ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 63

[FRL–6347–2]

RIN 2060–AE78

National Emission Standards for Hazardous Air Pollutants for Source Categories; Portland Cement Manufacturing Industry

AGENCY: Environmental Protection Agency (EPA).

ACTION: Final rule.

SUMMARY: This action promulgates national emission standards for hazardous air pollutants (NESHAP) for new and existing sources in the portland cement manufacturing industry. This action also adds Method 320 for the measurement of vapor phase organic and inorganic emissions by extractive Fourier Transform Infrared (FTIR) spectroscopy and Method 321 for the measurement of gaseous hydrogen chloride emissions from portland cement kilns by FTIR spectroscopy to appendix A of part 63.

Some of the hazardous air pollutants (HAPs) released from portland cement manufacturing facilities include, but are not limited to, acetaldehyde, arsenic, benzene, cadmium, chromium, chlorobenzene, dibenzofurans, formaldehyde, hexane, hydrogen chloride, lead, manganese, mercury, naphthalene, nickel, phenol, polycyclic organic matter, selenium, styrene, 2,3,7,8-tetrachlorodibenzo-p-dioxin, toluene, and xylenes. Exposure to these

HAPs can cause reversible or irreversible health effects including carcinogenic, respiratory, nervous system, developmental, reproductive and/or dermal health effects. The EPA estimates that this final rule will reduce nationwide emissions of HAPs from portland cement manufacturing facilities by approximately 82 megagrams per year (Mg/yr) [90 tons per year (tpy)], and particulate matter (PM) by approximately 4,700 Mg/yr (5,200 tpy).

These standards implement section 112(d) of the Clean Air Act (CAA) and are based on the Administrator's determination that portland cement manufacturing facilities may reasonably be anticipated to emit several of the 188 HAPs listed in section 112(b) of the CAA from the various process operations found within the industry. The final rule provides protection to the public by requiring portland cement manufacturing plants to meet emission standards reflecting the application of the maximum achievable control technology (MACT).

EFFECTIVE DATE: June 14, 1999. See the **SUPPLEMENTARY INFORMATION** section concerning judicial review.

ADDRESSES: *Docket.* Docket No. A–92–53, containing information considered by the EPA in development of the promulgated standards, is available for public inspection between 8:00 a.m. to 5:30 p.m., Monday through Friday, except Federal holidays, at the following address: U.S. Environmental Protection Agency, Air and Radiation Docket and Information Center (6102),

401 M Street S.W., Washington, DC 20460, telephone number (202) 260–7548. The docket is located at the above address in room M–1500, Waterside Mall (ground floor). A reasonable fee may be charged for copying docket materials.

FOR FURTHER INFORMATION CONTACT: For further information concerning applicability and rule determinations, contact the appropriate State or local agency representative. If no State or local representative is available, contact the EPA Regional Office staff listed in the Supplementary Information section of this preamble. For information concerning the analyses performed in developing this rule, contact Mr. Joseph Wood, P. E., Minerals and Inorganic Chemicals Group, Emission Standards Division (MD–13), Office of Air Quality Planning and Standards, U.S. EPA, Research Triangle Park, North Carolina 27711, telephone number (919) 541–5446, facsimile number (919) 541–5600, electronic mail address ''wood.joe@epamail.epa.gov''. For information regarding Methods 320 and 321 contact Ms. Rima Dishakjian, Emission Measurement Center, Emissions, Monitoring and Analysis Division (MD–19), U.S. Environmental Protection Agency, Research Triangle Park, NC 27711, telephone number (919) 541–0443.

SUPPLEMENTARY INFORMATION:

Regulated entities. Entities potentially regulated by this action are those that manufacture portland cement. Regulated categories and entities shown in Table 1.

TABLE 1.—REGULATED ENTITIES

Category	NAICS Code	SIC Code	Examples of Regulated Entities
Industry	32731	3241	Owners or operators of portland cement manufacturing plants.
State	32731	3241	Owners or operators of portland cement manufacturing plants.
Tribal associations	32731	3241	Owners or operators of portland cement manufacturing plants.
Federal agencies	(1)	(1)	None.

[1] None.

This table is not intended to be exhaustive, but rather provides a guide for readers regarding entities likely to be regulated by this action. This table lists the types of entities that the EPA is now aware could potentially be regulated by this action. To determine whether your facility, company, business organization, etc. is regulated by this action, you should carefully examine the applicability criteria in § 63.1340 of the rule. If you have questions regarding the applicability of this action to a

particular entity, consult the appropriate regional representative:

Region 1—Janet Bowen, Office of Ecosystem Protection, U.S. EPA, Region I, CAP, JFK Federal Building, Boston, MA 02203, (617) 565–3595.

Region II—Kenneth Eng, Air Compliance Branch Chief, U.S. EPA, Region II, 290 Broadway, New York, NY 10007–1866 (212) 637–4000.

Region III—Bernard Turlinski, Air Enforcement Branch Chief, U.S. EPA, Region III (3AT10), 841 Chestnut

Building, Philadelphia, PA 19107, (215) 566–2110.

Region IV—Lee Page, Air Enforcement Branch, U.S. EPA, Region IV, Atlanta Federal Center, 61 Forsyth Street, Atlanta, GA 30303–3104, (404) 562–9131.

Region V—George T. Czerniak, Jr., Air Enforcement Branch Chief, U.S. EPA, Region V (5AE–26), 77 West Jackson Street, Chicago, IL 60604, (312) 353–2088.

Region VI—John R. Hepola, Air Enforcement Branch Chief, U.S. EPA,

Region VI, 1445 Ross Avenue, Suite 1200, Dallas, TX 75202–2733, (214) 665–7220.

Region VII—Donald Toensing, Chief, Air Permitting and Compliance Branch, U.S. EPA, Region VII, 726 Minnesota Avenue, Kansas City, KS 66101, (913) 551–7446.

Region VIII—Douglas M. Skie, Air and Technical Operations Branch Chief, U.S. EPA, Region VIII, 999 18th Street, Suite 500, Denver, CO 80202–2466, (303) 312–6432.

Region IX—Barbara Gross, Air Compliance Branch Chief, U.S. EPA, Region IX, 75 Hawthorne Street, San Francisco, CA 94105, (415) 744–1138.

Region X—Anita Frankel, Air and Radiation Branch Chief, U.S. EPA, Region X (AT–092), 1200 Sixth Avenue, Seattle, WA 98101–1128, (206) 553–1757.

Judicial Review. The NESHAP for portland cement manufacturing was proposed on March 24, 1998 (63 FR 14182). Today's **Federal Register** action announces the EPA's final decision on the rule. Under section 307(b)(1) of the Act, judicial review of the final rule is available by filing a petition for review in the U.S. Court of Appeals for the District of Columbia Circuit within 60 days of today's publication of this final rule. Under section 307(b)(2) of the Act, the requirements that are the subject of today's notice may not be challenged later in civil or criminal proceedings brought by the EPA to enforce these requirements.

Technology Transfer Network. In addition to being available in the docket, an electronic copy of today's document, which includes the regulatory text, is available through the Technology Transfer Network (TTN) at the Office of Air and Radiation Policy and Guidance website. Following promulgation, a copy of the rule will be posted at the TTN's policy and guidance page for newly proposed or promulgated rules (http://www.epa.gov/ttn/oarpg/t3pfpr.html). A copy of the Response to Comments document for this rule will be posted on the TTN at http://www.epa.gov/ttn/oarpg/t3bid.html. The TTN provides information from EPA in various areas of air pollution technology or policy. If more information on the TTN is needed, call the TTN help line at (919) 541–5384.

Outline. The following outline is provided to aid in reading this preamble to the final rule.

I. Statutory Authority

The statutory authority for this rule is provided by sections 101, 112, 113, 114, 116, and 301 of the Clean Air Act, as amended (42 U.S.C. 7401, 7412, 7413, 7414, 7416, and 7601). This rule is also subject to section 307(d) of the CAA (42 U.S.C. 7407(d)).

II. Background and Public Participation

The Clean Air Act was created in part "to protect and enhance the quality of the Nation's air resources so as to promote the public health and welfare and the productive capacity of its population." (Clean Air Act, section 101(b)(1)) Section 112(b), as revised in 61 FR 30816 (June 18, 1996), lists 188 HAPs believed to cause adverse health or environmental effects. Section 112(d) requires that emission standards be promulgated for all categories and subcategories of "major" sources of these HAP and for "area" sources listed for regulation, pursuant to section 112(c). Major sources are defined as those that emit or have the potential to emit (from all emission points in all source categories within the facility) at least 10 tons per year of any single HAP or 25 tons per year of any combination of HAP. Area sources are stationary sources of HAP that are not major sources.

On July 16, 1992 (57 FR 31576), the EPA published a list of categories of sources slated for regulation. This list included the portland cement source category regulated by the standards being promulgated today. The statute requires emissions standards for the listed source categories to be promulgated between November 1992 and November 2000. On June 4, 1996, the EPA published a schedule for promulgating these standards (61 FR 28197). Standards for the portland cement manufacturing source category covered by this rule were proposed on March 24, 1998 (63 FR 14182).

As in the proposal, the final standards give existing sources 3 years from the date of promulgation to comply. New sources are required to comply with the standard upon initial startup. The EPA believes these standards to be achievable for affected sources within the time provided.

Operating limits, methods for determining initial compliance, as well as monitoring, recordkeeping, and reporting requirements are included in the final rule. All of these components are necessary to ensure that sources will comply with the standards both initially and over time. However, the EPA has made every effort to simplify the requirements in the rule.

The amended Clean Air Act requires the EPA to promulgate national emission standards for sources of HAPs. Section 112(d) provides that these standards must reflect:

"* * * the maximum degree of reduction in emissions of the HAP * * * that the Administrator, taking into consideration the cost of achieving such emission reduction, and any nonair quality health and environmental impacts and energy requirements, determines is achievable for new or existing sources in the category or subcategory to which such emission standard applies * * *" [42 U.S.C. 7412(d)(2)].

This level of control is referred to as MACT. The Clean Air Act goes on to establish the least stringent level of control for MACT; this level is termed the "MACT floor."

For new sources, the standards for a source category or subcategory "shall not be less stringent than the emission control that is achieved in practice by the best controlled similar source, as determined by the Administrator" [section 112(d)(3)]. Existing source

standards shall be no less stringent than the average emission limitation achieved by the best performing 12 percent of the existing sources for source categories and subcategories with 30 or more sources, or the average emission limitation achieved by the best performing 5 sources for sources or subcategories with fewer than 30 sources [section 112(d)(3)]. These two minimum levels of control define the MACT floor for new and existing sources.

The standards were proposed in the **Federal Register** on March 24, 1998 (63 FR 14182). The preamble for the proposed standards described the rationale for the proposed standards. Public comments were solicited at the time of proposal. To provide interested individuals the opportunity for oral presentation of data, views, or arguments concerning the proposed standards, a public hearing was offered at proposal. However, the public did not request a hearing and, therefore, one was not held. The public comment period, which was extended by thirty days in response to requests from commenters, was from March 24, 1998 to June 26, 1998. A total of 28 comment letters were received. Commenters included industry representatives, State and local agencies, and environmental groups. Today's final rule reflects the EPA's full consideration of all of the comments. These public comments along with the EPA's responses to comments on the proposed rule are summarized in this preamble. A more detailed discussion of public comments and the EPA's responses can be found in the Response to Comment Document (Docket No. A–92–53, Item V–C–1).

III. Summary of Final Rule

A. Applicability

The standards apply to each portland cement manufacturing plant at any facility which is a major source or an area source, with the following exception. Some portland cement plants fire hazardous wastes in the kiln to provide part or all of the fuel requirement for clinker production. Portland cement kilns and in-line kiln/raw mills subject to the NESHAP for hazardous waste combustors (HWC), 40 CFR 63, subpart EEE, are not subject to this standard; however other affected sources at portland cement plants where hazardous waste is burned in the kiln

are subject to this standard. HW kilns and HW in-line kiln/raw mills that temporarily or permanently stop burning hazardous waste may be subject to the emission standards, notification, testing, and monitoring requirements of today's rule, as provided by subpart EEE of this part.

Except for hazardous waste burning (HW) cement kilns and HW in-line kiln/ raw mills, these standards apply to all cement kilns and in-line kiln/raw mills regardless of the material being combusted in the kiln. Currently, cement kilns which combust municipal solid waste, medical waste, or other waste materials (other than HW) are subject to today's rule. Since these devices currently are not subject to section 129 standards, EPA is including them in this rule to avoid a situation where they aren't regulated at all. This measure, however, is potentially an interim step. EPA could determine that cement kilns combusting solid waste materials should be regulated under section 129 of the Clean Air Act, 42 U.S.C. § 7429, and if so, EPA would revise the applicability section of these regulations accordingly at the time section 129 regulations applicable to cement kilns are promulgated.

EPA also considered but rejected the possibility of subcategorizing cement kilns based on the nature of feed preparation for the kiln. As discussed in the proposal preamble, there are two types of portland cement manufacturing processes differentiated on the basis of feed preparation: wet process, and dry process (which includes the long kiln dry process, preheater process, and preheater/precalciner process). The wet process kilns and all variations of the dry process kilns use the same raw materials and use the same types of air pollution controls. Therefore, if subcategories were defined based on process type, the MACT floor technology would be identical (docket item II–B–73). For this reason, the EPA is not promulgating separate rules based on process (kiln) type.

For portland cement plants with on-site non-metallic minerals processing facilities, the first affected source in the sequence of materials handling operations subject to this NESHAP is the raw material storage, which is just prior to the raw mill. The primary and secondary crushers and any other equipment in the non-metallic minerals

processing plant, which precede the raw material storage are not affected sources under this NESHAP. The first conveyor system transfer point subject to this NESHAP is the transfer point associated with the conveyor transferring material from the raw material storage to the raw mill.

This regulation does not apply to the emissions from cement kiln dust (CKD) storage facilities (e.g., CKD piles or landfills). A separate rulemaking will be forthcoming utilizing RCRA authority that will apply to air emissions associated with CKD management and disposal facilities.

B. Emission Limits and Operating Limits

In today's notice, the EPA is establishing emission limitations for particulate matter (as a surrogate for HAP metals), dioxins/furans (D/F), and total hydrocarbons (as a surrogate for organic HAPs, including polycyclic organic matter). The NESHAP for portland cement manufacturing applies to both major and area sources of HAPs. The affected sources for which emission limits are established include the non-hazardous waste (NHW) kiln, NHW in-line kiln/raw mill, clinker cooler, raw material dryer, and materials handling processes that include the raw mill, finish mill, raw material storage, clinker storage, finished product storage, conveyor transfer points, bagging and bulk loading and unloading systems (hereafter referred to as materials handling processes).

The NESHAP limits PM (surrogate for HAP metals) emissions, as well as opacity, from new and existing NHW kilns, NHW in-line kiln/raw mills, and clinker coolers, and limits opacity from raw material dryers and materials handling processes, at portland cement plants which are major sources. The rule also limits D/F emissions from new and existing NHW kilns and NHW in-line kiln/raw mills located at portland cement plants which are major or area sources of HAPs. In addition, the rule limits total hydrocarbon (THC) as a surrogate for organic HAP emissions from new greenfield NHW kilns, new greenfield NHW in-line kiln/raw mills, and new greenfield raw material dryers at portland cement plants which are major or area sources. Tables 2 and 3 present a summary of the emission limits for new and existing portland cement affected sources.

TABLE 2.—SUMMARY OF EMISSION LIMITS[a,b] FOR AFFECTED SOURCES AT PORTLAND CEMENT PLANTS

(Metric units)

Affected source and pollutant	Emission limit for existing sources	Emission limit for new sources
NHW kiln and NHW in-line kiln/raw mill[c] PM	0.15 kg/Mg dry feed[e] and opacity level[c]c no greater than 20 percent	0.15 kg/Mg dry feed[e] and opacity level[c]c no greater than 20 percent
NHW kiln and NHW in-line kiln/raw mill D/F[c,d]	0.2 ng TEQ/dscm or 0.4 ng TEQ/dscm with PM control device operated at ≤204°C[g]	0.2 ng TEQ/dscm or 0.4 ng TEQ/dscm with PM control device operated at ≤204°C[g]
NHW kiln and NHW in-line kiln/raw mill THC[d]	none	50 ppmvd[f] (as propane)
Clinker cooler PM	0.05 kg/Mg dry feed and opacity level no greater than 10 percent	0.05 kg/Mg dry feed and opacity level no greater than 10 percent
Raw material dryer and materials handling processes (raw mill system, finish mill system, raw material storage, clinker storage, finished product storage, conveyor transfer points, bagging, and bulk loading and unloading systems) PM.	10 percent opacity	10 percent opacity
Raw material dryer THC[d]	none	50 ppmvd[f] (as propane)

[a] All concentration limits at 7 percent oxygen.
[b] Applies to major sources only, except as noted.
[c] Includes main and alkali bypass stacks.
[d] Applies to both major and area source portland cement plants.
[e] If there is an alkali bypass stack associated with the kiln or in-line kiln/raw mill, the combined PM emission from the kiln or in-line kiln/raw mill and the alkali bypass must be less than 0.15 kg/Mg dry feed.
[f] Applies only to new greenfield affected sources.
[g] The average temperature of the test run averages during performance test must be less than or equal to 204 degrees C.

TABLE 3.—SUMMARY OF EMISSION LIMITS[a,b] FOR AFFECTED SOURCES AT PORTLAND CEMENT PLANTS

(English units)

Affected source and pollutant	Emission limit for existing sources	Emission limit for new sources
NHW kiln and NHW in-line kiln/raw mill[c] PM	0.30 lb/ton dry feed[e] and opacity level[c] no greater than 20 percent	0.30 lb/ton dry feed[e] and opacity level[c] no greater than 20 percent
NHW kiln and NHW in-line kiln/raw mill D/F[c,d]	8.7×10^{-11} gr TEQ/dscf or 1.7×10^{-10} gr TEQ/dscf with PM control device operated at ≤400°F[g]	8.7×10^{-11} gr TEQ/dscf or 1.7×10^{-10} gr TEQ/dscf with PM control device operated at ≤400°F[g]
NHW kiln and NHW in-line kiln/raw mill THC[d]	none	50 ppmvd[f] (as propane)
Clinker cooler PM	0.10 lb/ton dry feed and opacity level no greater than 10 percent	0.10 lb/ton dry feed and opacity level no greater than 10 percent
Raw material dryer and materials handling processes (raw mill system, finish mill system, raw material storage, clinker storage, finished product storage, conveyor transfer points, bagging, and bulk loading and unloading systems) PM.	10 percent opacity	10 percent opacity
Raw material dryer THC[d]	none	50 ppmvd[f] (as propane)

[a] All concentration limits at 7 percent oxygen.
[b] Applies to major sources only, except as noted.
[c] Includes main and alkali bypass stacks.
[d] Applies to both major and area source portland cement plants.
[e] If there is an alkali bypass stack associated with the kiln or in-line kiln/raw mill, the combined PM emission from the kiln or in-line kiln/raw mill and the alkali bypass must be less than 0.30 lb/ton dry feed.
[f] Applies only to new greenfield affected sources.
[g] The average temperature of the test run averages during performance test must be less than or equal to 400 degrees F.

The NESHAP imposes operating limits on affected sources that are subject to D/F emission limits. These operating limits are summarized in Table 4.

TABLE 4.—SUMMARY OF OPERATING LIMITS FOR AFFECTED SOURCES AT PORTLAND CEMENT PLANTS

Affected Source/Pollutant	Pollutant	Operating Limits
All kilns and in-line kiln raw mills at major and area sources (including alkali bypasses).	D/F	Operate such that the 3-hour rolling average particulate matter control device (PMCD) inlet temperature is no greater than temperature established at performance test. Operate such that the three-hour rolling average activated carbon injection rate is no less than the rate established at performance test (if applicable). Operate such that the three-hour rolling average activated carbon injection nozzle pressure drop or carrier fluid flow rate is no less than that specified by manufacturer (if applicable).

The rule requires the owner or operator to operate such that the temperature at the inlet to the kiln or in-line kiln raw mill particulate matter control device (PMCD) is at a level no greater than the level established during the successful Method 23 performance test. The three-hour rolling average temperature limit is established by taking the average of the one-minute average temperatures for each test run conducted during the successful Method 23 performance test, then averaging each test run average. Further, sources may petition the Administrator for an alternate averaging period or method for establishing operating parameter limits.

Owners or operators of in-line kiln/raw mills are required to establish separate PMCD inlet temperatures applicable to periods when the raw mill is operating and periods when the raw mill is not operating. The appropriate "raw mill operating status dependent" PMCD inlet temperature shall not be exceeded. Owners or operators of kilns or in-line kiln/raw mills equipped with alkali bypasses are required to establish a separate temperatures for the inlet to the kiln or in-line kiln raw mill PMCD and the kiln or in-line kiln/raw mill alkali bypass PMCD. The applicable temperature limit for the alkali bypass is established during the performance test in which the raw mill is operating.

After a transition period in which the status of the raw mill was changed from "off" to "on" or from "on" to "off", compliance with the operating limits for the new mode of operation begins, and the three-hour rolling average is established anew, i.e., without considering previous recordings.

If carbon injection is used for D/F control, the carbon injection system must be operated such that the carbon injection rate shall be maintained at a level equaling or exceeding the rate which existed during the successful Method 23 performance test. The three-hour rolling average carbon injection rate limit is established in the same way as the temperature limit, as described

above. The injection nozzle pressure drop or carrier fluid flow rate must also be monitored, and the minimum levels for these parameters are established based on manufacturers specifications. The nozzle pressure drop or carrier fluid flow rate is monitored with a 3-hour rolling averaging period.

C. Performance Test Provisions

A performance test is required to demonstrate initial compliance with each applicable numerical limit. The rule requires the owner or operator to use EPA Method 5, "Determination of Particulate Emissions from Stationary Sources" to measure PM emissions from kilns, in-line kiln/raw mills and clinker coolers. These tests will be repeated every 5 years. Kilns and in-line kiln/raw mills equipped with alkali bypasses are required to meet the particulate standard based on combined emissions from the kiln exhaust and the alkali bypass. Owners or operators of in-line kiln/raw mills are required to conduct a Method 5 performance test while the raw mill is operating and a separate Method 5 performance test while the raw mill is not operating. In conducting the Method 5 tests, a determination of the particulate matter collected in the impingers ("back half") of the particulate sampling train is not required to demonstrate initial compliance with the standard, however the permitting authority may require a "back half" for permitting, determination of emission fees, particulate matter monitoring or other purposes. Owners or operators are also required to determine the kiln or in-line kiln/raw mill dry feed rate, because the PM emission standards for kilns, in-line kiln/raw mills and clinker coolers are expressed as lb PM/ton (kg PM/Mg) dry feed.

