dry Gas Meter Calibration—Pre-Test (average correction factor— $Y_c$ ).	$1.00 \pm 1\%$ .....	Pre-test .....	Adjust the dry gas meter and recalibrate.
Dry Gas Meter Calibration—Post-test.	Average dry gas meter calibration factor agrees with $\pm 5\% Y_c$ .	Each Test .....	Adjust sample volumes using the factor that gives the smallest volume.
Temperature sensor calibration.	Absolute temperature measures by sensor within $\pm 1.5\%$ of a reference sensor.	Prior to initial use and before each test thereafter.	Recalibrate; sensor may not be used until specification is met.
Barometer calibration .....	Absolute pressure measured by instrument within $\pm 10$ mm Hg of reading with a mercury barometer or NIST traceable barometer.	Prior to initial use and before each test thereafter.	Recalibrate; instrument may not be used until specification is met.

**TABLE 326-3—ANALYTICAL QUALITY ASSURANCE AND QUALITY CONTROL**

QA/QC criteria	Acceptance criteria	Frequency	Consequence if not met
Calibration—Method Blanks ...	$< 5\%$ level of expected analyte .....	Each analytical method blank	Locate source of contamination; reanalyze.
Calibration—Calibration Points	At least six calibration point bracketing the expected range of analysis.	Each analytical batch .....	Incorporate additional calibration points to meet criteria.
Calibration—Linearity .....	Correlation coefficient $> 0.995$ .....	Each analytical batch .....	Verify integration, reintegrate. If necessary, recalibrate.
Calibration—secondary standard verification.	Within $\pm 10\%$ of true value .....	After each calibration .....	Repeat secondary standard verification, recalibrate if necessary.
Calibration—continual calibration verification.	Within $\pm 10\%$ of true value .....	Daily and after every ten samples.	Invalidate previous ten sample analysis, recalibrate and repeat calibration, reanalyze samples until successful.
Sample Analysis .....	Within the valid calibration range .....	Each sample .....	Invalidate the sample if greater than the calibration range and dilute the sample so that it is within the calibration range. Appropriately flag any value below the calibration range.
Replicate Samples .....	Within $\pm 10\%$ of RPD .....	Each sample .....	Evaluate integrations and repeat sample analysis as necessary.
Field Train Proof Blank .....	$\leq 10\%$ level of expected analyte .....	Each test program .....	Evaluate source of contamination.
Field Train Spike .....	Within $\pm 30\%$ of true value .....	Each test program .....	Evaluate performance of the method and consider invalidating results.
Breakthrough .....	Final two impingers Mass collected is $> 5\%$ of the total mass or $> 20\%$ of the total mass when the measured results are 20% of the applicable standard. Alternatively, there is no breakthrough requirement when the measured results are 10% of the applicable standard.	Each test run .....	Invalidate test run.

## 10.0 Calibration and Standardization

**Note:** Maintain a laboratory log of all calibrations.

10.1 Probe Nozzle, Pitot Tube Assembly, Dry Gas Metering System, Probe Heater, Temperature Sensors, Leak-Check of Metering System, and Barometer. Same as Method 5, sections 10.1, 10.2, 10.3, 10.4, 10.5, 8.4.1, and 10.6, respectively.

10.2 High Performance Liquid Chromatograph. Establish the retention times for the isocyanates of interest; retention times will depend on the chromatographic conditions. The retention times provided in Table 10-1 are provided as a guide to relative retention times when using a C18, 250 mm x 4.6 mm ID, 5µm particle size column, a 2 ml/min flow rate of a 1:9 to 6:4 Acetonitrile/Ammonium Acetate Buffer, a 50 µl sample loop, and a UV detector set at 254 nm.

TABLE 326-4—EXAMPLE RETENTION TIMES

Retention times	
Compound	Retention time (minutes)
MI .....	10.0
1,6-HDI .....	19.9
2,4-TDI .....	27.1
MDI .....	27.3

## 10.3 Preparation of Isocyanate Derivatives.

10.3.1 HDI, TDI, MDI. Dissolve 500 mg of each isocyanate in individual 100 ml aliquots of methylene chloride (MeCl<sub>2</sub>), except MDI which requires 250 ml of MeCl<sub>2</sub>. Transfer a 5-ml aliquot of 1,2-PP (see section 7.3.8) to each solution, stir and allow to stand overnight at room temperature. Transfer 150 ml aliquots of hexane to each solution to precipitate the isocyanate-urea derivative. Using a Buchner funnel, vacuum filter the solid-isocyanate-urea derivative and rinse with 50 ml of hexane. Dissolve the precipitate in a minimum aliquot of MeCl<sub>2</sub>. Repeat the hexane precipitation and filtration twice. After the third filtration, dry the crystals at 50 °C and transfer to bottles for storage. The crystals are stable for at least 21 months when stored at room temperature in a closed container.

10.3.2 MI. Prepare a 200 µg/ml stock solution of methyl isocyanate-urea, transfer 60 mg of 1,2-PP to a 100-ml volumetric flask containing 50 ml of MeCl<sub>2</sub>. Carefully transfer 20 mg of methyl isocyanate to the volumetric flask and shake for 2 minutes. Dilute the solution to volume with MeCl<sub>2</sub> and transfer to a bottle for storage. Methyl isocyanate does not produce a solid derivative and standards must be prepared from this stock solution.