The opacity exhibited during the period of the initial Method 5 performance test shall be determined, if feasible, through the use of a continuous opacity monitor (COM). Where the control device exhausts through a monovent or where the use of a COM in

accordance with the installation specifications of EPA Performance Specification (PS)–1 of appendix B to 40 CFR part 60, is not feasible, EPA Method 9, "Visual Determination of the Opacity of Emissions from Stationary Sources" shall be used. Where the control device discharges through a fabric filter (FF) with multiple stacks or an electrostatic precipitator (ESP) with multiple stacks, the owner or operator has the option of conducting an opacity test in accordance with Method 9, in lieu of installing a COM.

The rule requires the owner or operator to use EPA Method 23, "Determination of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans from Stationary Sources" to measure D/F emissions from kilns and in-line kiln/raw mills. These D/F tests shall be repeated every 2 and one-half years. The temperature at the inlet to the particulate matter control device (PMCD) during the period of the Method 23 performance test shall be continuously recorded. One minute average temperatures must be calculated for each minute of each run of the test. The average of the one-minute averages must be calculated for each test run and included in the performance test report. The average of one-minute averages for each test run is averaged for all test runs, and this is the operating temperature limit not-to-be-exceeded by any 3-hour rolling average temperature during subsequent operations of the affected source. If carbon injection is used for D/F control, the carbon injection rate and other associated operating parameters must be measured during the period of each run of the Method 23 performance tests. The average carbon injection rate and other associated operating parameters measured for the three runs must be determined and included in the test report.

Owners or operators of in-line kiln/raw mills are required to conduct a Method 23 performance test, and record the temperature at the inlet to the PMCD

while the raw mill is operating, and a separate Method 23 performance test with PMCD inlet temperature recording while the raw mill is not operating. If applicable, the carbon injection rate shall be determined during both performance tests. Where applicable, the exhausts from both the kiln or in-line kiln/raw mill and the alkali bypass are required to meet the D/F standard.

The owner or operator is required to repeat the performance tests for opacity, PM, and D/F emissions from kilns and in-line kiln/raw mills within 90 days of any significant change in the raw material components or fuels fed to the kiln (e.g, when there is an increase in the input rate of municipal solid waste, tire-derived fuel, medical waste, or other solid wastes to the kiln or in-line kiln/raw mill, above the rate used in the previous performance test.) Under the standard, the owner or operator shall use a THC continuous emission monitor (CEM) to conduct a performance test of THC emissions from new greenfield kilns, new greenfield in-line kiln/raw mills, and new greenfield raw material dryers. Owners or operators of new greenfield in-line kiln/raw mills are required to demonstrate initial compliance by measuring THC emissions while the raw mill is operating and while the raw mill is not operating. The standard for THC does not apply to the exhaust from the alkali bypass of kilns or the alkali bypass of in-line kiln/raw mills, and these streams are not subject to a performance test for THC. Each THC CEM is required to be designed, installed, and operated in accordance with EPA Performance Specification (PS)–8A of 40 CFR part 60, appendix B.

Under the standard, the owner or operator shall use EPA Method 9, ''Visual Determination of the Opacity of Emissions from Stationary Sources'' to measure the opacity of gases discharged from raw mills, finish mills, raw material dryers and materials handling processes. These tests would be repeated every five years. A summary of performance test requirements is given in Table 5.

TABLE 5.—SUMMARY OF PERFORMANCE TEST REQUIREMENTS

Affected source and pollutant	Performance Test
New and existing NHW kiln and NHW in-line kiln/raw mill [b c] PM	EPA Method 5 [a]
New and existing NHW kiln and NHW in-line kiln/raw mill [b c] Opacity	COM if feasible [d e] or EPA Method 9 visual opacity readings.
New and existing NHW kiln and NHW in-line kiln/raw mill [b c f g] D/F	EPA Method 23 [j]
New greenfield NHW kiln and NHW in-line kiln/raw mill THC	THC CEM (EPA PS–8A) [h]
New and existing clinker cooler PM	EPA Method 5 [a]
New and existing clinker cooler opacity	COM [d i] or EPA Method 9 visual opacity readings
New and existing raw and finish mill PM	EPA Method 9 [a i]
New and existing raw material dryer and materials handling processes (raw material storage, clinker storage, finished product storage, conveyor transfer points, bagging, and bulk loading and unloading systems) PM.	EPA Method 9 [a i]
New greenfield raw material dryer THC	THC CEM (EPA PS–8A) [h]

[a] Required initially and every 5 years thereafter.
[b] Includes main exhaust and alkali bypass.
[c] In-line kiln/raw mill to be tested with and without raw mill in operation.
[d] Must meet COM performance specification criteria. If the fabric filter or electrostatic precipitator has multiple stacks, daily EPA Method 9 visual opacity readings may be taken instead of using a COM.
[e] Opacity limit is 20 percent.
[f] Alkali bypass is tested with the raw mill on.
[g] Temperature parameters determined separately with and without the raw mill operating.
[h] EPA Performance Specification (PS)-8A of appendix B to 40 CFR part 60.
[i] Opacity limit is 10 percent.
[j] Required initially and every 2.5 years thereafter.

D. Monitoring Requirements

The owner or operator of each portland cement manufacturing plant shall prepare for each affected source subject to the rule, a written operations and maintenance plan. The plan shall be submitted to the Administrator for review and approval as part of the application for a part 70 permit. The operations and maintenance plan shall include procedures for proper operation and maintenance of the affected source and air pollution control devices in order to meet the emission limits of the rule. The operations and maintenance plan shall also include procedures to be used during an inspection of the components of the combustion system of each kiln and each in-line kiln/raw mill. This inspection must be conducted at least once per year. Additionally, the operations and maintenance plan shall include corrective action procedures for the raw mill and finish mill, and associated particulate matter control devices (PMCDs), which must be implemented when required by the rule. The operations and maintenance plan shall also include provisions for monitoring opacity from materials handling sources, and to conduct M. 9 tests if visible emissions are observed. (Further details of this are discussed in the preamble section ''Summary of Changes Since Proposal''.) Finally, failure to implement procedures consistent with the operations and maintenance plan will be a violation of this subpart.

The rule requires owners or operators to monitor the opacity of gases discharged from kilns, in-line kiln/raw mills, alkali bypasses and clinker coolers using a COM, if a COM can be feasibly installed in accordance with PS–1 of appendix B to 40 CFR part 60. Where it is not feasible to install a COM, e.g. where the control device discharges through a monovent, the owner or operator is required to monitor emissions by conducting daily Method 9 tests. Where the control device discharges through a FF with multiple stacks or an ESP with multiple stacks, the owner or operator has the option of conducting daily tests in accordance with Method 9, in lieu of installing a COM. The duration of the Method 9 tests is 30 minutes.

The rule requires that kilns and in-line kiln raw mills subject to the particulate matter (PM) standards must install, correlate, and operate PM continuous emission monitors (CEMs). However, the compliance date for

installing PM CEMs is deferred pending further rulemaking. Further discussion of this issue is found in the preamble sections ''Summary of Changes Since Proposal'' and ''Summary of Responses to Major Comments.''

The owner or operator of a kiln or in-line kiln raw mill must install, calibrate, maintain and continuously operate a device to monitor and record the temperature of the exhaust gases from the kiln, in-line kiln/raw mill, and/or alkali bypass (if applicable), at the inlet to or upstream of the kiln, in-line kiln/raw mill, and alkali bypass PMCD. The calibration of the thermocouple or other temperature sensor must be verified at least once every three months.

If activated carbon injection is used for D/F control, the owner or operator must install, operate, calibrate and maintain a device to continuously monitor and record the weight of activated carbon injected and record the weight in 1 minute rolling averages. The accuracy of the weight measurement device must be ± 1 percent of the weight being measured. The calibration of the device must be verified at least once every three months. The owner or operator must record the feeder setting at least once per day and determine the mass of carbon injected for every three-hour rolling average period. In addition, the carbon injection nozzle pressure drop or activated carbon carrier fluid

flow rate must be monitored and recorded. Further, the activated carbon specifications must be the same as or better than the specifications of the carbon used during the previous performance test.

To clarify how the three-hour rolling average is calculated at initial start-up, operating parameter limits will not become effective on the compliance date until enough data have been accumulated to calculate the rolling average for the limit. For example, given that compliance with the standards begins nominally at 12:01 am on the compliance date, the three-hour rolling average temperature limit does not become effective as a practical matter until 3:01 am on the compliance date. This approach is adopted for all continuous monitoring systems, including CEMs.

During intermittent operations, however, periods of time when operating parameters are not recorded for any reason (e.g., source shutdown) are to be ignored when calculating rolling averages. For example, consider how the three-hour rolling average for a parameter would be calculated if a source shuts down for yearly maintenance for a three week period. The first one-minute average value recorded for the parameter for the first minute of renewed operations is added to the last 179 one-minute averages

before the source shut down, to calculate the three-hour rolling average. This approach is adopted for all continuous monitoring systems, including CEMs. This approach would inhibit a source from intentionally interrupting the monitoring system to avoid unwanted parameter values.

The rule requires the owner or operator to monitor THC emissions from the main exhaust of greenfield kilns; the main exhaust of greenfield in-line kiln/raw mills; and greenfield raw material dryers using a CEM installed in accordance with PS–8A in 40 CFR part 60, appendix B.

The rule requires the owner or operator to monitor the opacity from raw mills and finish mills by conducting a daily six-minute test in accordance with Method 22, ''Visual Determination of Fugitive Emissions from Material Sources and Smoke Emissions from Flares.''

Owners or operators of raw mills and finish mills are required to initiate corrective action within one hour of a Method 22 test during which visible emissions are observed. A 30-minute Method 9 opacity test must be started within 24 hours of observing visible emissions.

A summary of monitoring requirements is given in Table 6.

TABLE 6.—SUMMARY OF MONITORING REQUIREMENTS

Affected source and pollutant or opacity	Monitor/Type/Operation/Process	Monitoring requirement
All affected sources	Operations and maintenance plan	Prepare written plan for all affected sources and control devices.
All kilns and in-line kiln raw mills at major sources (including alkali bypass)/opacity.	COM, if applicable	Install, calibrate, maintain and operate in accordance with general provisions and with PS–1.
	Method 9 opacity test, if applicable	Daily test of at least 30-minutes, while kiln is at highest load or capacity level.
All kilns and in-line kiln raw mills at major sources (including alkali bypass)/PM.	PM CEM	The compliance date is deferred until a future rulemaking, at which time EPA will consider what performance specification requirements should be established.
All kilns and in-line kiln raw mills at major and area sources (including alkali bypass)/D/F.	Combustion system inspection	Conduct annual inspection of components of combustion system.
	Continuous temperature monitoring at PMCD inlet.	Install, operate, calibrate and maintain continuous temperature monitoring and recording system; calculate 3-hour rolling average; verify temperature sensor calibration at least quarterly.
	Activated carbon injection rate, nozzle pressure drop or carrier fluid flow rate, and carbon type/brand, if applicable.	Install, operate, calibrate and maintain continuous activated carbon injection rate monitor; verify calibration at least quarterly; record feeder setting daily; calculate average injection rate for each 3-hour rolling average. Monitor nozzle pressure drop or carrier fluid flow rate according to manufacturers specifications, and calculate rolling 3-hour averages.
New greenfield kilns and in-line raw mills at major and area sources/THC.	THC CEM	Install, operate, and maintain THC CEM in accordance with PS–8A; calculate 30-day block average THC concentration.
All clinker coolers at major sources/opacity.	COM, if applicable	Install, calibrate, maintain and operate in accordance with general provisions and with PS–1.
	Method 9 opacity test, if applicable	Daily test of at least 30-minutes, while kiln is at highest load or capacity level.

TABLE 6.—SUMMARY OF MONITORING REQUIREMENTS—Continued

Affected source and pollutant or opacity	Monitor/Type/Operation/Process	Monitoring requirement
All materials handling operations (MHO) at major sources/opacity.	M. 22 visible emissions test as part of operations and maintenance plan.	For each MHO, conduct monthly 1-minute Method 22 visible emissions test; if visible emissions are observed, initiate corrective action within one hour and conduct 30-minute Method 9 test within 10 minutes. For each MHO, if no visible emissions are observed after first 6 months, reduce monitoring to semi-annual. If no VE are observed thereafter, reduce monitoring to annual basis. If VE are observed for a MHO, revert back to conducting VE tests on a monthly basis.
All raw mills and finish mills at major sources/opacity.	Method 22 visible emissions test ..	Conduct daily 6-minute Method 22 visible emissions test while mill is operating at highest load or capacity level; if visible emissions are observed, initiate corrective action within one hour and conduct 30-minute Method 9 test within 24 hours.
New greenfield raw material dryers at major and area sources/THC.	THC CEM	Install, operate, and maintain THC CEM in accordance with PS–8A; calculate 30-day block average THC concentration.

E. Notification, Recordkeeping, and Reporting Requirements

All notification, recordkeeping, and reporting requirements in the general provisions (40 CFR part 63, subpart A) apply to portland cement manufacturing plants. These include: (1) Initial notification(s) of applicability, notification of performance test, and notification of compliance status; (2) a report of performance test results; (3) a startup, shutdown, and malfunction plan with semiannual reports of reportable events (if they occur); and (4) semiannual reports of excess emissions. If excess emissions are reported, the owner or operator shall report quarterly until a request to return the reporting frequency to semiannual is approved.

The NESHAP general provisions (40 CFR part 63, subpart A) require that records be maintained for at least 5 years from the date of each record. The owner or operator must retain the records onsite for at least 2 years but may retain the records offsite the remaining 3 years. The files may be retained on microfilm, microfiche, on a computer disk, or on magnetic tape. Reports may be made on paper or on a labeled computer disk using commonly available and compatible computer software.

IV. Summary of Changes Since Proposal

In response to comments received on the proposed standards, changes have been made to the final standards. These changes include clarifications designed to make the EPA's intent clearer as well as changes to the requirements of the proposed standards. A summary of the substantive changes made since the proposal is given in the following sections, along with the rationales for these changes. Further details on the rationales for these changes can be found in Section VI of the preamble:

Summary of Responses to Major Comments.

A. Designation of Affected Sources

The section of the rule on designated affected sources is being clarified to include new greenfield raw material dryers that are located at facilities that are area sources. The EPA is clarifying today that these affected sources are subject to limitations on THC. The preamble for the proposed rule stated that polycyclic organic matter (POM) emissions (using THC as a surrogate) from portland cement NHW kiln area sources would be subject to MACT standards under EPA's interpretation of section 112(c)(6). The EPA proposed to use THC as a surrogate for organic HAPs, and today it is clarifying that POM is an organic HAP for which THC is a surrogate. Since POM was a listed HAP from portland cement NHW cement kilns (at both area and major source portland cement plants) in the section 112(c)(6) listing (63 FR 17838, April 10, 1998), the EPA is clarifying that the limitation of emissions of THC applies to new greenfield cement kilns, in-line kiln raw mills and raw material dryers at major and area source cement plants in the portland cement industry. Further discussion of this change is found below in the discussion of standards.

B. Definitions

The definitions of "alkali bypass" and "feed" have been expanded to reflect cement industry practices. Definitions of "greenfield" and new "brownfield" affected sources have been added to the final rule to clarify the applicability of the final THC standards to specific affected sources. A definition of "one-minute average" has been added to clarify the monitoring provisions of the final rule. A definition of rolling average has been added to clarify and maintain

consistency with the requirements for HW kilns.

C. Emission Standards and Operating Limits

Based on comments received, the EPA is clarifying today that the THC limitation applicable to new kilns, new in-line kiln/raw mills, and new raw material dryers is restricted to greenfield sources, in recognition of the difficulty that owners or operators of reconstructed and new brownfield affected sources might have in obtaining suitable kiln feed materials while remaining competitive. The selection of a site tied to feed materials with relatively low levels of naturally occurring organic matter is the basis for the MACT standard and is an option only available to greenfield sources. Further, as discussed above, the EPA is clarifying that this THC limitation applies to new greenfield kilns, new greenfield in-line kiln/raw mills, and greenfield raw material dryers located at facilities that are area, as well as major, sources.

The requirements in the proposal for initiating a site-specific operating and maintenance plan, and implementation of a quality improvement plan, due to stipulated exceedences of a 15 percent kiln opacity limit, have been removed. The EPA agrees with commenters who questioned this tiered approach, and so the final rule will retain only a 20 percent opacity limit for the kiln and in-line kiln/raw mill.

In response to a comment, the EPA is clarifying that the opacity limitation on gases discharged from raw mills and finish mills is restricted to the mill sweep and air separator air pollution control devices. This is consistent with the MACT floor technology for control of gases from these affected sources.

The final rule has been reformatted to provide a separate section for operating

limits. Control of temperature at the inlet to kiln and in-line kiln/raw mill PMCDs and control of the activated carbon injection parameters (if applied as a D/F control technique) are provisions promulgated as operating limits.

The averaging period for the operating limit for the inlet kiln and in-line kiln/raw mill PM control device temperature (to demonstrate compliance with the D/F emission limits) has been changed from a 9-hour block average period to a three-hour rolling average period. Comments were received that the averaging period should be shorter. In addition, the rule has been clarified to include data reduction procedures to be followed to demonstrate compliance. Furthermore, sources may petition the Administrator for an alternate averaging period or method for establishing operating parameter limits.

The provisions for establishing the PM control device inlet temperature limit based on the D/F performance test have been changed to correct an error in drafting the proposal. A commenter pointed out that the proposal would allow a source to conduct its D/F performance test with an inlet PM control device temperature below 400 degrees F, but after the performance test, the source would be allowed to operate its PM control device with an inlet temperature up to 400 degrees F. In drafting the proposal, the EPA did not intend to allow a source to operate its PM control device at a temperature higher than the temperature during the performance test, and so the EPA is clarifying today that the inlet temperature limit is established as and capped at the average temperature during the D/F performance test. To further achieve consistency with the D/F temperature requirements for HW kilns and to better assure that the standard reflects MACT, the EPA is dropping the proposed provision which would have allowed the temperature limit to be established as the average temperature during the performance test plus 25 degrees F if the D/F level was below 0.15 ng/dscm. To clarify and maintain consistency with the requirements for HW kilns (and to best implement standards representing MACT), if the source complies with the O.4 ng TEQ/dscm D/F limit, the average temperature of the test run averages during the performance test must be below 400 degrees F. To further achieve consistency with the requirements for HW kilns, additional operating parameter limits associated with the use of activated carbon injection must be established and these parameters must be monitored continuously. The

averaging period for the activated carbon injection rate and other operating parameters has been changed from a 9-hour period to a 3-hour rolling average period. Further details on the establishment of the temperature and other operating parameter limits are discussed in section VI. of this preamble.

D. Performance Test Requirements

In response to comment, the EPA is clarifying that both during the performance test and to demonstrate continuous compliance, opacity limitations for the kiln and clinker cooler must be met for each 6-minute block period. (The proposal incorrectly required a 30-minute averaging time.) This is consistent with the requirements of the NSPS, which is the basis for the MACT floor for PM/metals and opacity.

Based on comments received that there should be consistency with the requirements for HW kilns, the performance tests for D/F must be conducted every 2 and one-half years. (The proposal would have required that the D/F emissions tests be conducted every 5 years.) To further achieve consistency, and to assure that the kiln continues to achieve the requisite emissions reductions reflected in the standard, the EPA is also clarifying today that in addition to repeating performance tests every five years (or 2.5 years for the D/F performance tests), performance tests for kilns or in-line kiln/raw mills must be repeated within 90 days of initiating any significant change in the feed materials or fuels fed to the kilns (e.g., an increase in the input rate of municipal solid waste, tire-derived fuel, or medical waste to the kiln or in-line kiln/raw mill above the rate used in the previous performance test; or a switch from burning natural gas to coal). Such changes in fuel or feeds could result in changes to emissions.

E. Monitoring Requirements

In response to a comment, clarification has been added to the final rule to establish that any required Method 9 and Method 22 tests must be conducted while the affected source is operating at the highest load or capacity level reasonably expected to occur within the day that the test is performed.

The option for use of triboelectric bag leak detection systems for monitoring raw mill and finish mill fabric filter performance is not being promulgated at this time. Numerous commenters expressed concern regarding installation, operation, calibration and maintenance, and that the lack of clear-

cut specifications would lead to open-ended liability for owners/operators. Those owners or operators who want to use bag leak detection systems may petition the Administrator for approval of alternative monitoring requirements under the General Provisions.

Requirements for temperature monitoring devices (including range and reference standard) have been added to the final rule. In response to a comment, monitoring requirements for activated carbon injection system accuracy, calibration frequency, and data recording and reduction have also been added to the final rule. To achieve consistency with the requirements for HW kilns, activated carbon injection nozzle pressure drop or carrier fluid flow rate, and carbon specifications, must also be monitored and recorded.

An explicit monitoring requirement for an inspection of the components of the combustion system of each kiln or in-line kiln/raw mill has been added to the rule. This inspection must be conducted at least once per year, in accordance with the procedures specified in the operation and maintenance plan for the affected source. This change was made in response to several comments that were received suggesting that provisions (such as limitations on and monitoring of carbon monoxide) be added to the final rule to ensure good combustion and thus minimize formation of D/F.

The operations and maintenance plan requirement has been changed to explain that the plan must also include provisions for observing opacity from materials handling sources, and for conducting a M. 9 test if visible emissions (VE) are observed. Specifically, materials handling sources' VE shall be monitored via M. 22 once per month. After 6 months without VE for each individual source, the monitoring frequency would be reduced to a semi-annual basis. If there are no VE in the next 6 month period for a particular source, the monitoring frequency would be reduced to an annual basis. If VE occurs during the annual inspection, the frequency would revert back to once per month. If VE are observed during one of these inspections, a Method 9 test is required. This change was made to provide greater assurance that these units are in compliance with the opacity limit and to meet the Agency's commitment to incorporate enhanced monitoring in all MACT standards.

Finally, the final rule is being clarified that failure to implement procedures consistent with the operations and maintenance plan will be a violation of this subpart.

In the preamble to the proposal, the EPA noted its intent to include a requirement for PM continuous emission monitors (CEMs) in the final rule, unless the analyses of new information and data showed that it is not appropriate. (See 63 FR at 14205). Based on successful testing on an incinerator, as well as extensive use of these monitors in Europe, EPA believes there is sound evidence the PM CEMs should work at cement kilns. Accordingly, the final rule contains a requirement to install PM CEMs. However, we are deferring the effective date of this requirement pending further testing and additional rulemaking. Please see the preamble section ''Summary of Responses to Major Comments'' for further details on this issue.

F. Additional Test Methods

The final rule has been changed to permit the use of either Method 320 or Method 321 for the determination of hydrogen chloride (HCl) for the purpose of making an applicability determination. These methods are being promulgated as part of this rulemaking.

Since proposal of Method 322 for the measurement of HCl along with the portland cement NESHAP, the EPA attempted to utilize Method 322 to gather data from lime kilns (which have a matrix similar to portland cement sources) and encountered technical problems with the gas filter correlation infrared spectroscopy (GFCIR). Many of these problems were adequately identified by the data quality indicators in the method. However, as a backup option, the Agency collected data sets at lime kilns using both GFCIR and Fourier transform infrared spectroscopy (FTIR). These paired data sets provide unexpected contradictory results.

The dynamic spiking results of the GFCIR would indicate that Method 322 results should be biased by overpredicting the true value (the spike recovery consistently showed greater than 100 percent recovery). However, FTIR data collected nearly simultaneously with the GFCIR data show that the GFCIR results were significantly lower than FTIR results. Since the Agency applied statistical methods to analyze the FTIR data and concluded that the FTIR method did not have a significant bias, the Agency is confident in the values reported by the FTIR instrument. Therefore, this leads

to a paradox with the GFCIR data; the results are contradictory for the GFCIR. At this point, the Agency has not determined the cause of the paradox, which has led to the decision to postpone promulgation of Method 322 as an alternative method for measurement of HCl from portland cement kilns.

The EPA will continue to investigate the reasons for the differences in the two methods, and if a satisfactory solution is found to correct the problem, may consider further action on this method if additional evaluation data are available. For this reason proposed Method 322 is not being promulgated at this time and may not be used in applicability determinations for portland cement plants. (A more detailed discussion of this can be found in comment 2.5.1 in the Response to Comment Document.)

In the proposal, we stated that Methods 26 and 26A may be used in applicability determinations provided that these methods are validated concurrently using M. 321 or 322. Several comments were received stating that EPA is restricting M. 26 and M. 26A use by requiring that they be validated each time they are used, and that Method 26 has long been an approved EPA test method. Based on these comments, this requirement has been changed such that Methods 26 and 26A may be used to confirm a source is a major source without concurrent validation with M. 321 or M. 322. However, M. 26 or 26A may not be used to make the assertion that the source is an area source. Only the FTIR methods may be used for the measurement of HCl if the source wishes to claim it is not a major source. See the preamble section ''Summary of Responses to Major Comments'' for further discussion of this issue about how a source should determine whether it is a major or area source.

G. Reporting

A provision has been added to the final rule requiring that the semi-annual summary report for the period in which the annual combustion system component inspection was conducted include the results of the inspection.

H. Exemption from New Source Performance Standards

To eliminate overlap or duplicate coverage of NSPS and MACT standards

for portland cement facilities, affected sources subject to requirements under this NESHAP are exempted from requirements under 40 CFR 60, subpart F, the New Source Performance Standards. However, there are two exceptions to this: kiln and in-line kiln/raw mills, and greenfield raw material dryers, that are new or reconstructed sources under the definition in Subpart F, and are located at area source cement plants, would still be subject to applicable PM limits, opacity limits, and recordkeeping and reporting requirements of the NSPS. The reason for this is that these ''NSPS'' kilns and in-line kiln/raw mills, and greenfield raw material dryers that are located at area source cement plants would be subject to the NESHAP's D/F and/or THC limits, but would not be subject to the NESHAP's PM limits, because they are located at area source cement plants.

I. Delegation of Authority

The final rule reserves authority for approval of alternate emission standards, major alternatives to test methods, major alternatives to monitoring procedures and waivers of recordkeeping.

J. Test Methods 320, 321, and 322

Test Methods 320 and 321 are being promulgated with minor corrections to clarify and improve test procedures, and correct equations incorrectly stated in the proposal notice. Proposed Test Method 322 is not being promulgated at this time as noted in Section F above.

V. Summary of Impacts

A. Air Quality Impacts

The air quality impacts of the final rule are identical to those of the proposed rule. Nationwide baseline HAP emissions from portland cement manufacturing plants are estimated to be 260 Mg/yr (290 tpy) at the current level of control. This rule will reduce emissions of HAPs by 82 Mg/yr (90 tpy) from baseline levels. Estimates of annual emissions of HAPs and expected reductions from implementation of this rule are given in metric and English units in Tables 7 and 8. The following text reviews the information provided in Tables 7 and 8.

TABLE 7.—NATIONWIDE ANNUAL EMISSION REDUCTIONS OF HAPS AND OTHER POLLUTANTS FROM PORTLAND CEMENT MANUFACTURING PLANTS

(Metric units)

Source	Pollutant	Baseline emissions (Mg/yr)	Emission reduction [Mg/yr]
Kilns, in-line kiln/raw mills, and alkali bypasses	HAP Metals[a]	150	35
	PM[a]	14,000	3,400
	D/F (TEQ)[b]	44 g/yr	16 g/yr
	Organic HAPs[c]	120	47
	THC[c]	530	200
Clinker coolers	HAP Metals[a]	1.1	0.18
	PM[a]	8,100	1,300

  [a] These numbers pertain to existing sources only.
  [b] These numbers pertain to both new and existing NHW kilns.
  [c] These numbers pertain to new greenfield NHW kilns only.

TABLE 8.—NATIONWIDE ANNUAL EMISSION REDUCTIONS OF HAPS AND OTHER POLLUTANTS FROM PORTLAND CEMENT MANUFACTURING PLANTS

[English units]

Source	Pollutant	Baseline emissions (tpy)	Emission reduction (tpy)
Kilns, in-line kiln/raw mills, and alkali bypasses	HAP Metals[a]	160	38
	PM[a]	16,000	3,800
	D/F (TEQ)[b]	0.096 lbs/yr	0.035 lbs/yr
	Organic HAPs[c]	130	52
	THC[c]	580	220
Clinker coolers	HAP Metals[a]	1.2	0.2
	PM[a]	8,800	1,400

  [a] These numbers pertain to existing sources only.
  [b] These numbers pertain to both new and existing NHW kilns.
  [c] These numbers pertain to new greenfield NHW kilns only.

This rule will reduce PM emissions from the existing NHW cement kilns and in-line kiln/raw mills by 3,400 Mg/yr (3,800 tpy) from the baseline level, a reduction of 24 percent. Emissions of HAP metals from the affected existing NHW cement kilns and in-line kiln/raw mills will be reduced by 35 Mg/yr (38 tpy), a reduction of 24 percent from the baseline level. Emissions of D/F TEQ will be reduced by 15 grams (g)/yr (0.033 lb/yr), a reduction of 36 percent from the baseline level, at existing NHW cement kilns and in-line kiln/raw mills located at major source and area source facilities.

For new NHW cement kilns and in-line kiln/raw mills, the MACT standards are projected to reduce emissions of D/F TEQ by an average of 0.6 g/yr (0.001 lb/yr) over the next 5 years (from major and area sources), a 36 percent reduction from projected baseline emissions. For new kilns, the MACT standards will also reduce projected emissions of THC by an average of 200 Mg/yr (220 tpy) and organic HAPs by an average of 47 Mg/yr (52 tpy) over the next 5 years, an emissions reduction for each of 39 percent from corresponding estimated nationwide baseline emissions.

The MACT standards will reduce PM emissions from 35 percent of the existing clinker coolers by 1,300 Mg/yr (1,400 tpy) from the baseline level, a reduction of 16 percent. Emissions of HAP metals from affected existing clinker coolers will be decreased by 0.18 Mg/yr (0.2 tpy), a reduction of 16 percent from the baseline level.

Additional reductions of THC and organic HAPs will result from the MACT standards for new greenfield raw material dryers. However, information on THC emission rates from raw material dryers and a projection of the number of such affected sources is not currently available, so nationwide reductions cannot be estimated.

B. Water Impacts

The impacts of the final rule are identical to those of the proposed rule. Control of D/F emissions using water injection for temperature reduction will result in an estimated increased water consumption (evaporated into the kiln exhaust gas for cooling) of 190 million gallons per year for existing NHW kilns and NHW in-line kiln/raw mills and 8 million gallons per year for new NHW kilns and NHW in-line kiln/raw mills.

C. Solid Waste Impacts

The impacts of the final rule are identical to those of the proposed rule. The amount of solid waste from existing NHW kilns, in-line kiln/raw mills, and clinker coolers (located at major sources) will increase by an estimated 4,700 Mg/yr (5,200 tpy) due to the requirements for PM control in the final rule.

D. Energy Impacts

The impacts of the final rule are identical to those of the proposed rule. For existing NHW kilns and NHW in-line kiln/raw mills the MACT standards for PM and D/F will increase energy consumption by an estimated 11 million kilowatt hours (KWh)/yr [38 billion British thermal units (Btu)/yr]. For new NHW kilns and NHW in-line kiln/raw mills the MACT standards for D/F will increase energy consumption by an estimated 10,600 KWh/yr (36 million Btu/yr).

E. Nonair Health and Environmental Impacts

The reduction in HAP emissions will have a beneficial effect on nonair health and environment impacts. Dioxin/furan and HAP metals have been found in the Great Lakes and other water bodies and

have been listed as pollutants of concern due to their persistence in the environment, potential to bioaccumulate, and toxicity to humans and the environment. Implementation of the NESHAP will aid in reducing aerial deposition of these emissions.

Occupational exposure limits under 29 CFR part 1910 are in place for some of the regulated HAPs (and surrogates) not including D/F. The National Institute for Occupational Safety and Health recommends an exposure level for D/F at the lowest feasible concentration. The final rule will reduce emissions, and consequently, occupational exposure levels for plant employees.

F. Cost Impacts

For new and existing NHW kilns, NHW in-line kilns/raw mills, clinker coolers, raw and finish mills, and materials handling facilities, the projected overall total capital costs of the final rule for controlling and monitoring emissions of D/F, PM (includes opacity), and THC are $108 million. The overall projected annual costs of the rule, for controlling and monitoring for D/F, PM (includes opacity), and THC, are $37 million. For new and existing NHW kilns and NHW in-line kiln/raw mills, the projected total capital and annual costs of complying with the MACT standard for D/F (includes controls and monitoring) are $15 million and $3.6 million, respectively. For new and existing sources subject to PM and/or opacity limits, the projected total capital and annual costs of complying with the MACT standards for PM and opacity (including PM controls, PM CEMs, and continuous opacity monitors) are $92 million and $33 million, respectively. With respect to PM CEMs costs only, the projected total capital and annual costs of PM CEMs are $15 million and 7.6 million, respectively. The THC emissions limit for new greenfield NHW kilns, NHW in-line kiln/raw mills and raw material dryers can be met by processing materials with typical levels of organic content, without installing and operating add-on pollution control systems that would be relatively costly. Feed materials that have sufficiently low levels of organic matter are widespread across the U.S., and the siting of new greenfield kilns is not expected to be significantly limited by the emission limit. The projected fifth-year national capital and annual costs of monitoring THC with a continuous emission monitor for new greenfield NHW kilns, in-line kiln/raw mills and raw material dryers are $0.75 million and $0.45 million, respectively (based

on an estimated four new affected sources).

G. Economic Impacts

EPA conducted an economic analysis of the proposed NESHAP, and has reconducted its analysis to include the costs of PM CEMs and the monitoring of materials handling sources. The economic impacts of the final rule are slightly greater than those of the rule as proposed. Because the final standards may potentially include costs associated with PM CEMs and the monitoring of materials handling sources, EPA reconducted its economic analysis. This revised analysis evaluates a regulatory option that is more stringent than the final standards. Analyzing this more stringent option, which overstates the expected compliance costs, causes the economic impacts presented here to over estimate the expected impacts of the final standards. However, these economic impacts are only slightly greater than those of the proposal analysis.

The EPA estimates that regional market price increases of portland cement will be between 0.3 and 2.6 percent. The national average price increase is estimated to be 1.1 percent. The related decreases in quantity demanded of portland cement are estimated to range from 0.3 to 2.3 percent, with a national average of 1.0 percent. Domestic production of portland cement is estimated to decrease more than consumption (2.2 percent compared to 1.0 percent nationally because imports are estimated to increase by 5.5 percent). The decreases in domestic production may lead to the loss of approximately 334 jobs in the United States. No plants are expected to close; four kilns are expected to cease operating.

VI. Summary of Responses to Major Comments

A complete summary of all of the public comments on the proposal, and responses to these comments is provided in the "Response to Comments" document available in the docket and from EPA's Technology Transfer Network. The responses to major comments are given in this section.

Portland Cement Source Category

Comment: Commenters raised objections to splitting the portland cement category for cement kilns by the type of fuel (hazardous waste vs. fossil fuels) burned in the kiln. The commenters stated that splitting the industry by fuel type deviates from EPA's original source category list (July

16, 1992 FR) which included only a portland cement manufacturing category, and that no distinction is made regarding fuel type under the New Source Performance Standards (NSPS) for portland cement plants. The commenters were concerned that EPA's decision not to use the NSPS category will result in what Congress hoped to avoid (through section 112(c)(1)) by causing unnecessary costs and dislocations in the cement industry.

Response: Section 112(d)(1) of the Clean Air Act specifically provides that "the Administrator may distinguish among classes, types and sizes of sources within a category or subcategory in establishing standards. . . .". With regard to having separate categories/ subcategories, the EPA believes that there can be significant differences in emissions due to hazardous waste burning that warrant separate classes for these devices. The types of HAPs found in emissions from hazardous waste-burning kilns are different from, and more numerous than, those from NHW kilns. Hazardous wastes can contain virtually any HAP, which in turn can be in stack emissions. The fact that hazardous waste-burning kilns are dealt with separately under a different statute (RCRA section 3004(q)(special standards for industrial furnaces which burn hazardous waste fuels)) likewise indicates that hazardous waste-burning cement kilns can be dealt with legitimately as a separate class. Indeed, this existing RCRA regulatory regime has created a different data base, and system of existing controls, which can result in different analyses, different floor controls and standards under the section 112 MACT process, again indicating that these sources can reasonably be classified as a distinct class. To summarize, this NESHAP for portland cement manufacturing covers NHW kilns and NHW in-line kiln raw mills; it does not apply to HW cement kilns which are subject to subpart EEE of this part. This NESHAP also covers affected sources located at portland cement manufacturing plants (such as clinker coolers, raw material dryers, and materials handling processes), regardless of whether the plant operates HW kilns.

Comment: Two commenters stated that EPA has not met its legal burden to be consistent when regulating HW and NHW cement kilns. The commenters stated that the EPA has not used consistent rationales and approaches to develop emission limitations for the same pollutants.

Response: There are a number of differences between kilns that burn hazardous waste and those that do not

in terms of process feed/fuel, process operation, pollutants and pollutant quantities generated, existing regulations that impact MACT floor determinations, and the economics of their operations. These differences provide the bases for differences in determinations of MACT floors, emission limits, and other regulatory requirements. When there is no rational reason for differences between the two standards, EPA has changed the two sets of rules (see section IV. of this preamble for a discussion of changes made to this rule since proposal) to make them more consistent.

Regulation of Cement Kilns Under Section 129

Comment: According to one commenter, the EPA is required to regulate any facility that combusts any solid waste under section 129 of the Clean Air Act. However, EPA's current section 129 regulations either: (1) Exempt portland cement kilns that burn any amount of hospital waste, medical waste, and infectious waste from the medical waste incinerator (MWI) rule, (2) exempt cement kilns that burn less than thirty percent waste from the municipal waste combustor (MWC) rule, or (3) have yet to be promulgated as the commercial and industrial waste rule. The commenter asserts that the EPA cannot fail to promulgate section 129 regulations for cement kilns that burn non-hazardous solid waste by suggesting that it may promulgate section 129 regulations in the future. Cement kilns would then be permitted to combust any of these wastes without complying with section 129, despite the fact that the Clean Air Act expressly mandates that any unit burning any solid waste must comply with section 129. Therefore, the commenter asserts that the EPA must promulgate section 129 standards for cement kilns that burn any solid waste now. If EPA cannot promulgate section 129 standards immediately, the commenter asserts that EPA must, at a minimum, include numerical emission standards for the pollutants listed in section 129 (including mercury, cadmium, and lead) in its proposed regulations under section 112.

Response: EPA does not read section 129 as precluding EPA from promulgating an interim section 112 (d) standard for portland cement kilns which burn non-hazardous solid waste. The interim alternative is to have no regulation at all for HAP emissions. This is because the only rules implementing section 129 explicitly do not apply to waste-burning cement kilns (see 40 CFR sections 60.50b(p), 60.32b(m), 60.50c(g)

and 60.32e(g)) and the explanation for these provisions in 62 FR at 45117 (Aug. 25, 1997) and 62 FR at 48538 (Sept. 15, 1997)). Neither the commenter or any other person challenged these provisions, and EPA is not reopening the section 129 rules for consideration here.

EPA does not regard interim non-regulation of non-hazardous waste burning cement kilns as a reasonable alternative to including them within the scope of these portland cement MACT regulations. Indeed, were the Agency to exempt waste burning cement kilns from these MACT standards, it would create a strong incentive for cement kilns to burn waste to escape MACT regulation. EPA emphasizes, however, as we did at proposal, that the standards in today's rule do not represent EPA's final determination that only section 112 (d) standards are appropriate or required for solid non-hazardous waste-burning cement kilns. Today's action does not in any way foreclose an eventual section 129 standard.[1]

With regard to the commenter's suggestion that EPA adopt specific emission limits in this MACT rule for mercury, lead, and cadmium—which are pollutants identified in Section 129 for regulation—as EPA discussed at proposal, emission limits were considered in the MACT rule for these pollutants. As discussed at proposal, EPA was unable to identify a MACT floor for mercury. As a result, there is no mercury emission limit which can be associated with a MACT floor. The use of activated carbon injection (ACI) was considered by EPA as a "beyond the floor" alternative. However, as also discussed at proposal, based on the relatively low levels of existing mercury emissions from individual NHW cement kilns and the costs of reducing these emissions by ACI, EPA does not consider this beyond the floor alternative justified. Thus, no mercury emission limit is included in the final MACT rule, and thus would not be included even if this was a section 129 rule. Finally, as also discussed at proposal, EPA considers PM a surrogate for semi-volatile metals (e.g., lead, cadmium, etc.). The proposed rule and the final rule include a PM emission limit based on the use of MACT. As a result, the final rule achieves reductions in emissions of these pollutants consistent with MACT. Furthermore, sufficient data do not exist to identify emission limits for lead and/or

[1] Any waste burning cement kiln subject to a section 129 standard would no longer be subject to these section 112 (d) MACT standards. See CAA section 129 (h) (2).

cadmium associated with MACT and EPA is unable to establish emission limits for these pollutants in this rule. See *Sierra Club* v. *EPA,* no. 97–1686 (D.C. Cir. 1999) slip op. at 15 (EPA is not obliged to establish a MACT standard for HAPs for which the Agency is unable to quantify emission reductions). Even if such emission limits could be developed, however, they would not result in any further reduction in emissions beyond that achieved by the MACT rule, given the PM standard.

Comment: Other commenters believe that cement kilns, irrespective of their fuel or raw material mix, should be regulated under the portland cement NESHAP and not under section 129 of the Clean Air Act. Commenters stated that the EPA's discussion of its authority under section 129 is irrelevant to, and inappropriate in, the proposed portland cement NESHAP. They said that if EPA intends to regulate cement kilns that burn solid waste materials under section 129, the proper venue would be in a proposal pursuant to section 129. Commenters stated that, based on the discussion of section 129, EPA has apparently already determined how it intends to treat solid waste burning cement kilns in the section 129 rulemaking. Ten commenters were concerned that cement kilns could be subject to different regulations from year-to-year (or day-to-day) depending on whether they trigger the section 129 applicability thresholds. The commenters believe that such a regulatory structure is confusing, burdensome, inappropriate, and raises serious legal issues. Commenters noted that the EPA's proposed regulation of solid waste burning cement kilns under section 129 could lead to increased fuel consumption and emissions of greenhouse gases as cement kilns try to avoid triggering section 129 regulation by not burning alternative fuels like solid waste.

Response: The EPA acknowledges all the comments dealing with the potential future regulation under section 129 of the CAA of air emissions from cement kilns that burn solid waste (other than hazardous waste). Both the proposed and final promulgated portland cement NESHAP apply to cement kilns which burn solid waste (other than hazardous waste). If the EPA decides in the future that emission standards developed under the authority of section 129 of the CAA are warranted for cement kilns that burn solid waste, a separate rule will be proposed to allow for public comment. The commenters' concerns regarding duplicative regulations are misplaced, however. See CAA section 129(h)(2)

(units can't be regulated simultaneously under both sections 129 and 112(d)(2)).

Regulation Under 112(c)(6)

Comment: Commenters stated that the EPA should not exercise its authority under section 112(c)(6) to regulate dioxin/furan emissions from area sources since the area sources have *de minimis* dioxin/furan emissions and regulating them under section 112 will impose significant burdens (for reporting, recordkeeping, monitoring, and control technology) while providing negligible environmental benefits. These commenters further state that EPA's own estimates indicate D/F emissions from NHW kilns contribute only 0.8 percent of total nationwide D/F emissions. The commenters do not believe that Congress intended such a result in drafting section 112(c)(6).

Response: Regarding the above comments about regulation of D/F under section 112(c)(6), the EPA is required by section 112(c)(6) to ''list categories and subcategories of sources assuring that sources accounting for not less than 90 per centum of the aggregate emissions of each such pollutant are subject to standards under subsection (d)(2) or (d)(4) of this section.'' The method for identifying and selecting sources for listing and regulation under these subsections was discussed at length in **Federal Register** notices published on June 20, 1997 (62 FR 33625) and April 10, 1998 (63 FR 17838). Section 112(c)(6) does not provide for de minimis exemptions for source categories, but rather directs EPA to make findings on the basis of what is necessary to meet the requirement to assure that sources accounting for 90 percent of the emissions of these pollutants are subject to standards. Moreover, because the pollutants addressed by section 112(c)(6) are persistent, that is, they remain in the environment for extremely long periods of time without breaking down, the EPA believes that any claims of de minimis contributions should be considered with great caution, and granted in only very exceptional circumstances. Consequently, the EPA believes that its decisions in response to section 112(c)(6) represent a reasonable exercise of its discretion within the constraints of that subsection.