10.4 Preparation of calibration standards. Prepare a 100 µg/ml stock solution of the isocyanates of interest from the individual isocyanate-urea derivative as prepared in sections 10.3.1 and 10.3.2. This is accomplished by dissolving 1 mg of each isocyanate-urea derivative in 10 ml of Acetonitrile. Calibration standards are prepared from this stock solution by making

appropriate dilutions of aliquots of the stock into Acetonitrile.

10.5 Preparation of Method Blanks. Prepare a method blank for each test program (up to twenty samples) by transferring 300 ml of the absorption solution to a 1,000-ml round bottom flask and concentrate as outlined in section 11.2.

10.6 Preparation of Field Spike Solution. Prepare a field spike solution for every test program in the same manner as calibration standards (see Section 10.4). The mass of the target isocyanate in the volume of the spike solution for the field spike train shall be equivalent to that estimated to be captured from the source concentration for each compound; alternatively, you may also prepare a solution that represents half the applicable standard.

10.7 HPLC Calibrations. See Section 11.1.

## 11.0 Analytical Procedure

11.1 Analytical Calibration. Perform a multipoint calibration of the instrument at six or more upscale points over the desired quantitative range (multiple calibration ranges shall be calibrated, if necessary). The field samples analyzed must fall within at least one of the calibrated quantitative ranges and meet the performance criteria specified below. The lowest point in your calibration curve must be at least 5, and preferably 10, times the MDL. For each calibration curve, the value of the square of the linear correlation coefficient, *i.e.*, *r*<sup>2</sup>, must be ≥0.995, and the analyzer response must be within ±10 percent of the reference value at each upscale calibration point. Calibrations must be performed on each day of the analysis, before analyzing any of the samples. Following calibration, a secondary standard shall be analyzed. A continual calibration verification (CCV) must also be performed prior to any sample and after every ten samples. The measured value of this independently prepared standard must be within ±10 percent of the expected value. Report the results for each calibration standard secondary standard, and CCV as well as the conditions of the HPLC. The reports should include at least the peak area, height, and retention time for each isocyanate compound measured as well as a chromatogram for each standard.

11.2 Concentration of Samples. Transfer each sample to a 1,000-ml round bottom flask. Attach the flask to a rotary evaporator and gently evaporate to dryness under vacuum in a 65 °C water bath. Rinse the round bottom flask three times each with 2 ml of acetonitrile and transfer the rinse to a 10-ml volumetric flask. Dilute the sample to volume with acetonitrile and transfer to a 15-ml vial and seal with a PTFE lined lid. Store the vial ≤4 °C until analysis.

11.3 Analysis. Analyze replicative samples by HPLC, using the appropriate conditions established in section 10.2. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst

should weigh heavily in the interpretation of the chromatograms. If the peak area exceeds the linear range of the calibration curve, the sample must be diluted with acetonitrile and reanalyzed. Average the replicate results for each run. For each sample you must report the same information required for analytical calibrations (Section 11.1). For non-detect or values below the detection limit of the method, you shall report the value as “<” numerical detection limit.

## 12.0 Data Analysis and Calculations

Nomenclature and calculations, same as in Method 5, section 6, with the following additions below.

## 12.1 Nomenclature.

AS = Response of the sample, area counts.  
b = Y-intercept of the linear regression line, area counts.  
BR = Percent Breakthrough  
C<sub>A</sub> = Concentration of a specific isocyanate compound in the initial sample, µg/ml.  
C<sub>B</sub> = Concentration of a specific isocyanate compound in the replicate sample, µg/ml.  
C<sub>i</sub> = Concentration of a specific isocyanate compound in the sample, µg/ml.  
C<sub>rec</sub> = Concentration recovered from spike train, µg/ml.  
C<sub>S</sub> = Concentration of isocyanate compound in the stack gas, µg/dscm  
C<sub>T</sub> = Concentration of a specific isocyanate compound (Impingers 1-4), µg/dscm  
C<sub>spike</sub> = Concentration spiked, µg/ml.  
C<sub>i</sub> = Concentration of a specific isocyanate compound (Impingers 14), µg/dscm  
FL<sub>m</sub> = Mass of Free Isocyanate  
FTS<sub>rec</sub> = Field Train Spike Recovery  
I<sub>m</sub> = Mass of the Isocyanate  
I<sub>mw</sub> = MW of the Isocyanate  
IU<sub>m</sub> = Mass of Isocyanate-urea derivative  
IU<sub>mw</sub> = MW of the isocyanate-urea  
M = Slope of the linear regression line, area counts-ml/µg.  
m<sub>i</sub> = Mass of isocyanate in the total sample  
MW = Molecular weight  
RPD = Relative Percent Difference  
VF = Final volume of concentrated sample, typically 10 ml.  
V<sub>m,ad</sub> = Volume of gas sample measured by the dry-gas meter, corrected to standard conditions, dscm (dscf).  
Conversion from Isocyanate to the Isocyanate-urea derivative. The equation for converting the amount of free isocyanate to the corresponding amount of isocyanate-urea derivative is as follows:

12.2 Conversion from Isocyanate to the Isocyanate-urea derivative. The equation for converting the amount of free isocyanate to the corresponding amount of isocyanate-urea derivative is as follows:

$$IU_m = I_m \frac{IU_{mw}}{I_{mw}} \quad \text{Eq. 326-1}$$

The equation for converting the amount of IU derivative to the corresponding amount of FL<sub>m</sub> is as follows:

$$I_m = IU_m \frac{I_{mw}}{IU_{mw}} \quad \text{Eq. 326-2}$$

12.3 Calculate the correlation coefficient, slope, and intercepts for the calibration data



using the least squares method for linear regression. Concentrations are expressed as the x-variable and response is expressed as the y-variable.

12.4 Calculate the concentration of isocyanate in the sample:

$$C_I = \frac{A_s - b}{M} \quad \text{Eq. 326-3}$$

12.5 Calculate the total amount collected in the sample by multiplying the concentration ( $\mu\text{g}/\text{ml}$ ) times the final volume of acetonitrile (10 ml).

$$m_I = C_I \times V_f \quad \text{Eq. 326-4}$$

12.6 Calculate the concentration of isocyanate ( $\mu\text{g}/\text{dscm}$ ) in the stack gas.

$$C_s = \frac{M_I}{V_{m\text{std}}} K \quad \text{Eq. 326-5}$$

12.7 Calculate Relative Percent Difference (RPD) for each replicative sample

$$\%RPD = \left| \frac{(C_A - C_B)}{(C_A + C_B)/2} \right| \times 100$$

Eq. 326-6

12.8 Calculate Field Train Spike Recovery

$$FTS_{\text{rec}} = \left[ \frac{C_{\text{rec}}}{C_{\text{spike}}} \right] \times 100$$

Eq. 326-7

12.9 Calculate Percent Breakthrough

$$BR = \left[ \frac{C_4}{C_7} \right] \times 100 \quad \text{Eq. 326-8}$$

Where:

$K = 35.314 \text{ ft}^3/\text{m}^3$  if  $V_{m(\text{std})}$  is expressed in English units.  $= 1.00 \text{ m}^3/\text{m}^3$  if  $V_{m(\text{std})}$  is expressed in metric units.

#### 13.0 Method Performance

Evaluation of sampling and analytical procedures for a selected series of compounds must meet the quality control criteria (See Section 9) for each associated analytical determination. The sampling and analytical procedures must be challenged by the test compounds spiked at appropriate levels and carried through the procedures.

#### 14.0 Pollution Prevention [Reserved]

#### 15.0 Waste Management [Reserved]

#### 16.0 Alternative Procedures [Reserved]

#### 17.0 References

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#### 18.0 Diagrams

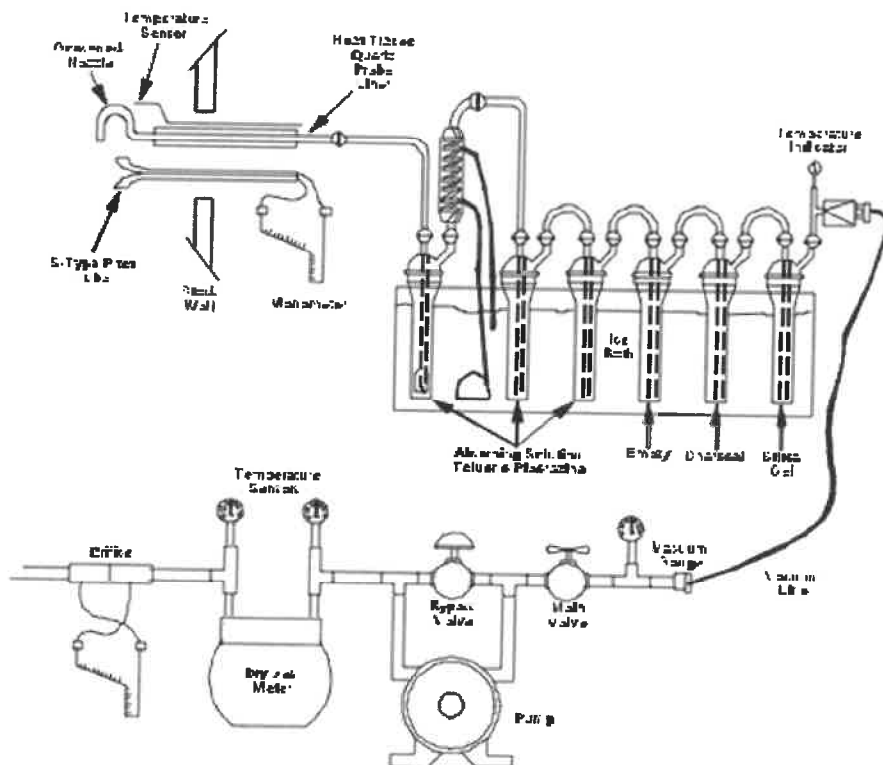


Figure 326-1—Method 326 Sampling Train