Comment: Several commenters stated that EPA's proposed action to regulate cement kiln ''area sources'' under CAA section 112(c)(6) violates the CAA and is arbitrary and capricious. They stated that the EPA has improperly proposed to apply the MACT standards to area source cement kilns and other HWCs before deciding upon listing criteria and

preparing the overall list or lists of sources required by that provision. In referring to EPA's proposal to regulate area sources of 112(c)(6) pollutants, they stated their view that only those 112(c)(6) pollutants for which a source category is listed under 112(c)(6) should be regulated.

Response: Regarding the initial portion of the above comment, the notice of the final source category listing for section 112(d)(2) rulemaking pursuant section 112(c)(6) requirements was published April 10, 1998, in 63 FR 17838–17855. The referenced notice provides the required listing of area sources, and therefore the commenter's point is moot.

The proposed rules for NHW kiln portland cement manufacturing would only have regulated area sources for D/F emissions, which is one of the pollutants for which these plants are listed as area sources. The pollutants for which portland cement NHW kilns were listed under 112(c)(6) are polycyclic organic matter (POM), D/F, and mercury. At proposal, the EPA had conducted an analysis under section 112(d)(2) for D/F and mercury with respect to establishing emission standards, and concluded that area sources of D/F should be regulated. The analysis for mercury showed that the MACT floor for new and existing sources was no control. The BTF technology, use of activated carbon injection, was determined not to be cost-effective. Therefore, no emission standard was proposed for mercury.

The preamble for the proposed rule stated that POM emissions (using THC as a surrogate) from portland cement NHW kiln area sources would be subject to MACT standards under EPA's interpretation of section 112(c)(6). A THC emission standard was proposed for new raw material dryers and new NHW in-line kiln/raw mill main exhausts at cement plants that are major sources. At proposal, THC was identified as a surrogate for organic HAP emissions, which would include POM. The final rule's limits on THC emissions are applicable only to new greenfield kilns, in-line kiln raw mills, and raw material dryers, for reasons discussed in section IV.C. of this preamble. EPA is clarifying today that since THC is a surrogate for POM, the THC emission limits are applicable to new greenfield kilns and raw material dryers at cement plants that are major and area sources.

Comment: Several commenters stated their support for an alternative interpretation of regulating area sources emitting HAPs listed under 112(c)(6). They stated that section 112(d)(5) does not exclude area source categories listed

pursuant to section 112(c)(6) from the Agency's discretionary authority to apply GACT standards nor does section 112(c)(6) prohibit EPA from exercising its discretionary authority under section 112(d)(5). According to the commenters, section 112(d)(5) grants the Administrator authority to establish GACT standards for any area sources listed pursuant to section 112(c), whether such sources are listed pursuant to section 112(c)(3) or (c)(6). They contended that had Congress intended to exclude section 112(c)(6) area sources from the GACT standards under section 112(d)(5), Congress would have stated this exclusion in section 112(d)(5).

Another commenter argued against the alternative interpretation owing to the bioaccumulation potential of the 112(c)(6) pollutants and the fact that the GACT approach would include no floor analysis or residual risk assessment.

Response: Section 112(c)(6) specifically states that EPA is to assure that sources of the pollutants to which this subsection applies be subject to standards under subsections (d)(2) or (d)(4). These subsections refer, respectively, to MACT and standards for pollutants for which a health threshold has been established (a null set of purposes for this rule). The natural reading of the provision (and at the least, a permissible one) is to say that MACT standards apply to emissions of 112(c)(6) HAPs from all sources. The alternative reading, that GACT requirements could apply because GACT requirements apply in lieu of section 112d(2) MACT requirements reads language into section 112c(6) not apparent on its face. Moreover, where Congress wished to reference subsection (d) without limitation, it omitted references to specific paragraphs. Compare the language of section 112(c)(6), which refers to standards under subsection (d)(2) or (d)(4), with the language of section 112(k)(3)(B)(ii), which refers to standards under subsection (d). In addition, the reading suggested by the industry commenters goes against the natural purpose of section 112c(6), namely, to assure that the maximum available control technology is applied to control the emission of the most dangerous HAPs. (This is also the thrust of the comment summarized above criticizing the reading suggested by industry commenters. EPA agrees with this comment.) The Agency has therefore concluded that none of the comments provided compelling facts or arguments to overcome the interpretation that section 112(d)(2) specifically refers to MACT standards.

Regulatory Flexibility Act and the Small Business Regulatory Enforcement Fairness Act

Comment: Several commenters stated or supported the belief that the proposed rulemaking was incorrectly certified, contending that no factual basis was provided for the Agency's certification of no significant impact on a substantial number of small entities, and thus, EPA is not in compliance with provisions of the Regulatory Flexibility Act (RFA), 5 U.S.C. 601 *et seq.* They stated that EPA needs to review its certification and provide a factual basis for it or complete an initial regulatory flexibility analysis, as required by the RFA.

The commenters contended the certification was deficient in that the Agency's guidance allows regulators to bypass a regulatory flexibility analysis if the industry has fewer than 100 firms. Furthermore, the seven small companies, representing 16 percent of the total number of affected companies, constitutes a ''substantial number.'' Some commenters also stated their concern that even at a less than one percent cost-to-sales ratio effect on small businesses there could be a significant economic impact. Another commenter stated that EPA had not evaluated ''reasonable worst case'' impacts for any single plant. Several commenters requested more information regarding EPA's assessment of small business impacts and steps taken to minimize the impacts.

Response: The following discussion responds to the small business impact issues raised by the commenters. In accordance with the RFA, the Agency conducted a small business assessment and based its finding of ''no significant impact on a substantial number of small entities'' on the reported impacts of the proposed NESHAP on small businesses within the cement industry (Docket Item II–A–46, Table 4–7; Docket Item IV–C–15). The Agency did not intend to suggest that this certification was based solely upon the number of small businesses potentially affected by the rule, nor that the Agency sets thresholds for determining whether a particular number of businesses is a substantial number or a particular impact is a significant impact. The EPA did not certify that the rule would have no significant impact on a substantial number of small firms based solely on there being less than 100 firms subject to the rulemaking (Docket Item II–C–14). To clarify the factual basis of EPA's determination and address subsequent comments, a summary of the Agency's

small business assessment is provided below.

Based on SBA-defined small business criteria, the Agency originally identified nine of the 44 companies within the U.S. cement industry as small businesses, or roughly 20 percent of total. However, based on updated information and changes in ownership since 1993, the Agency determined that four of these companies should not be considered small businesses. The APCA indicated that there are currently seven small businesses within this industry. This list includes the remaining five identified by the Agency plus Dacotah Cement and Royal Cement Company. Dacotah Cement is owned by the State of South Dakota and, thus, was not considered a small business by the Agency. Royal Cement Company began operations in 1995 after the Agency had completed its small business assessment and, thus, was not included in the Agency's small business assessment because EPA's engineering and economic data base did not contain information on this relatively new facility.

The Agency typically uses the cost-to-sales ratio as a measure of impact on small businesses. This ratio refers to the change in the annual control cost divided by the annual revenue generated from sales of the particular good or goods being produced in the process for which additional pollution control is required. It can be estimated for either individual firms or as an average for some set of firms such as affected small companies. While it has different significance for different market situations, it is a good rough gage of potential impact. In this case, to develop the cost-to-sales ratios, the Agency used the estimated control costs specific to the kilns operating at each manufacturing plant owned by a small business divided by their baseline cement sales. Contrary to industry's comments, the cost-to-sales measure of impact used by the Agency is a conservative approach and may, in fact, overstate the regulatory burden on small businesses for two reasons: (1) The Agency's sales estimate understates company sales because it only reflects cement operations and most companies have other vertical or horizontal business lines; and (2) this measure does not account for the expected market adjustments, i.e., increase in market prices that can potentially offset a portion of the regulatory costs.

For the economic impact analyses, the regulatory control costs were input to an economic model to predict outcomes at the market and plant level, including the impacts for markets served by

manufacturing plants owned by small businesses. As shown in Table 4–7 of the EIA report (Docket Item II–A–46), the Agency did not project any plants or kilns owned by the original nine small businesses to close as a result of the proposed NESHAP.

As summarized in the Agency's June 10, 1998, letter to industry (Docket Item IV–C–15), a second small business assessment was conducted for the small businesses identified by the APCA. The weighted average cost-to-sales ratio for these small businesses was 0.93 percent with no plants or kilns projected to cease operations (Docket Item IV–B–5).

A third small business assessment was conducted to include the cost of PM CEMs and the monitoring of materials handling operations. (The promulgated rule requires the installation of PM CEMs, and more frequent monitoring of materials handling operations than included in the proposed rule. See Section IV and this section for further discussion of these requirements). The new weighted average cost-to-sales ratio for the small businesses was 1.4 percent with no plants or kilns projected to cease operations. See Docket Item IV–B–11 for the resulting company-specific cost-to-sales ratios for this third analysis. Further, to measure the relative regulatory burden on small businesses, these impacts at small businesses can be compared to those for the whole industry. See Docket Item IV–A–4 for this comparison.

As discussed above, based on the Agency's revised small business impacts assessments, which now include the cost of PM CEMs and other monitoring costs not considered at proposal, the Agency concludes that this NESHAP as promulgated today will not have a significant impact on a substantial number of small businesses. Nevertheless, EPA will reassess, as appropriate, small business impacts in the future proposed rulemaking that will establish the date that PM CEMs must be installed on NHW cement kilns.

Comment: One commenter stated that EPA must have objective, reasonable certainty that there will be no pertinent impacts on small entities or it cannot validly certify. The EPA must create a testable record against which the validity of certifications could be judicially reviewed. 5 U.S.C. 611(a) and (b). The commenter further claimed that EPA's SBREFA Guidance states that when EPA ''cannot or does not certify that a proposed rule will not have a significant impact on a substantial number of small entities, it must prepare a regulatory flexibility analysis for the proposed rule.'' The commenter

does not believe EPA has met this burden for the proposed rule.

Response: Section 605(b) provides an exemption from the requirements in sections 603 and 604 to conduct a regulatory flexibility analysis when the Agency ''certifies that the rule will not, if promulgated, have a significant economic impact on a substantial number of small entities.'' The EPA has made this certification for the rulemaking. The EPA believes its interpretation of the requirements of the RFA is reasonable and that its factual basis for certification is also reasonable.

To the extent that the commenter is suggesting that the RFA requires more than a reasonable basis for its decision to certify, the EPA disagrees. Courts review compliance with the RFA in accordance with Chapter 7 of the Administrative Procedure Act (APA), 5 U.S.C. 701, *et seq.* See 5 U.S.C. 611(a)(1) and (2). Under the APA, courts generally provide substantial deference to agency decisionmaking and will only set aside administrative actions or findings if the court concludes that the agency's action or finding was arbitrary, capricious, or otherwise contrary to law. 5 U.S.C. 706(2)(A). The Supreme Court has explained, ''To make this finding the court must consider whether the decision was based on consideration of the relevant factors and whether there has been a clear error of judgement.'' *Citizens to Preserve Overton Park* v. *Volpe,* 401 U.S. 415 (1971). The EPA believes that its detailed economic analysis more than adequately supports its conclusion that the rule will not result in a significant impact on a substantial number of small entities.

Comment: The same commenter believes SBREFA can only be interpreted to allow numerical cutoffs based on the percentage of all small entities in the regulated universe that experience any impact. The commenter contends that when a rule impacts all the small entities in an industry, the statute a fortiori requires an analysis of whether those impacts are significant, and precludes a certification based solely on any absolute number of small entities impacted. By the same token, if the percentage of small entities experiencing any impact is more than *de minimis,* a similar analysis appears required. The commenter contends that this concept has been repeatedly recognized by EPA findings that impacts on more than 20 percent of the small entities within a universe proposed to be regulated constitute a ''significant number.'' 61 FR 48206, 48228 (September 12, 1996); 59 FR 62585, 62588 (December 6, 1994). It also lies at the heart of the ''impacts'' matrix in

EPA's SBREFA Guidance. The commenter notes that under that matrix, greater ''impact'' priority is assigned to rules that will impact a larger percentage of small entities, even if the impacts are relatively low.

Response: Other than small entities, the RFA does not define the term, or any part of the term, ''significant impact on a substantial number of small entities.'' Thus, the statute does not specify whether an agency may properly certify a rule either because there is not a significant impact on small entities, or because, even if the impact is significant, there are not a substantial number of small entities affected. In any event, the EPA has chosen not to establish any mechanistic approach for determining when an impact is significant or when the number of small entities is substantial. Instead the EPA considers a variety of approaches depending on the particular circumstances of the rulemaking. In general, the EPA looks at both the extent of the potential impact and the number of small entities impacted to decide whether a more detailed regulatory flexibility analysis pursuant to sections 603 and 604 of the RFA is warranted. The EPA's Guidance repeatedly explains that the criteria offered in the Guidance cannot be applied mechanistically and that rule writers should consider other relevant information in deciding whether or not to certify a rule.

EPA's analysis of both the number of small entities impacted and the extent of that impact are described in previous responses in this section of this preamble, and as indicated above, the EPA has not certified this rulemaking based solely on the number (or percentage) of small entities.

Economic Impact Analysis

Comment: Several commenters believe that the final EPA economic analysis at proposal was inaccurate and should be either revised to reflect industry's comments (in Attachment G to docket item IV–D–26) or withdrawn. Another commenter stated that EPA's model economic impacts data are seriously flawed because:

1. The model would not detect company-level impacts.

2. The economic analysis is not based on any estimate or analysis of actual small-entity impacts but is based on an aggregated industry wide economic model based on theoretically constructed model kilns.

3. The model predicts that older smaller dry kilns will close, which is counterintuitive because wet kilns are

substantially more costly to operate per unit of product.

4. Flaws in the market-specific part of the model which lead directly to the modeled conclusion that profits will increase with more stringent control.

Response: The EPA disagrees with the preceding comments suggesting the analysis is inaccurate and should be withdrawn. The Agency developed its economic analysis based on the best available information using an accepted approach firmly rooted in economic theory to provide the necessary impact results to satisfy legislative and administrative requirements. Furthermore, the Agency conducted a revised economic impact analysis in response to the additional monitoring requirements for cement kilns and materials handling operations at major source cement plants (as fully described in Appendix G recently added to the July 1996 EIA report, Docket Item II–A– 46). In conducting this revised analysis, the Agency also updated the original 1993 baseline information that supported the economic analysis for proposal to 1995 and is thereby consistent with the baseline used by the Agency for the Cement Kiln Dust (CKD) rulemaking and Hazardous Waste Combustion MACT Standards. This adjustment to the baseline characterization results in some differences in the projected economic impacts from the proposal analysis. In particular, under 1995 baseline conditions, the model predicts an aggregate loss in industry profits because of the sharp reduction in excess U.S. cement capacity from 1993 to 1995. This increase in capacity utilization to roughly 94 percent in 1995 severely limits the ability of unaffected (and slightly affected) domestic producers to offset production declines at affected cement plants. As a result, the potential profit gains to these producers from offsetting these reductions is no longer present in 1995 as in 1993 and the economic model predicts an aggregate loss in pre-tax earning of the U.S. industry, which is consistent with the expectations of the commenter. However, this occurs through the difference in baseline characterization rather than flaws in the Agency economic model and approach.

The following responses address the above comments that are specific to the economic analysis conducted for the regulation as originally proposed. First, the comments are specific to a draft version of the EIA report that has been revised. Comments were addressed in changes to the analysis prior to proposal as follows:

1. As the commenter suggested, the economic model incorporated a more realistic assumption for the elasticity of supply from foreign imports.

2. According to the commenter the draft EIA report did not adequately describe the basis for defining the regional markets used in the economic analysis and led to some confusion and/or misinterpretation by the industry as reflected in its comments. Contrary to assertions, the Agency's economic model does not omit any market areas as all U.S. production and consumption of cement is accounted for within the 20 regional markets as defined by the Agency. The Agency utilized the best available information in defining regional markets to better account for the regional competition within the industry.

3. The commenter claimed the draft EIA report did not adequately describe the basis for selecting the imperfectly competitive market structure for the cement industry and the implications of this selection of the economic impact results. The Agency's selection of market structure was not an attempt to distort the economic impact results or to infer that the industry is collusive and lacks any competition. Rather it was selected to provide better estimates given well-known characteristics of the industry. The Agency has appropriately modeled the competitive interaction between domestic producers of cement as well as foreign imports (where applicable) within each regional market in a manner that is consistent with the empirical evidence for cement markets and economic theory.

In regard to the statement that the economic impact data are flawed and accompanying reasons, the Agency responds as follows:

1. The economic impact analysis does allow the Agency to detect company-level impacts by aggregating the estimated control costs and related economic impacts at all manufacturing plants owned by each company, both large and small. Although the issue of capital availability is an important consideration for small businesses, it is not typically addressed in EPA economic analyses of regulatory actions as it requires company-specific information not available to the Agency and, moreover, there is not a generally accepted method with which to model and analyze this complex issue in the context of environmental regulation.

2. The Agency's characterization of costs at individual kilns was based on the econometric estimation of cost functions for cement kilns by Das (1991 and 1992). Using the best information available, the EPA made adjustments to

these cost functions to better reflect the operating costs of kilns by process type and capacity (as fully described in Appendix C, Docket Item II–A–46). However, in accounting for size or economies of scale in estimating baseline operating costs, the Agency was limited by the two capacity size classifications of less than and greater than 500,000 short tons per year for which labor productivity and fuel consumption were reported by the Portland Cement Association. This data limitation prevents the EPA from developing baseline cost functions for very small kilns and, effectively, "lumps smaller kilns in with mid-size kilns into a larger class" of all kilns as stated by industry. Therefore, it is possible that the EPA's economic model understates the baseline operating costs at very small kilns. However, the Agency is able to estimate the incremental compliance costs for many categories of kiln capacity below 500,000 short tons per year ranging from 55,000 to 450,000 short tons per year. This more detailed classification scheme for estimating the regulatory compliance costs reduces the uncertainty related to the Agency's estimates of kiln closures.

3. The Agency agrees with the industry comment that wet kilns are generally more costly to operate, which has contributed to their use of hazardous waste to reduce their fuel costs and remain competitive with the dry process kilns, especially those using precalciner and/or preheater technologies. However, the economic impacts of the proposed NESHAP depend not only on the baseline costs of cement production but also on the incremental costs of compliance for each kiln. The proposed NESHAP largely impacts non-hazardous waste burning kilns as opposed to hazardous waste kilns that are most often wet process kilns. As stated in the EIA report, it is the higher relative incremental cost impact compared to that for its competitors that causes the Agency's model to project closure for two dry process kilns under the proposed NESHAP. Furthermore, the baseline costs of cement production were high for these kilns because they were each older and smaller than average. Thus, the projected closures are actually consistent with the commenter's statement that older and smaller kilns are more vulnerable to closure with regulation. Moreover, in the final EIA report, the Agency provides closure estimates for additional regulatory alternatives and, for more stringent "above-the-floor" alternatives, the economic model

projects up to 10 kilns to close including 5 wet process kilns. Thus, the Agency believes that its economic model produces closure estimates that are consistent with the commenter's characterizations.

4. Although the Agency projects a net increase in profits for the cement industry as a whole in response to regulation, there is a "social cost" to reducing hazardous air emissions from the manufacture of cement. As shown in the final report, the Agency estimates that society must give up $34.5 million per year for the expected environmental benefits (as compared to the $28.8 million in regulatory compliance costs incurred by industry after market adjustments). Furthermore, factors cited by industry are not the reason for the model's prediction of a net increase in profits for the industry as a whole. The Agency believes that it has appropriately modeled the competitive interaction between domestic producers of cement as well as foreign imports (where applicable) within each regional market in a manner that is consistent with the empirical evidence for cement markets and economic theory.

Related to the net increase in profits for the industry as a whole, several commenters were surprised that the economic analysis predicts an increase in cement plants' pretax earnings. They interpreted this as applying to individual plants, which is a misinterpretation. The economic analysis projects a net increase in the U.S. cement industry's pre-tax earnings, which reflects profit gains at unaffected or relatively less affected cement plants and profit losses at affected plants that incur higher relative compliance costs. Thus, the commenter's statement that each cement plant's pre-tax earnings will increase by X dollars for every dollar spent on compliance is incorrect as these impacts are distributed across different plants. Also, the estimated price increase applies to all cement produced by U.S. manufacturing plants whereas the MACT compliance costs apply only to cement produced at affected plants. Therefore, the commenter's calculation of the projected price increase as a share of MACT compliance costs is also incorrect as the commenter is understating the relevant change in cost by dividing the MACT compliance costs by all cement produced rather than only the affected share of cement production. The projected increase in pre-tax earnings is a net result for the industry that results from losses at some cement plants that are offset by gains at other cement plants.

PM CEMs

Comment: Numerous comments were received stating that the EPA has not fully considered the impacts of a potential requirement for PM CEMS applied to NHW kilns, and that PM CEMs have not been adequately demonstrated on cement kilns.

Response: In the preamble to the proposal, EPA noted its intent to include a requirement for PM continuous emission monitoring system (CEMS) in the final rule, unless the analysis of existing or newly acquired data and information showed that it is not appropriate (see 63 FR at 14205). Based on successful testing on an incinerator conducted in the interim, as well as extensive use of these monitors in Europe, EPA believes there is sound evidence that PM CEMS should work at cement kilns. In addition, preliminary analyses of the cost of PM CEMS applied to cement kilns (docket items IV–C–1 and IV–C–21) and hazardous waste combustors (HWC) suggest that these costs are reasonable. Accordingly, the final rule contains a requirement to install PM CEMS. However, we agree with comments that indicate a need to develop cement kiln-specific performance requirements for CEMS and to resolve other outstanding technical issues. These issues include all questions related to implementation of the CEM requirement (i.e. relation to all other testing, monitoring, notification, and recordkeeping), relation of the CEM requirement to the PM emission standard, as well as technical issues involving performance, maintenance and correlation of the CEM itself. These issues will be addressed in a subsequent rulemaking. Therefore, we are deferring the effective date of this requirement pending further testing and additional rulemaking. As a result, in today's final rule, EPA is requiring that particulate matter continuous emission monitoring systems (PM CEMS) be installed at cement kilns. However, since the Agency has not finalized the performance specifications for the use of these instruments at cement kilns or resolved some of the technical issues noted above, we are deferring the effective date of the requirement to install, correlate, maintain and operate PM CEMS until these actions can be completed. The PM CEMS installation deadline will be established through future rulemaking, along with other pertinent requirements, such as final Performance Specification 11, Appendix F Procedure 2. It should finally be noted that EPA has a concurrent rulemaking process underway for hazardous waste

combustors (HWC) and plans to adopt the same approach in that rule.

EPA also is taking action now to avoid facilities being in violation of the PM standard during CEM correlation testing. Commenters properly observed that CEM correlation testing would require sources to manipulate their PM control device during correlation tests to obtain higher PM emissions levels than the emission limit. It is necessary to do so because a good PM CEMS correlation must include CEMS and manual method data above the stated emission standard in order to have a wide enough range of data to meet the correlation coefficient statistical requirement and to assure that calibrated readings above the level of the emission standard can be properly interpreted. Such data, however, could be misconstrued by state or local enforcement authorities or citizens as violations of the PM standard. It is important to address this issue now to encourage the development of additional PM CEMS data, and not to discourage facilities from choosing to install a CEM before the deferred effective date.

We are addressing this concern here in the same manner we plan to address it in the HWC MACT rule by providing that the particulate matter and opacity standards of parts 60, 61, 63 (i.e., all applicable Parts of Title 40) do not apply during particulate matter CEMS correlation testing, provided that you comply with certain provisions discussed below that ensure that the provision is not abused. EPA is also making this provision effective immediately, so that sources need not wait for the compliance date to take advantage of this particulate matter CEMS correlation test provision. We believe this approach adequately addresses commenters' concerns.

The temporary exemption from particulate matter and opacity standards is conditioned on several requirements. Sources are required to develop and submit to permitting officials a PM CEMS correlation test plan along with a statement of when and how any excess emissions will occur during the correlation tests (*i.e.,* how you will modify operating conditions to ensure a wide range of particulate emissions, and thus a valid correlation test). If the permitting officials fail to respond to the test plan in 30 days, the source may proceed with the tests as described in the test plan. If the permitting officials comment on the plan, the source must address those comments and resubmit the plan for approval. In addition, runs that exceed any PM or opacity emission standard are limited to no more than a total of 96 hours per correlation test.

This 96 hours is sufficient time for a source to increase emissions to the desired level and reach system equilibrium, conduct testing at the equilibrium condition followed by a return to normal settings indicative of compliance with emissions standard(s) after those higher emissions data have been obtained, and return to equilibrium at normal conditions. Finally, to ensure these periods of high emissions are due to the bona fide need described here, a manual method test crew must be on-site and making measurements (or in the event some unforeseen problem develops, prepared to make measurements) at least 24 hours after you make equipment or workplace modifications to increase PM emissions to levels of the high correlation runs.

Selection of Emission Limits in General

Comment: One commenter stated that according to section 112(d) EPA may not base the floors of its emission standards on a particular technology. Instead, emission standards for existing sources must be no less stringent than "the average emission limitation achieved by the best performing twelve percent of the existing sources" (for which EPA has data). The commenter further stated that for new sources, standards must be based on the emission control that is achieved in practice by the best controlled similar source. Thus, the standards proposed for emissions of dioxins, mercury, total hydrocarbons, and hydrogen chloride are not valid.

Response: First, it should be noted most of the commenter's points were recently rejected by the DC Circuit in *Sierra Club* v. *EPA* (March 2, 1999). That case holds that because MACT standards must be achievable in practice, EPA must assure that the standards are achievable "under most adverse circumstances which can reasonably be expected to recur" (assuming proper design and operation of control technology). Slip op. p. 13. The case further holds that EPA can reasonably interpret the MACT floor methodology language so long as the Agency's methodology in a particular rule allows it to "make a reasonable estimate of the performance of the top 12 percent of units", slip op. p. 7; that evaluating how a given MACT technology performs is a permissible means estimating this performance, id. at 13; and that new source standards need not be based on performance of a single source, id.

Second, the commenter provided no additional emissions data for any pollutant. The EPA has selected emission limits at the floor level of

control. Section 112(d) requires EPA to promulgate emission standards based on what is determined to be achievable through the application of techniques, methods, etc. The rule does not require the use of any specific technology to meet the emission standard. The emission standards are based on the emissions levels achieved through the application of MACT floor technologies and account for variation in the process and in the air pollution control device effectiveness.

Although the commenter did not specifically mention PM, the following discussion using PM as an example will help clarify EPA's approach in setting MACT standards for this source category. The EPA evaluated the PM MACT floor technology for both existing and new sources at proposal and determined that the MACT floor technology is properly designed and operated FFs and ESPs. Commenters provided no data to suggest that a particular design or operating mode, or an alternative technology could achieve a lower level of PM emissions on a consistent basis. Nor did EPA identify other technologies for existing or new kilns or in-line kiln/raw mills that would consistently achieve lower emission levels of PM than the NSPS limit.

As discussed in docket item number IV–B–10, the data upon which the MACT floor was based were obtained from EPA Method 5 compliance tests on kilns subject to the NSPS and represent performance of PMCDs associated with new kilns over a relatively short period (typically three 1-hour test runs). These test data were obtained at kilns equipped with well designed and operated ESPs and FFs representative of the MACT floor, which is also represented by the NSPS emission level. Method 5 testing of these cement kilns equipped with MACT floor technology showed a range of emissions up to the NSPS level. Additional Method 5 tests performed on some of the same kilns included in the MACT floor analysis showed PM variations after control as plotted in docket item IV–B–10. EPA believes that the data base—which shows cement kilns with properly designed and operated fabric filters and electrostatic precipitators achieving levels up to and including the NSPS level—adequately accounts for the variability inherent in the air pollution control technologies, and indicates what PM levels are consistently achievable in practice. See Sierra Club, slip op. p. 13. In summary, the PM emission limit reflects an emission level consistently achievable with the use of well designed and operated MACT floor technology.

The emission standard for dioxin is based on the emission level achievable through the application of the MACT floor control technology, which is exhaust gas temperature control at the inlet to the PM control device to less than 400° F, and efficient combustion. Based on data evaluated at proposal, the technology can be represented by the dual standard of 0.2 ng TEQ/dscm or 0.4 ng TEQ/dscm with a PM control device inlet temperature of 400° F or less. Since the commenter provided no additional data, the EPA has reviewed, in response to this comment, the existing test data and literature on D/F formation and concluded that the selected emission limits are consistently achievable and represent the MACT floor. Similar to the discussion above regarding the PM data, the D/F performance test data are based on short-term tests of facilities using the MACT floor technology. Thus the proposed emission limits are retained and account for normal, inherent process and air pollution control operating variability, including the use of various fuels.

As discussed in the proposal preamble, there are no standards for THC emissions from existing sources because the MACT floor for control of THC for existing sources is no control. Further, the BTF control technique for existing sources, and a floor control for new sources, would be based on the performance of precalciner/no preheater technology. However, as discussed in the proposal, EPA rejected this technology as a basis for setting THC emission limits because of the technology's negative environmental and energy impacts. The basis for the THC limit for new greenfield kilns is site selection to ensure low hydrocarbon content in feed materials. (In the proposal, the THC limit applied to all new kilns, but based on comments received, the rule has been changed such that the THC limit will only apply to new greenfield kilns. See comment responses regarding this issue for more detail.) As discussed in the proposal, this option is not available to existing (and new brownfield) kilns, in that facilities are generally tied to existing raw material sources in close proximity to the facility, so that raw material proximity (*i.e.*, transportation cost) is usually a major (indeed, critical) factor in plant site selection.

As discussed in the proposal preamble, no standards are being adopted for Hg and HCl because the MACT floor has been determined to be no control and the BTF controls are not cost effective (docket item II–B–67).

This standard was developed under section 112, not section 129, so there is

no statutory requirement to establish standards for individual HAP metals. However, control of cadmium, lead, and other non-volatile and semi-volatile metal HAPs is achieved via the floor level-based emission limit for PM, which serves as a surrogate for the non-volatile and semi-volatile metals. This is supported by data from coal-fired electric utility boilers which show relatively high HAP metals (except mercury) removal with fabric filters and electrostatic precipitators. (Study of Hazardous Air Pollutant Emissions from Electric Utility Steam Generating Units—Final Report to Congress, volume 1, 453/R–98–004a, February 1998, p. 13–23 and 13–26).

PM Limits

Comment: Numerous commenters supported the use of PM as a surrogate for non-volatile HAP metals. One commenter questioned the use of PM as a surrogate for HAP metals, and suggested that the EPA require stack testing for specific metal content.

Response: The final rule retains the use of PM as a surrogate for HAP metals because the MACT floor equipment and level of control for HAP metals, *i.e.*, properly designed and operated fabric filters (FFs) and electrostatic precipitators (ESPs), is identical to that for PM. Using PM as a surrogate for specific HAP metals eliminates the cost of performance testing to comply with numerous standards for individual metals, and achieves exactly the same level of HAP metal emissions limitation.

Comment: Although many commenters were in favor of the MACT floor determination and associated emission limit for PM (see docket item, number to be assigned), several other commenters suggested that more stringent PM standards were required in recognition of the performance test data presented in the preamble showing that many affected sources achieved lower levels of PM emissions than the proposed standard.

Response: The proposed PM standards have been retained in the final rule. EPA evaluated the MACT floor technology for both existing and new sources at proposal and determined that the MACT floor technology is properly designed and operated FFs and ESPs. Commenters provided no data to support that an alternative design or technology represents a floor that could achieve a lower level of PM emissions on a consistent basis. The EPA did not identify other technologies for existing or new kilns or in-line kiln/raw mills that would consistently achieve lower emission levels of PM than the NSPS limit.

As discussed in the proposal preamble, the data upon which the MACT floor was based were obtained from EPA Method 5 compliance tests on kilns subject to the NSPS and represent performance of PMCDs associated with new kilns over a relatively short period (typically three 1-hour test runs). These test data were obtained at kilns equipped with well designed and operated ESPs and FFs representative of the MACT floor, which is also represented by the NSPS emission level. Method 5 testing of these cement kilns equipped with MACT floor technology showed a range of emissions up to the NSPS level. Additional Method 5 tests performed on some of the same kilns included in the MACT floor analysis showed PM variations after control as plotted in the reference, confirming that some operating variability is inherent. EPA believes that these data reasonably represent levels achievable in practice by the average of the best performing 12 percent of sources, and by accounting adequately for variability, further assure that the standard will be achievable under the worst forseeable circumstances consistent with proper design and operation. Sierra Club, slip. op. p. 13. In summary, the PM emission limit reflects an emission level consistently achievable with the use of well designed and operated MACT floor technology.

Comment: One commenter stated that it is feasible, both technically and economically, for portland cement kilns to use fuels and raw materials with low metals content. Because feed limits are an achievable measure that would further reduce emissions, EPA must require them.

Response: Feed and/or fossil-fuel switching has not been undertaken by any NHW kilns to reduce metals emissions, and therefore this is not a MACT floor option.

The use of feed material selection and feed material blending to achieve lower metals emissions thus is a potential beyond-the-floor technology. Cost is a consideration in the decision to go beyond-the-floor. The ability of a facility to remain cost competitive typically depends on the use of raw materials mined in close proximity to the facility. Several commenters described the economic difficulties in locating, purchasing, and transporting feed materials to existing sites; the comment to the contrary stated the opposite categorically, but provided no supporting cost, economic or technical data. See Sierra Club, slip op. p. 13 (rejecting argument that pollution prevention measures had to be included as part of a standard where costs were

not adequately quantified). EPA disagrees with this comment. Cement kilns require enormous amounts of raw material, and the costs of transporting the raw material are enormous, given the volumes involved. Finding a new source of raw material will often (if not invariably) entail more costs because the source of the raw materials will be further from the facility. The Agency believes that in many cases a facility could not even remain economically viable were existing sources of raw material to become unavailable. In many cases, costs of the change in raw material would exceed air pollution benefits.[2]

In the case of NHW kilns, fuel switching is not a demonstrated metals control technology. There are no data available to EPA that indicate that this technology can or has achieved metals emission reductions from NHW kilns. A HW kiln operator can control metals via the hazardous waste fuel, but this is not an option available to NHW kiln operations.

D/F Limits

Comment: Several comments were received regarding the D/F limits in the proposed rule, which were based on the MACT floor. Some commenters suggested that a lower D/F emission limit was appropriate for both new and existing sources, based on the performance test data reported in the proposal preamble. Other commenters felt that the proposed emission limit was too stringent and unjustified, and was not representative of the MACT floor technology. Many other commenters supported the proposed standards.

Response: In response to these comments, the EPA has reviewed the existing test data and literature on D/F formation and concluded that the selected emissions limits represent the MACT floor and are consistently achievable. Again, EPA is influenced by the fact that cement kilns using the floor control technology achieved different D/

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[2] As discussed above, EPA considered control of feed materials as a potential beyond the floor technology. EPA is aware of the Conference Report to the 1990 amendments which state that controls on feed materials are not to be part of MACT for mineral processing facilities. H.R. Rep. No. 952, 101st Cong., 2d sess. 339. However, the text of the statute does not reflect this legislative history, stating unambiguously that MACT for all sources includes eliminating HAP emissions through "substitution of materials". Section 112 (d) (2) (A). EPA is following the explicit statutory text in considering (albeit rejecting) feed control as a potential beyond the floor technology in this rule. At the very least, this is a permissible interpretation of the statute, given the statutory goal of protecting and enhancing of the Nation's air resources. Section 101 (b)(1).

F levels in their performance tests— indicating that different levels reflect normal variability of the process and control technology. Consequently, EPA is retaining the proposed standard for D/ F emissions from kilns and in-line kiln/ raw mills in the final rule.

In order to establish a more stringent emission limit for new kilns, it is necessary to identify a different technology to which better performance is attributable. Since EPA could not identify a different technology for new kilns, the standard is based on the range of available data, considering process and control variability.

The EPA determined that the MACT floor technology for both existing and new sources was inlet PM control device temperature control to 400° F accompanied by good combustion and process control. Based on data evaluated at proposal, the technology can be represented by the dual standard of 0.2 ng TEQ/dscm or 0.4 ng TEQ/dscm with a PM control device inlet temperature of 400° F or less. The performance test data are based on short-term tests but do indicate that all kilns will achieve the numerical emission limit of 0.4 ng TEQ/ dscm with the application of the floor technology. Thus the 0.4 ng TEQ/dscm emission limit is retained to account for normal inherent process and air pollution control operating variability, including the use of various fuels, such as tires.

THC Limit

Comment: Several comments were received questioning the specification of a THC standard for reconstructed kilns or new kilns built at existing sites. Commenters asserted that these facilities could not economically locate, purchase and transport suitable feed materials to meet this standard.

Response: In recognition of these comments, the final rule has been changed to make the THC limitation applicable only to greenfield kilns, greenfield in-line kiln/raw mills and greenfield raw material dryers. EPA agrees that only greenfield sources would be able to apply MACT, which is the site selection of feed materials with low levels of naturally occurring organic material. The EPA considered the use of precalciner/no preheater kilns for THC control, (docket items II–B–47, II–B–48, II–B–67, and II–B–76), but concluded that because of negative energy impacts and increased emissions of criteria pollutants these did not provide the maximum achievable control technology for either existing or new sources. Further discussion of this technology is provided in the response to the next comment.

Comment: Commenters stated that the proposed rulemaking provides no justification or insufficient support for the selection of 50 ppmvd as the total hydrocarbon (THC) standard for new or modified kilns. Another commenter noted that EPA has recognized that portland cement kilns use a variety of methods and technologies to control their THC emissions, including precalciner/no preheater technology and a combination of feed material selection, site location, and feed material blending. All of these methods and technologies are reflected in existing sources' actual performance, on which EPA must base the floors for its THC standard. That commenter stated that under section 112(d) the THC emission standard would be much lower than 50 ppmvd.

Response: First, with regard to the methods and technologies determined to be the MACT floor, the ''precalciner, no preheater'' kiln is not considered maximum achievable control technology when other considerations such as energy impacts and NO$_X$ emissions are taken into account. As explained in the preamble to the proposed rule, EPA believes that use of these technologies would not be MACT for new or existing sources because of the adverse environmental impacts associated with these technologies' use, in particular increased emissions of certain criteria pollutants. See *Portland Cement Assn* v. *Ruckelshaus,* 486 F. 2d 375, 385–96 (D.C. Cir. 1973) (if use of a particular technology results in other, adverse environmental consequences, that technology need not be considered the ''best''). The proposal preamble also addressed consideration of feed material selection for existing sources as a MACT floor technology and concluded that this option is not available to existing (and new brownfield) kilns, in that facilities are generally tied to existing raw material sources in close proximity to the facility, and that raw material proximity (*i.e.,* transportation cost) is usually a major factor in plant site selection. This conclusion was supported by several commenters. The commenters described the economic difficulties in locating, purchasing, and transporting low organic feed materials to existing sites. However, for new ''greenfield'' kilns, feed material selection as achieved through appropriate site selection and feed material blending is considered new source MACT.

With regard to the level of standard, it is based upon data available to the Administrator and no data were provided after proposal which would justify a different standard. Based on a

review of available information (docket item II–B–62, docket item II–B–75, docket item II–D–195) the EPA believes that a THC concentration of 50 ppmvd represents a level that is achievable nationwide across a broad spectrum of feed materials. This level has been retained in the final rule.

Comment: Comments were received concerning the suitability of THC as a surrogate for organic HAP, in light of the high variability in the ratio of organic HAP to THC in cement kiln exhaust gas.

Response: The EPA recognizes the variability of the data but concludes that when speciated analyses of THC were undertaken organic HAPs were found to be present. No attempt was made to correlate organic HAP emissions with THC emissions. Because of the cost savings to industry in conducting performance tests to establish compliance with a THC standard, EPA has chosen not to set standards for individual speciated organic HAPs. Further, since the source of organic HAPs is the same source as for THC (feed materials), using MACT will also control organic HAP emissions. Adopting THC as a surrogate will result in cost savings to the cement industry and to the EPA during compliance testing and monitoring.

The EPA notes further that the same issue was presented when EPA adopted standards for boilers and industrial furnaces burning hazardous waste, and in the course of that rulemaking, not only the Agency but the Science Advisory Board concluded that THC was indeed a reasonable surrogate for toxic organic emissions from cement kilns. [See 56 FR at 7153–54 (Feb. 21, 1991).]

The proposal preamble stated that POM, one of the seven pollutants listed in section 112(c)(6), would be regulated using THC as a surrogate. The final source category listing notice for section 112(d) rulemaking pursuant to section 112(c)(6) requirements shows the NHW kiln facilities portion of the portland cement source category to be a significant source of POM (63 FR 17838, April 10,1998). For this reason, and to control other THC HAPs, the final rule limits emissions of THC from new greenfield raw material dryers and new greenfield kilns and greenfield in-line kiln/raw mills at area sources as well as major sources.

Mercury Limit

Comment: Comments were received concerning the need for an emission standard to limit the emissions of mercury from NHW cement kilns. Other commenters suggested that a mercury standard be established based on a

presumed floor or beyond the floor basis of fuel and/or feed material control, referring to the proposed Hazardous Waste Combustor rules and research on clean coal to reduce mercury emissions in the electric utility industry. Other commenters agreed with EPA's determination for no mercury emission limit.

Response: The EPA determined, at proposal, that the MACT floor for both new and existing sources was no control. The EPA evaluated activated carbon injection as a beyond the floor alternative for control of mercury emission from NHW kilns and in-line kiln/raw mills, and this technology was not found to be cost effective. Feed and/ or fossil-fuel switching or cleaning has not been undertaken by any NHW kilns in order to reduce mercury emissions, and therefore these are not MACT floor options. For this reason feed and/or fossil-fuel switching or cleaning would be considered a beyond the MACT floor option but the EPA does not have data, nor did commenters provide data, that show that this option would consistently decrease mercury emissions. Moreover, as noted earlier, raw material feed control is prohibitively costly for this industry.

The proposed rule for Hazardous Waste Combustors included a standard of mercury. However, control of mercury in that rule would be based on controlling the amount of mercury in the hazardous waste fuel, not controlling raw material or fossil fuel. This approach is thus not available to NHW kilns. In addition, based on the Electric Utility Report to Congress on HAP emissions, EPA believes that fuel switching among different coals and from coal to oil would not consistently reduce HAP metal emissions from cement manufacturing plants. (Study of Hazardous Air Pollutant Emissions from Electric Utility Steam Generating Units—Final Report to Congress, volume 1, 453/R–98–004a, February 1998, pp. 13–1 through 13–5.) Therefore, this final rule establishes MACT for mercury as no control. However, EPA will be performing research and development work with the objective of finding more cost effective methods to reduce mercury air emissions from fossil-fuel fired electric utilities, and EPA will in the future consider whether any more cost effective methods may be appropriate as a basis for reducing mercury emissions from NHW cement kilns.

Hydrogen Chloride Limit

Comment: Comments were received stating the need for an emission standard for HCl emissions from kilns

because EPA did not provide data to show that HCl emissions pose no threat to public health and that HCl is emitted in large quantities from new and existing NHW kilns. Other commenters stated that EPA appropriately concluded that there is no basis for a MACT standard for HCl.

Response: With regard to the threat to public health comment, the EPA is conducting this rulemaking under section 112(d)(2) and therefore the decision on an emission standard is not based on health risk. Impacts to public health will be studied and addressed later under section 112(f) of the Act. The EPA determined, at proposal, that the MACT floor for both new and existing sources was no control. Further, no cost effective beyond the floor alternatives were identified. The commenters provided no new information on the use of any control technologies to limit emissions of HCl from NHW kilns. For this reason no emission standard is being established for HCl.

Opacity Limit

Comment: One commenter requested that EPA clarify the duration of both the performance test and continuous compliance demonstrations for opacity emissions.

Response: The opacity requirements in the final rule have been changed to provide for compliance on the basis of average opacity for each and every 6-minute block of operating time. This is consistent with the NSPS which is the MACT floor level of PM control upon which the standard is based. (The proposed rule incorrectly required a thirty-minute averaging time for demonstrating continuous compliance.)

Comment: Commenters expressed concern regarding the requirement to initiate a Quality Improvement Plan (QIP) and the need to track and statistically analyze opacities at levels below the standards. One commenter stated that a violation triggered by not initiating a QIP when the source was not violating an emission standard was extreme.

Response: The requirements for developing and implementing a QIP in response to a 15 percent kiln and in-line kiln/raw mill opacity trigger have been removed from the final rule. The final rule retains the opacity limit of 20 percent which if exceeded during any 6-minute period is a violation.

Comment: One commenter requested that EPA specify the scope of monitoring opacity from raw and finish mills.

Response: The EPA has clarified that the opacity limitation on gases discharged from raw mills and finish mills is restricted to the mill sweep and air separator air pollution control devices. This is consistent with the MACT floor technology for control of gases from these affected sources.

Comment: A commenter noted that the proposed rule did not specify under what conditions visual opacity monitoring should be conducted.

Response: The final rule clarifies that Method 9 (and Method 22) tests must be conducted under the highest load or capacity level reasonably expected to occur.

Comment: Numerous commenters expressed concern regarding installation, operation, calibration and maintenance of triboelectric bag leak detection systems, and that the lack of clear-cut specifications would lead to open-ended liability for owners/operators.

Response: The option for use of triboelectric bag leak detection systems for monitoring fabric filter performance is not being promulgated at this time. The EPA is presently considering this issue and may propose revised bag leak detector requirements for some source categories. Those owners or operators who want to use bag leak detection systems may petition the Administrator for approval of alternative monitoring requirements under the General Provisions.

The rule requires the owner or operator to monitor the opacity from raw mills and finish mills by conducting a daily six-minute test in accordance with Method 22, ''Visual Determination of Fugitive Emissions from Material Sources and Smoke Emissions from Flares.''

Owners or operators of raw mills and finish mills are required to initiate corrective action within one hour of a Method 22 test during which visible emissions are observed. A 30-minute Method 9 opacity test must be started within 24 hours of observing visible emissions.

D/F Monitoring

Comment: Several commenters suggested averaging periods for temperature limits shorter than 9 hours as proposed. One commenter preferred one-hour rolling averages. Two commenters preferred ten-minute averages as rationalized in the proposed Hazardous Waste Combustor Rule.

Response: As noted in section IV. Summary of Changes Since Proposal, the final rule, in response to these comments, has been changed to a shorter averaging period. The nine-hour block average period used for the monitoring of temperature (as well as the activated carbon injection rate, if applicable) has been changed to a three-hour rolling average period. The three-hour averaging time will help to limit disproportionate increases in D/F emissions that could be caused by very short periods of higher temperatures. A three-hour averaging time is reasonable because it is within the range of values the Agency could have selected, ranging from an instantaneous limit (i.e., no averaging period) up to a nine-hour averaging period.

The enforceable operating limit for gas stream temperature is derived from the temperature measured during 3 three-hour measurements of D/F emission. The three-hour rolling average temperature limit is established by taking the average of the one-minute average temperatures for each test run conducted during the successful Method 23 performance test, then averaging each test run average. Further, sources may petition the Administrator for an alternative averaging period or an alternative method for establishing operating parameter limits.

Comment: A commenter pointed out that the proposal would allow a source to conduct its D/F performance test with an inlet PM control device temperature below 400 degrees F, but after the performance test, the source would be allowed to operate its PM control device with an inlet temperature up to 400 degrees F.

Response: In drafting the proposal, the EPA did not intend to allow a source to operate its PM control device at a temperature higher than the temperature during the performance test, and so the EPA has clarified that the inlet temperature limit is established as and capped at the average temperature during the D/F performance test.

Comment: One commenter stated that the D/F standard should be coordinated with the rule for hazardous waste combustors.

Response: As was previously noted, the EPA has adopted a shorter temperature averaging time. To further achieve consistency with the D/F temperature requirements for HW kilns, the EPA is dropping the proposed provision which would have allowed the temperature limit to be established as the average temperature during the performance test plus 25 degrees F if the D/F level (during compliance testing) was below 0.15 ng/dscm. Further, new activated carbon injection operating parameters (nozzle pressure drop or carrier fluid flow rate) and averaging time have been added and changed, respectively, to be consistent with the requirements for the HW kilns.

Comment: A comment was received requesting a clarification of the

procedure for demonstrating compliance for in-line kiln/raw mills during time periods which span a change in raw mill operating status.

Response: After a transition period in which the status of the raw mill was changed from ''off'' to ''on'' or from ''on'' to ''off'', compliance with the operating limits for the new mode of operation begins, and the three-hour rolling average is established anew, i.e., without considering previous recordings.

Comment: Comments were received suggesting that combustion parameters (e.g., CO and THC) should be monitored to demonstrate compliance with the D/F standard.

Response: The final rule does not require monitoring of these parameters as a means of monitoring combustion because the EPA believes that THC and CO emissions from NHW cement kilns are largely due to formation outside of the combustion zone, i.e., due to the feed materials. Therefore THC and carbon monoxide emissions might not accurately reflect combustion conditions, therefore the EPA has not included CO monitoring requirements to ensure good combustion. However, the final rule has been changed to include a monitoring requirement for an inspection of combustion system components to be conducted at least annually.

THC Monitoring

Comment: The EPA received comments related to the use of THC monitoring as a means of controlling combustion related pollutants and, therefore, organic HAPs (see comment 6.4.1 in the Response to Comments Document).

Response: Stack THC emissions from kilns, in-line kiln raw mills, and raw material dryers result mainly from organic material within the feed and not from incomplete combustion. As a result, the suggested combustion monitoring alternatives are not relevant.

Performance Testing Frequency

Comment: The EPA received a comment requesting that performance tests be required more frequently than once every five years, citing other rules with more frequent testing requirements.

Response: The EPA selected the five year testing interval to synchronize the testing schedule with Title V permit renewals. The testing frequency for NHW cement kilns and other affected sources at portland cement manufacturing facilities has not been changed. The exception to this is the D/F performance tests. To maintain consistency with the requirements for

HW kilns, the D/F performance testing frequency has been changed to every 2 and one half years.

Definitions

Comment: Commenters requested various changes to the definitions, including those of ''alkali bypass'' and ''feed'' to reflect cement industry practices.

Response: The final rule expands the definition of ''alkali bypass'', and defines ''kiln exhaust gas bypass'' as a synonym for alkali bypass. The final rule clarifies the definition of ''feed'' to include recycled cement kiln dust, consistent with past practice in enforcement of the NSPS.

Major Source Determination

Comment: Numerous comments were received regarding the use of emissions test data and emission factors (based on data provided in the proposal docket) in determining whether a source is major for hazardous air pollutants.

Response: The need for HAP-specific test methods and the validity of data obtained by various means to determine major source status are closely related. Hence this discussion covers both aspects under the overall title of major source determination.

Although emission standards are being promulgated for PM as a surrogate for semi-volatile and non-volatile HAP metals; THC as a surrogate for organic HAPs; and D/F, each facility owner/ operator must make a major source determination that requires an estimate of the facility's potential to emit all HAPs from all emission sources. HCl and organic HAP emissions such as (but not limited to) benzene, toluene, hexane, formaldehyde, hexane, naphthalene, phenol, styrene, and xylenes are the main HAPs from the kiln that may cause facilities to be major sources, but HAPs emitted from all sources at the plant site should be accounted for in making a major source determination.

Comment: Some commenters questioned the need for accurate HCl measurements, since there is no HCl emission standard. Others stated that EPA should provide industry the choice of conducting testing for HCl with either Method 26, 321, or 322. They objected to the restriction that Method 26 could be used only if validated by Method 321 or 322. They also stated their belief that the Agency's decision regarding the negative bias of Method 26 was based on a limited set of test results and an insufficient investigation of the potential cause. Additional comments noted that Method 26 may actually give false positives due to inclusion of

chloride salts in the calculation of measured results.

Response: As discussed above, HCl and organic HAPs emissions are the main HAPs from the kiln that will cause a source to be a major source, but HAPs emitted from all sources at the plant site, including metals emissions (discussed below) should be accounted for in making a major source determination. Accurate measurements of HCl in the kiln exhaust gases are necessary for major source determination. The EPA agrees with commenters that Method 26 may have positive biases attributable to chloride salts rather than to HCl; and negative biases due to condensation and/or removal of HCl on the filter and/or in the sampling probe. Therefore, the Agency has decided that Method 26 and 26A use without concurrent validation with M. 321 or M. 322 will only be acceptable for measuring HCl from NHW kilns to confirm that the portland cement plant is a major source. M. 26 or 26A may not be used to measure HCl in the determination that the source is an area source. Only the FTIR methods may be used in the measurement of HCl if the source claims it is not a major source.

Further, as a result of technical problems encountered by the Agency with the use of draft Method 322 (based on gas filter correlation/infrared technology) in the emission testing of lime kilns (which have a matrix similar to portland cement sources) [See Section IV.F. on Additional Test Methods for a description of the technical problems], and in response to concerns expressed by the commenters, the EPA is modifying its position regarding HCl measurements using this method in promulgating the final rule.

For the above reasons, the Agency has decided that only Methods 320 and 321 will be acceptable for measuring HCl from NHW kilns if the owner/operator wishes to claim its portland cement facility is not a major source. These methods are being promulgated as part of this rulemaking.

Comment: Commenters also requested that EPA allow cement manufacturers the option of using Method 25 (in addition to Method 18 or Method 320) for testing emissions of organic HAPs. The commenters suggest that the relatively inexpensive Method 25 could be used by cement plants that have low concentrations of organic matter in the raw material mix to verify that the plant's THC emissions are less than 10 tons/year.

Response: The focus of these commenters' point is alternatives to

measurement of organic HAPs in the process of making a major source determination. However, all HAPs (organic, HCl, metals, etc.) from all sources must be included in that determination, so it is necessary to obtain data that will allow summation of all HAP emissions to compare to the 10/25 ton per year thresholds specified in section 112 of the Clean Air Act. Depending on site-specific circumstances, EPA Method 25 may not provide sufficient information to make an accurate summation. For example, a source's determination that its THC emissions based on Method 25 or 25A are less than 10 tons per year does not necessarily signify that it is an area source; the source may be a major source based on the 25 ton per year criterion when all other HAP emissions are summed with the THC. If the source's THC emissions are over 10 tons per year, the source may choose to conduct emissions tests using EPA Method 320 to make a determination of actual organic HAP emissions. However, in lieu of conducting Method 320 emissions tests, the source could use Method 25A, but the source would have to assume that the mass emission rate (as propane) from all combustion sources combined at the site is attributed to one organic HAP. This amount would then have to be compared to the 10 ton per year threshold for one HAP. To summarize, in addition to accounting for organic HAPs (either through Method 320 testing or assuming all THC is one organic HAP), accurate measurements of HCl in the kiln exhaust gases would be necessary for major source determination, as well as measurements of HAP metals (see below), to obtain data that will allow summation of all HAP emissions to compare to the 10/25 ton per year thresholds.

Comment: Another commenter requested that EPA allow the use of an alternative to what they perceived as an EPA-suggested emission factor for metal emissions, of one percent of PM emissions, to determine major source status.

Response: If after the source determines that it is not major because it does not meet either the 10/25 ton per year thresholds based on the summation of HCl and organic HAP emissions from all sources at the plant, the source would need to determine its HAP metals emissions from all sources at the facility as well, to make a determination that it is not a major source. The use of a ''one percent HAP metals in PM'' emission factor assumption will not provide definitive evidence that the source is an area source. However, the Agency

would allow sources to forego the speciated HAP metals emission tests (through the use of Method 29) if it is assumed that 1 percent of the total PM emissions from all sources at the site are metal HAPs. This assumed amount of metal HAPs emissions would be added to the amount of HCl and organic HAPs emitted (determined as described above), and this total amount would then be compared to the 25 ton per year threshold for all HAPs combined. To reiterate, each facility owner/operator must make a major source determination that requires an estimate of the facility's potential to emit all HAPs from all emission sources, accounting for HCl, organic HAPs (either through speciation of organic HAPs or assuming all THC is one organic HAP), and metals (either through speciation of metal HAPs or assuming 1 percent of PM is metal HAP), to allow summation of all HAP emissions to compare to the 10/25 ton per year thresholds.

Voluntary Consensus Standards

Comment: One commenter (IV–D–17) stated that EPA's actions (in developing and proposing the precursor to EPA Fourier Transform Infrared Spectroscopy [FTIR] test method 320) directly conflict with the guidance of and directives of the 1995 National Technology Transfer and Advancement Act and the Office of Management and Budget (OMB) Circular A–119 because: (1) the American Society of Testing and Materials (ASTM) FTIR consensus based test method is available, and (2) the EPA Emission Measurement Center (EMC) representatives were made aware of the development of the ASTM method and chose duplicative measures in developing and proposing the precursor to EPA FTIR test method 320. (The OMB Circular states specifically that ''If a voluntary consensus standards body is in the process of developing or adopting a voluntary consensus standard that would likely be lawful and practical for an agency to use, and would be developed on a timely basis, an agency should not be developing its own government unique standard and instead should be participating in the activities of the voluntary consensus standards body.'')

Response: The Agency has been actively developing extractive FTIR-based methods for HAPs since 1992. Methods 320 and 321 are direct products of this long-term effort to apply an innovative approach to emissions measurement in the form of extractive FTIR. The Agency has tested these methods in the laboratory and in the field extensively (conducting testing

at two portland cement facilities), and has conducted multiple validation tests of these methods. The Portland Cement Association (PCA), in representing various members of the regulated industry, has conducted its own series of validation tests of these methods. Actually, Method 321 was developed and validated by PCA, and has been adopted by the Agency as Method 321. Agency personnel informed ASTM in 1996 that the Agency methods were in active development, and an ASTM standard seemed redundant. Additionally, the ASTM standard has not undergone field validation, which is essential in establishing the precision and accuracy of any test method.

The Agency has conducted a review of the ASTM method. While the ASTM method is in some ways similar to Method 320, the ASTM method is not sufficiently detailed to document proper application, and does not contain the quality assurance procedures the Agency requires in compliance methods. Specifically, the ASTM method does not address specific calibration transfer standards, nor does it address the preparation of reference spectra. Therefore, EPA has determined that it is impractical to adopt the ASTM method at this time and is promulgating Method 320.

Pollution Prevention

Comment: Comments were received stating that the proposed rule did not contain measures that prevent pollution or reduce energy requirements, and suggested specific pollution prevention measures, including process modifications, taken by specific facilities.

Response: The NESHAP is written in terms of emissions standards based on MACT floor technologies and allows pollution prevention techniques to achieve compliance. The EPA considered pollution prevention options available and the basis for the standard for THC for new greenfield sites, feed material selection, is a pollution prevention measure. In addition, the final standard includes a monitoring requirement for inspection of the combustion system components of kilns and in-line kiln raw mills (an energy efficiency and pollution prevention measure) and standards for PM from product handling affected sources (which leads to improved recovery of salable product and pollution prevention). Furthermore, the final standard clarifies that recovered cement kiln dust can be included in the calculation of kiln feed (encouraging recycling, improved PM control and pollution prevention).

Control Cost Impacts and Data Evaluation

Comment: Comments were received concerning the EPA's control cost estimates, including the assumptions regarding the number of sources requiring upgrades to meet the standards for PM and D/F, and the capital expenditures necessary to meet the standard. In particular one commenter projected that capital costs would exceed the threshold which triggers Executive Order 12866. Another commenter questioned the lack of cost data on upgrades to PMCDs for material handling affected sources.

Response: The costs to achieve compliance are expected to be highly site-specific and vary significantly. The commenters did not provide any details regarding their estimates of the cost to comply, so the EPA is unable to determine whether the commenters' cost estimates were limited to those costs necessary to comply with the provisions of the NESHAP.

The EPA has reviewed cost data provided by the Portland Cement Association prior to proposal. The foundation for the cost estimates, and initial point of criticism of EPA's cost estimates, is the model plant characteristics. For example, the APCA report provided a review of the model plant characteristics and suggested that the design characteristics for each model be 20 to 25 percent higher than the annual average production rate basis for the model. In particular, the APCA report stated that the EPA model plant gas flows for wet process and long dry kilns were 25 to 30 percent too low, based on their consultant's design practice.

The EPA developed design characteristics for the model plants based on data provided to the Agency in ICRs and test reports (docket items II–B–24 and II–B–37). For a kiln with a given nominal production rate that might be found in several different plants, variations in gas flow rates would be expected. The EPA used the flow rate and production data from actual installations to develop production rate versus gas flow graphs to establish the model plant characteristics. Owners may elect to design their upgrades or new equipment to accommodate higher production rates, but those costs and other impacts are not attributable to compliance with the MACT standards. EPA did not include costs associated with upgrading equipment used to control emissions from materials handling affected sources, as these affected sources have been subject to the NSPS for many years

(a longer period than the expected life of these affected sources), and compliance with the NESHAP, which is equivalent to the NSPS for these affected sources would not impose additional costs.

The basis of the control costs for model plants estimated in the docket memoranda and proposal preamble is the Office of Air Quality Planning and Standards Cost Manual. The cost algorithms in the manual were derived from control equipment vendor quotes, standard cost estimating factors, and contractor experience. Installation costs, utilities, maintenance, and other operating costs were estimated and included for impact estimation. The EPA maintains that the costs provided in the proposal preamble are a reasonable basis for projecting the national impacts of the these rules.

VII. Administrative Requirements

A. Docket

A record has been established for this rulemaking under docket number A–92–53. This record includes information considered by the EPA in the development of the promulgated standards. A public version of this record, which does not include any information included as confidential business information, is available for inspection from 8:00 a.m. to 5:30 p.m. Monday-Friday, excluding legal holidays. The public record is located in the Air & Radiation Docket & Information Center, Room M1500, 401 M Street S.W., Washington, D.C. 20460.

Response-to-Comment Document

The response-to-comment document for the promulgated standards contains a summary of all public comments received following proposal of the rule and the EPA's response to these comments. This document is located in the docket (Docket Item No. V–C–1) and is available for downloading from the Technology Transfer Network (TTN). The TTN is one of the EPA's electronic bulletin boards. The TTN provides information from EPA in various areas of air pollution technology or policy. The service is free except for the cost of a phone call. Dial (919) 541–5742 for up to a 14,400 bps modem, or connect through the internet to the following address: ''www.epa.gov/ttn/oarpg''. If more information on the Technology Transfer Network is needed, call the HELP line at (919) 541–5384.

B. Executive Order 12866

Under Executive Order 12866 (58 FR 5173, October 4, 1993), the EPA must determine whether the regulatory action

is ''significant'' and therefore subject to Office of Management and Budget (OMB) review and the requirements of the Executive Order. The Executive Order defines ''significant regulatory action'' as one that is likely to result in standards that may:

(1) Have an annual effect on the economy of $100 million or more or adversely affect, in a material way, the economy, a sector of the economy, productivity, competition, jobs, the environment, public health or safety, or State, local, or tribal governments or communities;

(2) Create a serious inconsistency or otherwise interfere with an action taken or planned by another agency;

(3) Materially alter the budgetary impact of entitlement, grants, user fees, or loan programs or the rights and obligations of recipients thereof; or

(4) Raise novel legal or policy issues arising out of legal mandates, the President's priorities, or the principles set forth in the Executive Order.

Because the projected annual costs (including monitoring) for this NESHAP are $37 million, a regulatory impact analysis has not been prepared. However this action is considered a ''significant regulatory action'' within the meaning of Executive Order 12866 (primarily due to this action's overlap with the Hazardous Waste Combustor MACT standard), and the promulgated regulation presented in this notice was submitted to the OMB for review. Any written comments are included in the docket listed at the beginning of today's notice under **ADDRESSESS.** The docket is available for public inspection at the EPA's Air Docket Section, which is listed in the **ADDRESSES** section of this preamble.

C. Executive Order 12875: Enhancing Intergovernmental Partnerships

Under Executive Order 12875, the EPA may not issue a regulation that is not required by statute and that creates a mandate upon a State, local or tribal government, unless the Federal government provides the funds necessary to pay the direct compliance costs incurred by those governments, or EPA consults with those governments. If EPA complies by consulting, Executive Order 12875 requires EPA to provide to the Office of Management and Budget a description of the extent of EPA's prior consultation with representatives of affected State, local and tribal governments, the nature of their concerns, copies of any written communications from the governments, and a statement supporting the need to issue the regulation. In addition, Executive Order 12875 requires EPA to

develop an effective process permitting elected officials and other representatives of State, local and tribal governments ''to provide meaningful and timely input in the development of regulatory proposals containing significant unfunded mandates.''

Today's rule does not create a mandate on State, local or tribal governments. The rule does not impose any enforceable duties on these entities. Accordingly, the requirements of section 1(a) of Executive Order 12875 do not apply to this rule.

D. Unfunded Mandates Reform Act

Title II of the Unfunded Mandates Reform Act of 1995 (UMRA), Public Law 104–4, establishes requirements for Federal agencies to assess the effects of their regulatory actions on State, local, and tribal governments and the private sector. Under section 202 of the UMRA, the EPA generally must prepare a written statement, including a cost-benefit analysis, for proposed and final rules with ''Federal mandates'' that may result in expenditures to State, local, and tribal governments, in the aggregate, or to the private sector, of $100 million or more in any one year. Before promulgating an EPA rule for which a written statement is needed, section 205 of the UMRA generally requires the EPA to identify and consider a reasonable number of regulatory alternatives and adopt the least costly, most cost-effective or least burdensome alternative that achieves the objectives of the rule. The provisions of section 205 do not apply when they are inconsistent with applicable law. Moreover, section 205 allows the EPA to adopt an alternative other than the least costly, most cost-effective, or least burdensome alternative if the Administrator publishes with the final rule an explanation why that alternative was not adopted. Before the EPA establishes any regulatory requirements that may significantly or uniquely affect small governments, including tribal governments, it must have developed under section 203 of the UMRA a small government agency plan. The plan must provide for notifying potentially affected small governments, enabling officials of affected small governments to have meaningful and timely input in the development of EPA regulatory proposals with significant Federal intergovernmental mandates, and informing, educating, and advising small governments on compliance with the regulatory requirements.

The EPA has determined that this rule does not contain a Federal mandate that may result in expenditures of $100 million or more for State, local, and

tribal governments, in aggregate, or the private sector in any one year, nor does the rule significantly or uniquely impact small governments, because it contains no requirements that apply to such governments or impose obligations upon them. Thus, the requirements of the UMRA do not apply to this rule.

E. Regulatory Flexibility Act

The EPA has determined that it is not necessary to prepare a regulatory flexibility analysis in connection with this final rule. As discussed earlier in the response to comments section of the preamble, the EPA has determined that this rule will not have a significant economic impact on a substantial number of small entities.

Although the rule will not have a significant impact on a substantial number of small entities, the EPA worked with portland cement small entities throughout the rulemaking process. Meetings were held on a regular basis with the Portland Cement Association (PCA) and industry representatives, including both small and large firms, to discuss the development of the rule, exchange information and data, solicit comments on draft rule requirements, and provide a list of the small firms. In addition, some cement industry representatives formed a group called the ''Small Cement Company MACT Coalition'', which designated the PCA as its representative in meetings with the EPA concerning the rulemaking for the portland cement industry.

The promulgated emission standards are representative of the floor level of emision control, which is the minimum level of control allowed under the Act. Further, the costs of required performance testing and monitoring have been minimized by specifying emission limits and monitoring parameters in terms of surrogates for HAP emissions, which are less costly to measure. The Agency has also tried to make the rule ''user friendly,'' with language that is easy to understand by all of the regulated community. EPA is also allowing affected firms up to 3 years from the effective date of the final rule to comply, which could lessen capital availability concerns. An extra year may be granted by the Administrator or delegated regulatory authority if necessary to install controls. Further, EPA has deferred the compliance date for installing PM CEMs pending a future proposed rulemaking.

F. Submission to Congress and the General Accounting Office

The Congressional Review Act, 5 U.S.C. 801 *et seq.*, as added by the Small

Business Regulatory Enforcement Fairness Act of 1996, generally provides that before a rule may take effect, the agency promulgating the rule must submit a rule report, which includes a copy of the rule, to each House of the Congress and to the Comptroller General of the United States. The EPA will submit a report containing this rule and other required information to the U.S. Senate, the U.S. House of Representatives, and the Comptroller General of the United States prior to publication of the rule in the **Federal Register**. This rule is not a ''major rule'' as defined by 5 U.S.C. § 804(2).

G. Paperwork Reduction Act

The information collection requirements in this rule are being submitted for approval to the Office of Management and Budget (OMB) under the *Paperwork Reduction Act,* 44 U.S.C. 3501 *et seq.* An Information Collection Request (ICR) document has been prepared by EPA (ICR No. 1801.02) and a copy may be obtained from Sandy Farmer by mail at OP Regulatory Information Division; U.S. Environmental Protection Agency (2137); 401 M St., S.W.; Washington, DC 20460, by email at farmer.sandy@epamail.epa.gov, or by calling (202) 260–2740. A copy may also be downloaded off the internet at http://www.epa.gov/icr. The information requirements are not effective until OMB approves them.

The EPA is required under section 112 (d) of the Clean Air Act to regulate emissions of HAPs listed in section 112 (b). The requested information is needed as part of the overall compliance and enforcement program. The ICR requires that portland cement manufacturing plants retain records of parameter and emissions monitoring data at facilities for a period of 5 years, which is consistent with the General Provisions to 40 CFR part 63 and the permit requirements under 40 CFR part 70. All sources subject to this rule will be required to obtain operating permits either through the State-approved permitting program or, if one does not exist, in accordance with the provisions of 40 CFR part 71, when promulgated.

The public reporting burden for this collection of information is estimated to average 2148 hours per respondent per year for an estimated 36 respondents. This estimate includes performance tests and reports (with repeat tests where needed); one-time preparation of an operation and maintenance plan with semiannual reports of any event where the procedures in the plan were not followed; semiannual excess emissions reports; notifications; and

recordkeeping. The total annualized capital costs associated with monitoring requirements over the three-year period of the ICR is estimated at $750,000. This estimate includes the capital and startup costs associated with installation of required continuous monitoring equipment for those affected sources subject to the standard. The total operation and maintenance cost is estimated at $682,000 per year. Burden means the total time, effort, or financial resources expended by persons to generate, maintain, retain, or disclose or provide information to or for a Federal agency. This includes the time needed to review instructions; develop, acquire, install, and utilize technology and systems for the purposes of collecting, validating, and verifying information, processing and maintaining information, and disclosing and providing information; adjust the existing ways to comply with any previously applicable instructions and requirements; train personnel to be able to respond to a collection of information; search data sources; complete and review the collection of information; and transmit or otherwise disclose the information.

An Agency may not conduct or sponsor, and a person is not required to respond to a collection of information unless it displays a currently valid OMB control number. The OMB control numbers for EPA's regulations are listed in 40 CFR Part 9 and 48 CFR Chapter 15.

H. Pollution Prevention Act

During the development of this rule, the EPA explored opportunities to eliminate or reduce emissions through the application of new processes or work practices. This NESHAP includes a monitoring requirement for an inspection of the components of the combustion system of each kiln and in-line kiln raw mill to be conducted at least once per year. Such an inspection will promote fuel efficiency and decrease the formation of combustion related pollutants.

I. National Technology Transfer and Advancement Act

Section 12(d) of the National Technology Transfer and Advancement Act (NTTAA) directs all Federal agencies to use voluntary consensus standards in regulatory and procurement activities unless to do so would be inconsistent with applicable law or otherwise impracticable. Voluntary consensus standards are technical standards (e.g., materials specifications, test methods, sampling procedures, and business practices)

developed or adopted by one or more voluntary consensus bodies. The NTTAA requires Federal agencies to provide Congress, through annual reports to OMB, with explanations when an agency does not use available and applicable voluntary consensus standards.

Consistent with the NTTAA, the EPA conducted a search to identify voluntary consensus standards. The search identified 21 voluntary consensus standards that appeared to have possible use in lieu of EPA standard reference methods. However, after reviewing available standards, EPA determined that 14 of the candidate consensus standards identified for measuring emissions of the HAPs or surrogates subject to emission standards in the rule would not be practical due to lack of equivalency, documentation, validation data and other important technical and policy considerations. Six of the remaining candidate consensus standards are new standards under development that EPA plans to follow, review and consider adopting at a later date.

One consensus standard, ASTM D6216–98, appears to be practical for EPA use in lieu of EPA Performance Specification 1 (See 40 CFR Part 60, Appendix B). On September 23, 1998, EPA proposed incorporating by reference ASTM D6216–98 under a separate rulemaking (63 FR 50824) that would allow broader use and application of this consensus standard. EPA plans to complete this action in the near future. For these reasons, EPA defers taking action in this rulemaking that would adopt D6216–98 in lieu of PS–1 requirements as it would be impractical for EPA to act independently from other rulemaking activity already undergoing notice and comment.

Additionally, EPA received comments that ASTM FTIR Standard D6348 should be used in lieu of EPA's proposed Fourier transform infrared spectroscopy (FTIR) emission test methods. EPA has determined for a number of reasons that the ASTM Standard D6348 is one of the 14 standards determined to be impractical to adopt for the purposes of this rulemaking. EPA review comments on ASTM Standard D6348 are included in the docket for this rulemaking and summarized in the response to comments section of this preamble. ASTM has also been advised of the reasons for impracticality and ASTM Subcommittee D22–03 is now undertaking a revision of the ASTM standard. Upon demonstration of technical equivalency with the EPA

FTIR methods, the revised ASTM standard could be incorporated by reference for EPA regulatory applicability at a later date.

This rule requires standard EPA methods known to the industry and States. Approved alternative methods also may be used with prior EPA approval.

J. Executive Order 13045

Executive Order 13045 applies to any rule that EPA determines (1) is "economically significant" as defined under Executive Order 12866, and (2) the environmental health or safety risk addressed by the rule has a disproportionate effect on children. If the regulatory action meets both criteria, the Agency must evaluate the environmental health or safety effects of the planned rule on children and explain why the planned regulation is preferable to other potentially effective and reasonably feasible alternatives considered by the Agency.

This final rule is not subject to E.O. 13045, entitled "Protection of Children from Environmental Health Risks and Safety Risks" (62 FR 19885, April 23, 1997), because it is not an economically significant regulatory action as defined by Executive Order 12866, and it does not address an environmental health or safety risk that would have a disproportionate effect on children.

K. Executive Order 13084: Consultation and Coordination With Indian Tribal Governments

Under Executive Order 13084, EPA may not issue a regulation that is not required by statute, that significantly or uniquely affects the communities of Indian tribal governments, and that imposes substantial direct compliance costs on those communities, unless the Federal government provides the funds necessary to pay the direct compliance costs incurred by the tribal governments, or EPA consults with those governments. If EPA complies by consulting, Executive Order 13084 requires EPA to provide to the Office of Management and Budget, in a separately identified section of the preamble to the rule, a description of the extent of EPA's prior consultation with representatives of affected tribal governments, a summary of the nature of their concerns, and a statement supporting the need to issue the regulation. In addition, Executive Order 13084 requires EPA to develop an effective process permitting elected officials and other representatives of Indian tribal governments "to provide meaningful and timely input in the development of regulatory policies on matters that

significantly or uniquely affect their communities.''

Today's rule does not significantly or uniquely affect the communities of Indian tribal governments. Accordingly, the requirements of section 3(b) of Executive Order 13084 do not apply to this rule.

List of Subjects in 40 CFR Part 63

Environmental protection, Air pollution control, Hazardous substances, Portland cement manufacturing, Reporting and recordkeeping requirements.

Dated: May 14, 1999.

Carol M. Browner,

Administrator.

For the reasons set out in the preamble, part 63 of title 40, chapter 1 of the Code of Federal Regulations is amended as follows:

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

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

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

2. Part 63 is amended by adding a new subpart LLL, consisting of §§ 63.1340 through 63.1359 to read as follows:

Subpart LLL—National Emission Standards for Hazardous Air Pollutants From the Portland Cement Manufacturing Industry

General

Sec.
63.1340   Applicability and designation of affected sources.
63.1341   Definitions.

Emission Standards and Operating Limits

63.1342   Standards: General.
63.1343   Standards for kilns and in-line kiln/raw mills.
63.1344   Operating limits for kilns and in-line kiln/raw mills.
63.1345   Standards for clinker coolers.
63.1346   Standards for new and reconstructed raw material dryers.
63.1347   Standards for raw and finish mills.
63.1348   Standards for affected sources other than kilns; in-line kiln raw mills; clinker coolers; new and reconstructed raw material dryers; and raw and finish mills.

Monitoring and Compliance Provisions

63.1349   Performance testing requirements.
63.1350   Monitoring requirements.
63.1351   Compliance dates.
63.1352   Additional test methods.

Notification, Reporting and Recordkeeping

63.1353   Notification requirements.
63.1354   Reporting requirements.
63.1355   Recordkeeping requirements.

Other

63.1356   Exemption from new source performance standards.
63.1357   Temporary, conditioned exemption from particulate and opacity standards.
63.1358   Delegation of authority.
63.1359   [Reserved]

Table 1 to Subpart LLL of Part 63—Applicability of General Provisions

Subpart LLL—National Emission Standards for Hazardous Air Pollutants From the Portland Cement Manufacturing Industry

General

§ 63.1340   Applicability and designation of affected sources.

(a) Except as specified in paragraphs (b) and (c) of this section, the provisions of this subpart apply to each new and existing portland cement plant which is a major source or an area source as defined in § 63.2.

(b) The affected sources subject to this subpart are:

(1) Each kiln and each in-line kiln/raw mill at any major or area source, including alkali bypasses, except for kilns and in-line kiln/raw mills that burn hazardous waste and are subject to and regulated under subpart EEE of this part;

(2) Each clinker cooler at any portland cement plant which is a major source;

(3) Each raw mill at any portland cement plant which is a major source;

(4) Each finish mill at any portland cement plant which is a major source;

(5) Each raw material dryer at any portland cement plant which is a major source and each greenfield raw material dryer at any portland cement plant which is a major or area source;

(6) Each raw material, clinker, or finished product storage bin at any portland cement plant which is a major source;

(7) Each conveying system transfer point at any portland cement plant which is a major source;

(8) Each bagging system at any portland cement plant which is a major source; and

(9) Each bulk loading or unloading system at any portland cement plant which is a major source.

(c) For portland cement plants with on-site nonmetallic mineral processing facilities, the first affected source in the sequence of materials handling operations subject to this subpart is the raw material storage, which is just prior to the raw mill. The primary and secondary crushers and any other equipment of the on-site nonmetallic mineral processing plant which precedes the raw material storage are not subject to this subpart. Furthermore,

the first conveyor transfer point subject to this subpart is the transfer point associated with the conveyor transferring material from the raw material storage to the raw mill.

(d) The owner or operator of any affected source subject to the provisions of this subpart is subject to title V permitting requirements.

§ 63.1341   Definitions.

All terms used in this subpart that are not defined in this section have the meaning given to them in the CAA and in subpart A of this part.

Alkali bypass means a duct between the feed end of the kiln and the preheater tower through which a portion of the kiln exit gas stream is withdrawn and quickly cooled by air or water to avoid excessive buildup of alkali, chloride and/or sulfur on the raw feed. This may also be referred to as the ''kiln exhaust gas bypass''.

Bagging system means the equipment which fills bags with portland cement.

Clinker cooler means equipment into which clinker product leaving the kiln is placed to be cooled by air supplied by a forced draft or natural draft supply system.

Continuous monitor means a device which continuously samples the regulated parameter specified in § 63.1350 of this subpart without interruption, evaluates the detector response at least once every 15 seconds, and computes and records the average value at least every 60 seconds, except during allowable periods of calibration and except as defined otherwise by the continuous emission monitoring system performance specifications in appendix B to part 60 of this chapter.

Conveying system means a device for transporting materials from one piece of equipment or location to another location within a facility. Conveying systems include but are not limited to the following: feeders, belt conveyors, bucket elevators and pneumatic systems.

Conveying system transfer point means a point where any material including but not limited to feed material, fuel, clinker or product, is transferred to or from a conveying system, or between separate parts of a conveying system.

Dioxins and furans (D/F) means tetra-, penta-, hexa-, hepta-, and octa-chlorinated dibenzo dioxins and furans.

Facility means all contiguous or adjoining property that is under common ownership or control, including properties that are separated only by a road or other public right-of-way.

Feed means the prepared and mixed materials, which include but are not limited to materials such as limestone, clay, shale, sand, iron ore, mill scale, cement kiln dust and flyash, that are fed to the kiln. Feed does not include the fuels used in the kiln to produce heat to form the clinker product.

Finish mill means a roll crusher, ball and tube mill or other size reduction equipment used to grind clinker to a fine powder. Gypsum and other materials may be added to and blended with clinker in a finish mill. The finish mill also includes the air separator associated with the finish mill.

Greenfield kiln, in-line kiln/raw mill, or raw material dryer means a kiln, in-line kiln/raw mill, or raw material dryer for which construction is commenced at a plant site (where no kilns and no in-line kiln/raw mills were in operation at any time prior to March 24, 1998) after March 24, 1998.

Hazardous waste is defined in § 261.3 of this chapter.

In-line kiln/raw mill means a system in a portland cement production process where a dry kiln system is integrated with the raw mill so that all or a portion of the kiln exhaust gases are used to perform the drying operation of the raw mill, with no auxiliary heat source used. In this system the kiln is capable of operating without the raw mill operating, but the raw mill cannot operate without the kiln gases, and consequently, the raw mill does not generate a separate exhaust gas stream.

Kiln means a device, including any associated preheater or precalciner devices, that produces clinker by heating limestone and other materials for subsequent production of portland cement.

Kiln exhaust gas bypass means alkali bypass.

Monovent means an exhaust configuration of a building or emission control device (e. g. positive pressure fabric filter) that extends the length of the structure and has a width very small in relation to its length (i. e., length to width ratio is typically greater than 5:1). The exhaust may be an open vent with or without a roof, louvered vents, or a combination of such features.

New brownfield kiln, in-line kiln raw mill, or raw material dryer means a kiln, in-line kiln/raw mill or raw material dryer for which construction is commenced at a plant site (where kilns and/or in-line kiln/raw mills were in operation prior to March 24, 1998) after March 24, 1998.

One-minute average means the average of thermocouple or other sensor responses calculated at least every 60 seconds from responses obtained at least once during each consecutive 15 second period.

Portland cement plant means any facility manufacturing portland cement.

Raw material dryer means an impact dryer, drum dryer, paddle-equipped rapid dryer, air separator, or other equipment used to reduce the moisture content of feed materials.

Raw mill means a ball and tube mill, vertical roller mill or other size reduction equipment, that is not part of an in-line kiln/raw mill, used to grind feed to the appropriate size. Moisture may be added or removed from the feed during the grinding operation. If the raw mill is used to remove moisture from feed materials, it is also, by definition, a raw material dryer. The raw mill also includes the air separator associated with the raw mill.

Rolling average means the average of all one-minute averages over the averaging period.

Run average means the average of the one-minute parameter values for a run.

TEQ means the international method of expressing toxicity equivalents for dioxins and furans as defined in U.S. EPA, Interim Procedures for Estimating Risks Associated with Exposures to Mixtures of Chlorinated Dibenzo-p-dioxins and -dibenzofurans (CDDs and CDFs) and 1989 Update, March 1989.

Emission Standards and Operating Limits

§ 63.1342 Standards: General.

(a) Table 1 to this subpart provides cross references to the 40 CFR part 63, subpart A, general provisions, indicating the applicability of the general provisions requirements to subpart LLL.

(b) Table 1 of this section provides a summary of emission limits and operating limits of this subpart.

TABLE 1 TO § 63.1342.—EMISSION LIMITS AND OPERATING LIMITS

Affected source	Pollutant or opacity	Emission and operating limit
All kilns and in-line kiln/raw mills at major sources (including alkali bypass).	PM	0.15 kg/Mg of feed (dry basis).
	Opacity	20 percent.
All kilns and in-line kiln/raw mills at major and area sources (including alkali bypass).	D/F	0.20 ng TEQ/dscm
		or
		0.40 ng TEQ/dscm when the average of the performance test run average particulate matter control device (PMCD) inlet temperatures is 204° C or less. [Corrected to 7 percent oxygen]
		Operate such that the three-hour rolling average PMCD inlet temperature is no greater than the temperature established at performance test.
		If activated carbon injection is used: Operate such that the three-hour rolling average activated carbon injection rate is no less than rate established at performance test. Operate such that either the carrier gas flow rate or carrier gas pressure drop exceeds the value established at performance test. Inject carbon of equivalent specifications to that used at performance test.
New greenfield kilns and in-line kiln/raw mills at major and area sources.	THC	50 ppmvd, as propane, corrected to 7 percent oxygen.
All clinker coolers at major sources	PM	0.050 kg/Mg of feed (dry basis)
	Opacity	10 percent.
All raw mills and finish mills at major sources	Opacity	10 percent.
New greenfield raw material dryers at major and area sources.	THC	50 ppmvd, as propane, corrected to 7 percent oxygen.
All raw material dryers and material handling points at major sources.	Opacity	10 percent.

§ 63.1343   **Standards for kilns and in-line kiln/raw mills.**

(a) *General.* The provisions in this section apply to each kiln, each in-line kiln/raw mill, and any alkali bypass associated with that kiln or in-line kiln/raw mill.

(b) *Existing, reconstructed, or new brownfield/major sources.* No owner or operator of an existing, reconstructed or new brownfield kiln or an existing, reconstructed or new brownfield in-line kiln/raw mill at a facility that is a major source subject to the provisions of this subpart shall cause to be discharged into the atmosphere from these affected sources, any gases which:

(1) Contain particulate matter (PM) in excess of 0.15 kg per Mg (0.30 lb per ton) of feed (dry basis) to the kiln. When there is an alkali bypass associated with a kiln or in-line kiln/raw mill, the combined particulate matter emissions from the kiln or in-line kiln/raw mill and the alkali bypass are subject to this emission limit.

(2) Exhibit opacity greater than 20 percent.

(3) Contain D/F in excess of:

(i) 0.20 ng per dscm (8.7×10^{-11} gr per dscf) (TEQ) corrected to seven percent oxygen; or

(ii) 0.40 ng per dscm (1.7×10^{-10} gr per dscf) (TEQ) corrected to seven percent oxygen, when the average of the performance test run average temperatures at the inlet to the particulate matter control device is 204 °C (400 °F) or less.

(c) *Greenfield/major sources.* No owner or operator that commences construction of a greenfield kiln or greenfield inline kiln/raw mill at a facility which is a major source subject to the provisions of this subpart shall cause to be discharged into the atmosphere from these affected sources any gases which:

(1) Contain particulate matter in excess of 0.15 kg per Mg (0.30 lb per ton) of feed (dry basis) to the kiln. When there is an alkali bypass associated with a kiln or in-line kiln/raw mill, the combined particulate matter emissions from the kiln or in-line kiln/raw mill and the bypass stack are subject to this emission limit.

(2) Exhibit opacity greater than 20 percent.

(3) Contain D/F in excess of:

(i) 0.20 ng per dscm (8.7×10^{-11} gr per dscf) (TEQ) corrected to seven percent oxygen; or

(ii) 0.40 ng per dscm (1.7×10^{-10} gr per dscf) (TEQ) corrected to seven percent oxygen, when the average of the performance test run average temperatures at the inlet to the

particulate matter control device is 204 °C (400 °F) or less.

(4) Contain total hydrocarbon (THC), from the main exhaust of the kiln or in-line kiln/raw mill, in excess of 50 ppmvd as propane, corrected to seven percent oxygen.

(d) *Existing, reconstructed, or new brownfield/area sources.* No owner or operator of an existing, reconstructed, or new brownfield kiln or an existing, reconstructed or new brownfield in-line kiln/raw mill at a facility that is an area source subject to the provisions of this subpart shall cause to be discharged into the atmosphere from these affected sources any gases which contain D/F in excess of:

(1) 0.20 ng per dscm (8.7×10^{-11} gr per dscf) (TEQ) corrected to seven percent oxygen; or

(2) 0.40 ng per dscm (1.7×10^{-10} gr per dscf) (TEQ) corrected to seven percent oxygen, when the average of the performance test run average temperatures at the inlet to the particulate matter control device is 204 °C (400 °F) or less.

(e) *Greenfield/area sources.* No owner or operator of a greenfield kiln or a greenfield in-line kiln/raw mill at a facility that is an area source subject to the provisions of this subpart shall cause to be discharged into the atmosphere from these affected sources any gases which:

(1) Contain D/F in excess of:

(i) 0.20 ng per dscm (8.7×10^{-11} gr per dscf) (TEQ) corrected to seven percent oxygen; or

(ii) 0.40 ng per dscm (1.7×10^{-11} gr per dscf) (TEQ) corrected to seven percent oxygen, when the average of the performance test run average temperatures at the inlet to the particulate matter control device is 204 °C (400 °F) or less.

(2) Contain THC, from the main exhaust of the kiln or in-line kiln/raw mill, in excess of 50 ppmvd as propane, corrected to seven percent oxygen.

§ 63.1344   **Operating limits for kilns and in-line kiln/raw mills.**

(a) The owner or operator of a kiln subject to a D/F emission limitation under § 63.1343 must operate the kiln such that the temperature of the gas at the inlet to the kiln particulate matter control device (PMCD) and alkali bypass PMCD, if applicable, does not exceed the applicable temperature limit specified in paragraph (b) of this section. The owner or operator of an in-line kiln/raw mill subject to a D/F emission limitation under § 63.1343 must operate the in-line kiln/raw mill, such that:

(1) When the raw mill of the in-line kiln/raw mill is operating, the applicable temperature limit for the main in-line kiln/raw mill exhaust, specified in paragraph (b) of this section and established during the performance test when the raw mill was operating is not exceeded.

(2) When the raw mill of the in-line kiln/raw mill is not operating, the applicable temperature limit for the main in-line kiln/raw mill exhaust, specified in paragraph (b) of this section and established during the performance test when the raw mill was not operating, is not exceeded.

(3) If the in-line kiln/raw mill is equipped with an alkali bypass, the applicable temperature limit for the alkali bypass, specified in paragraph (b) of this section and established during the performance test when the raw mill was operating, is not exceeded.

(b) The temperature limit for affected sources meeting the limits of paragraph (a) of this section or paragraphs (a)(1) through (a)(3) of this section is determined in accordance with § 63.1349(b)(3)(iv).

(c) The owner or operator of an affected source subject to a D/F emission limitation under § 63.1343 that employs carbon injection as an emission control technique must operate the carbon injection system in accordance with paragraphs (c)(1) and (c)(2) of this section.

(1) The three-hour rolling average activated carbon injection rate shall be equal to or greater than the activated carbon injection rate determined in accordance with § 63.1349(b)(3)(vi).

(2) The owner or operator shall either:

(i) Maintain the minimum activated carbon injection carrier gas flow rate, as a three-hour rolling average, based on the manufacturer's specifications. These specifications must be documented in the test plan developed in accordance with § 63.7(c), or

(ii) Maintain the minimum activated carbon injection carrier gas pressure drop, as a three-hour rolling average, based on the manufacturer's specifications. These specifications must be documented in the test plan developed in accordance with § 63.7(c).

(d) Except as provided in paragraph (e) of this section, the owner or operator of an affected source subject to a D/F emission limitation under § 63.1343 that employs carbon injection as an emission control technique must specify and use the brand and type of activated carbon used during the performance test until a subsequent performance test is conducted, unless the site-specific performance test plan contains documentation of key parameters that

affect adsorption and the owner or operator establishes limits based on those parameters, and the limits on these parameters are maintained.

(e) The owner or operator of an affected source subject to a D/F emission limitation under § 63.1343 that employs carbon injection as an emission control technique may substitute, at any time, a different brand or type of activated carbon provided that the replacement has equivalent or improved properties compared to the activated carbon specified in the site-specific performance test plan and used in the performance test. The owner or operator must maintain documentation that the substitute activated carbon will provide the same or better level of control as the original activated carbon.

§ 63.1345 Standards for clinker coolers.

(a) No owner or operator of a new or existing clinker cooler at a facility which is a major source subject to the provisions of this subpart shall cause to be discharged into the atmosphere from the clinker cooler any gases which:

(1) Contain particulate matter in excess of 0.050 kg per Mg (0.10 lb per ton) of feed (dry basis) to the kiln.

(2) Exhibit opacity greater than ten percent.

(b) [Reserved].

§ 63.1346 Standards for new and reconstructed raw material dryers.

(a) *Brownfield/major sources.* No owner or operator of a new or reconstructed brownfield raw material dryer at a facility which is a major source subject to this subpart shall cause to be discharged into the atmosphere from the new or reconstructed raw material dryer any gases which exhibit opacity greater than ten percent.

(b) *Greenfield/area sources.* No owner or operator of a greenfield raw material dryer at a facility which is an area source subject to this subpart shall cause to be discharged into the atmosphere from the greenfield raw material dryer any gases which contain THC in excess of 50 ppmvd, reported as propane, corrected to seven percent oxygen.

(c) *Greenfield/major sources.* No owner or operator of a greenfield raw material dryer at a facility which is a major source subject to this subpart shall cause to be discharged into the atmosphere from the greenfield raw material dryer any gases which:

(1) Contain THC in excess of 50 ppmvd, reported as propane, corrected to seven percent oxygen.

(2) Exhibit opacity greater than ten percent.

§ 63.1347 Standards for raw and finish mills.

The owner or operator of each new or existing raw mill or finish mill at a facility which is a major source subject to the provisions of this subpart shall not cause to be discharged from the mill sweep or air separator air pollution control devices of these affected sources any gases which exhibit opacity in excess of ten percent.

§ 63.1348 Standards for affected sources other than kilns; in-line kiln/raw mills; clinker coolers; new and reconstructed raw material dryers; and raw and finish mills.

The owner or operator of each new or existing raw material, clinker, or finished product storage bin; conveying system transfer point; bagging system; and bulk loading or unloading system; and each existing raw material dryer, at a facility which is a major source subject to the provisions of this subpart shall not cause to be discharged any gases from these affected sources which exhibit opacity in excess of ten percent.

Monitoring and Compliance Provisions

§ 63.1349 Performance testing requirements.

(a) The owner or operator of an affected source subject to this subpart shall demonstrate initial compliance with the emission limits of § 63.1343 and §§ 63.1345 through 63.1348 using the test methods and procedures in paragraph (b) of this section and § 63.7. Performance test results shall be documented in complete test reports that contain the information required by paragraphs (a)(1) through (a)(10) of this section, as well as all other relevant information. The plan to be followed during testing shall be made available to the Administrator prior to testing, if requested.

(1) A brief description of the process and the air pollution control system;

(2) Sampling location description(s);

(3) A description of sampling and analytical procedures and any modifications to standard procedures;

(4) Test results;

(5) Quality assurance procedures and results;

(6) Records of operating conditions during the test, preparation of standards, and calibration procedures;

(7) Raw data sheets for field sampling and field and laboratory analyses;

(8) Documentation of calculations;

(9) All data recorded and used to establish parameters for compliance monitoring; and

(10) Any other information required by the test method.

(b) Performance tests to demonstrate initial compliance with this subpart

shall be conducted as specified in paragraphs (b)(1) through (b)(4) of this section.

(1) The owner or operator of a kiln subject to limitations on particulate matter emissions shall demonstrate initial compliance by conducting a performance test as specified in paragraphs (b)(1)(i) through (b)(1)(iv) of this section. The owner or operator of an in-line kiln/raw mill subject to limitations on particulate matter emissions shall demonstrate initial compliance by conducting separate performance tests as specified in paragraphs (b)(1)(i) through (b)(1)(iv) of this section while the raw mill of the in-line kiln/raw mill is under normal operating conditions and while the raw mill of the in-line kiln/raw mill is not operating. The owner or operator of a clinker cooler subject to limitations on particulate matter emissions shall demonstrate initial compliance by conducting a performance test as specified in paragraphs (b)(1)(i) through (b)(1)(iii) of this section. The opacity exhibited during the period of the Method 5 of Appendix A to part 60 of this chapter performance tests required by paragraph (b)(1)(i) of this section shall be determined as required in paragraphs (b)(1)(v) through (vi) of this section.

(i) EPA Method 5 of appendix A to part 60 of this chapter shall be used to determine PM emissions. Each performance test shall consist of three separate runs under the conditions that exist when the affected source is operating at the highest load or capacity level reasonably expected to occur. Each run shall be conducted for at least one hour, and the minimum sample volume shall be 0.85 dscm (30 dscf). The average of the three runs shall be used to determine compliance. A determination of the particulate matter collected in the impingers ("back half") of the Method 5 particulate sampling train is not required to demonstrate initial compliance with the PM standards of this subpart. However this shall not preclude the permitting authority from requiring a determination of the "back half" for other purposes.

(ii) Suitable methods shall be used to determine the kiln or inline kiln/raw mill feed rate, except for fuels, for each run.

(iii) The emission rate, E, of PM shall be computed for each run using equation 1:

$$E = (C_s Q_{sd})/P \qquad \text{(Eq. 1)}$$

Where:

E = emission rate of particulate matter, kg/Mg of kiln feed.

c_s = concentration of PM, kg/dscm.

Q_{sd} = volumetric flow rate of effluent gas, dscm/hr.

P = total kiln feed (dry basis), Mg/hr.

(iv) When there is an alkali bypass associated with a kiln or in-line kiln/raw mill, the main exhaust and alkali bypass of the kiln or in-line kiln/raw mill shall be tested simultaneously and the combined emission rate of particulate matter from the kiln or in-line kiln/raw mill and alkali bypass shall be computed for each run using equation 2,

$$E_c = (C_{sk}Q_{sdk} + C_{sb}Q_{sdb})/P \qquad (Eq. 2)$$

Where:

E_c = the combined emission rate of particulate matter from the kiln or in-line kiln/raw mill and bypass stack, kg/Mg of kiln feed.

c_{sk} = concentration of particulate matter in the kiln or in-line kiln/raw mill effluent, kg/dscm.

Q_{sdk} = volumetric flow rate of kiln or in-line kiln/raw mill effluent, dscm/hr.

c_{sb} = concentration of particulate matter in the alkali bypass gas, kg/dscm.

Q_{sdb} = volumetric flow rate of alkali bypass gas, dscm/hr.

P=total kiln feed (dry basis), Mg/hr.

(v) Except as provided in paragraph (b)(1)(vi) of this section the opacity exhibited during the period of the Method 5 performance tests required by paragraph (b)(1)(i) of this section shall be determined through the use of a continuous opacity monitor (COM). The maximum six-minute average opacity during the three Method 5 test runs shall be determined during each Method 5 test run, and used to demonstrate initial compliance with the applicable opacity limits of § 63.1343(b)(2), § 63.1343(c)(2), or § 63.1345(a)(2).

(vi) Each owner or operator of a kiln, in-line kiln/raw mill, or clinker cooler subject to the provisions of this subpart using a fabric filter with multiple stacks or an electrostatic precipitator with multiple stacks may, in lieu of installing the continuous opacity monitoring system required by paragraph (b)(1)(v) of this section, conduct an opacity test in accordance with Method 9 of appendix A to part 60 of this chapter during each Method 5 performance test required by paragraph (b)(1)(i) of this section. If the control device exhausts through a monovent, or if the use of a COM in accordance with the installation specifications of Performance Specification 1 (PS–1) of appendix B to part 60 of this chapter is not feasible, a test shall be conducted in accordance with Method 9 of appendix A to part 60

of this chapter during each Method 5 performance test required by paragraph (b)(1)(i) of this section. The maximum six-minute average opacity shall be determined during the three Method 5 test runs, and used to demonstrate initial compliance with the applicable opacity limits of § 63.1343(b)(2), § 63.1343(c)(2), or § 63.1345(a)(2).

(2) The owner or operator of any affected source subject to limitations on opacity under this subpart that is not subject to paragraph (b)(1) of this section shall demonstrate initial compliance with the affected source opacity limit by conducting a test in accordance with Method 9 of appendix A to part 60 of this chapter. The performance test shall be conducted under the conditions that exist when the affected source is operating at the highest load or capacity level reasonably expected to occur. The maximum six-minute average opacity exhibited during the test period shall be used to determine whether the affected source is in initial compliance with the standard. The duration of the Method 9 performance test shall be 3-hours (30 6-minute averages), except that the duration of the Method 9 performance test may be reduced to 1-hour if the conditions of paragraphs (b)(2)(i) through (ii) of the section apply:

(i) There are no individual readings greater than 10 percent opacity;

(ii) There are no more than three readings of 10 percent for the first 1-hour period.

(3) The owner or operator of an affected source subject to limitations on D/F emissions shall demonstrate initial compliance with the D/F emission limit by conducting a performance test using Method 23 of appendix A to part 60 of this chapter. The owner or operator of an in-line kiln/raw mill shall demonstrate initial compliance by conducting separate performance tests while the raw mill of the in-line kiln/raw mill is under normal operating conditions and while the raw mill of the in-line kiln/raw mill is not operating. The owner or operator of a kiln or in-line kiln/raw mill equipped with an alkali bypass shall conduct simultaneous performance tests of the kiln or in-line kiln/raw mill exhaust and the alkali bypass, however the owner or operator of an in-line kiln/raw mill is not required to conduct a performance test of the alkali bypass exhaust when the raw mill of the in-line kiln/raw mill is not operating.

(i) Each performance test shall consist of three separate runs; each run shall be conducted under the conditions that exist when the affected source is operating at the highest load or capacity

level reasonably expected to occur. The duration of each run shall be at least three hours and the sample volume for each run shall be at least 2.5 dscm (90 dscf). The concentration shall be determined for each run and the arithmetic average of the concentrations measured for the three runs shall be calculated and used to determine compliance.

(ii) The temperature at the inlet to the kiln or in-line kiln/raw mill PMCD, and where applicable, the temperature at the inlet to the alkali bypass PMCD, must be continuously recorded during the period of the Method 23 test, and the continuous temperature record(s) must be included in the performance test report.

(iii) One-minute average temperatures must be calculated for each minute of each run of the test.

(iv) The run average temperature must be calculated for each run, and the average of the run average temperatures must be determined and included in the performance test report and will determine the applicable temperature limit in accordance with § 63.1344(b).

(v) If activated carbon injection is used for D/F control, the rate of activated carbon injection to the kiln or in-line kiln/raw mill exhaust, and where applicable, the rate of activated carbon injection to the alkali bypass exhaust, must be continuously recorded during the period of the Method 23 test, and the continuous injection rate record(s) must be included in the performance test report. In addition, the performance test report must include the brand and type of activated carbon used during the performance test and a continuous record of either the carrier gas flow rate or the carrier gas pressure drop for the duration of the test. Activated carbon injection rate parameters must be determined in accordance with paragraphs (b)(3)(vi) of this section.

(vi) The run average injection rate must be calculated for each run, and the average of the run average injection rates must be determined and included in the performance test report and will determine the applicable injection rate limit in accordance with § 63.1344(c)(1).

(4) The owner or operator of an affected source subject to limitations on emissions of THC shall demonstrate initial compliance with the THC limit by operating a continuous emission monitor in accordance with Performance Specification 8A of appendix B to part 60 of this chapter. The duration of the performance test shall be three hours, and the average THC concentration (as calculated from the one-minute averages) during the three hour performance test shall be

calculated. The owner or operator of an in-line kiln/raw mill shall demonstrate initial compliance by conducting separate performance tests while the raw mill of the in-line kiln/raw mill is under normal operating conditions and while the raw mill of the in-line kiln/raw mill is not operating.

(c) Except as provided in paragraph (e) of this section, performance tests required under paragraphs (b)(1) and (b)(2) of this section shall be repeated every five years, except that the owner or operator of a kiln, in-line kiln/raw mill or clinker cooler is not required to repeat the initial performance test of opacity for the kiln, in-line kiln/raw mill or clinker cooler.

(d) Performance tests required under paragraph (b)(3) of this section shall be repeated every 30 months.

(e) The owner or operator is required to repeat the performance tests for kilns or in-line kiln/raw mills as specified in paragraphs (b)(1) and (b)(3) of this section within 90 days of initiating any significant change in the feed or fuel from that used in the previous performance test.

(f) Table 1 of this section provides a summary of the performance test requirements of this subpart.

TABLE 1 TO § 63.1349.—SUMMARY OF PERFORMANCE TEST REQUIREMENTS

Affected source and pollutant	Performance test
New and existing kiln and in-line kiln/raw mill [b c] PM	EPA Method 5.[a]
New and existing kiln and in-line kiln/raw mill [b c] Opacity	COM if feasible [d e] or EPA Method 9 visual opacity readings.
New and existing kiln and in-line kiln/raw mill [b c f g] D/F	EPA Method 23[h].
New greenfield kiln and in-line kiln/raw mill [c] THC	THC CEM (EPA PS–8A) [i].
New and existing clinker cooler PM	EPA Method 5 [a].
New and existing clinker cooler opacity	COM [d,j] or EPA Method 9 visual opacity readings.
New and existing raw and finish mill opacity	EPA Method 9.[a j]
New and existing raw material dryer and materials handling processes (raw material storage, clinker storage, finished product storage, conveyor transfer points, bagging, and bulk loading and unloading systems) opacity.	EPA Method 9.[a j]
New greenfield raw material dryer THC	THC CEM (EPA PS–8A).[i]

[a] Required initially and every 5 years thereafter.
[b] Includes main exhaust and alkali bypass.
[c] In-line kiln/raw mill to be tested with and without raw mill in operation.
[d] Must meet COM performance specification criteria. If the fabric filter or electrostatic precipitator has multiple stacks, daily EPA Method 9 visual opacity readings may be taken instead of using a COM.
[e] Opacity limit is 20 percent.
[f] Alkali bypass is tested with the raw mill on.
[g] Temperature and (if applicable) activated carbon injection parameters determined separately with and without the raw mill operating.
[h] Required initially and every 30 months thereafter.
[i] EPA Performance Specification (PS)–8A of appendix B to 40 CFR part 60.
[j] Opacity limit is 10 percent.

§ 63.1350 Monitoring requirements.

(a) The owner or operator of each portland cement plant shall prepare for each affected source subject to the provisions of this subpart, a written operations and maintenance plan. The plan shall be submitted to the Administrator for review and approval as part of the application for a part 70 permit and shall include the following information:

(1) Procedures for proper operation and maintenance of the affected source and air pollution control devices in order to meet the emission limits and operating limits of §§ 63.1343 through 63.1348;

(2) Corrective actions to be taken when required by paragraph (e) of this section;

(3) Procedures to be used during an inspection of the components of the combustion system of each kiln and each in-line kiln raw mill located at the facility at least once per year; and

(4) Procedures to be used to periodically monitor affected sources subject to opacity standards under §§ 63.1346 and 63.1348. Such procedures must include the provisions of paragraphs (a)(4)(i) through (a)(4)(iv) of this section.

(i) The owner or operator must conduct a monthly 1-minute visible emissions test of each affected source in accordance with Method 22 of Appendix A to part 60 of this chapter. The test must be conducted while the affected source is in operation.

(ii) If no visible emissions are observed in six consecutive monthly tests for any affected source, the owner or operator may decrease the frequency of testing from monthly to semi-annually for that affected source. If visible emissions are observed during any semi-annual test, the owner or operator must resume testing of that affected source on a monthly basis and maintain that schedule until no visible emissions are observed in six consecutive monthly tests.

(iii) If no visible emissions are observed during the semi-annual test for any affected source, the owner or operator may decrease the frequency of testing from semi-annually to annually for that affected source. If visible emissions are observed during any annual test, the owner or operator must resume testing of that affected source on a monthly basis and maintain that schedule until no visible emissions are observed in six consecutive monthly tests.

(iv) If visible emissions are observed during any Method 22 test, the owner or operator must conduct a 6-minute test of opacity in accordance with Method 9 of appendix A to part 60 of this chapter. The Method 9 test must begin within one hour of any observation of visible emissions.

(b) Failure to comply with any provision of the operations and maintenance plan developed in accordance with paragraph (a) of this section shall be a violation of the standard.

(c) The owner or operator of a kiln or in-line kiln/raw mill shall monitor opacity at each point where emissions are vented from these affected sources including alkali bypasses in accordance with paragraphs (c)(1) through (c)(3) of this section.

(1) Except as provided in paragraph (c)(2) of this section, the owner or operator shall install, calibrate, maintain, and continuously operate a

continuous opacity monitor (COM) located at the outlet of the PM control device to continuously monitor the opacity. The COM shall be installed, maintained, calibrated, and operated as required by subpart A, general provisions of this part, and according to PS–1 of appendix B to part 60 of this chapter.

(2) The owner or operator of a kiln or in-line kiln/raw mill subject to the provisions of this subpart using a fabric filter with multiple stacks or an electrostatic precipitator with multiple stacks may, in lieu of installing the continuous opacity monitoring system required by paragraph (c)(1) of this section, monitor opacity in accordance with paragraphs (c)(2)(i) through (ii) of this section. If the control device exhausts through a monovent, or if the use of a COM in accordance with the installation specifications of PS–1 of appendix B to part 60 of this chapter is not feasible, the owner or operator must monitor opacity in accordance with paragraphs (c)(2)(i) through (ii) of this section.

(i) Perform daily visual opacity observations of each stack in accordance with the procedures of Method 9 of appendix A of part 60 of this chapter. The Method 9 test shall be conducted while the affected source is operating at the highest load or capacity level reasonably expected to occur within the day. The duration of the Method 9 test shall be at least 30 minutes each day.

(ii) Use the Method 9 procedures to monitor and record the average opacity for each six-minute period during the test.

(3) To remain in compliance, the opacity must be maintained such that the 6-minute average opacity for any 6-minute block period does not exceed 20 percent. If the average opacity for any 6-minute block period exceeds 20 percent, this shall constitute a violation of the standard.

(d) The owner or operator of a clinker cooler shall monitor opacity at each point where emissions are vented from the clinker cooler in accordance with paragraphs (d)(1) through (d)(3) of this section.

(1) Except as provided in paragraph (d)(2) of this section, the owner or operator shall install, calibrate, maintain, and continuously operate a COM located at the outlet of the clinker cooler PM control device to continuously monitor the opacity. The COM shall be installed, maintained, calibrated, and operated as required by subpart A, general provisions of this part, and according to PS–1 of appendix B to part 60 of this chapter.

(2) The owner or operator of a clinker cooler subject to the provisions of this subpart using a fabric filter with multiple stacks or an electrostatic precipitator with multiple stacks may, in lieu of installing the continuous opacity monitoring system required by paragraph (d)(1) of this section, monitor opacity in accordance with paragraphs (d)(2)(i) through (ii) of this section. If the control device exhausts through a monovent, or if the use of a COM in accordance with the installation specifications of PS–1 of appendix B to part 60 of this chapter is not feasible, the owner or operator must monitor opacity in accordance with paragraphs (d)(2)(i) through (ii) of this section.

(i) Perform daily visual opacity observations of each stack in accordance with the procedures of Method 9 of appendix A of part 60 of this chapter. The Method 9 test shall be conducted while the affected source is operating at the highest load or capacity level reasonably expected to occur within the day. The duration of the Method 9 test shall be at least 30 minutes each day.

(ii) Use the Method 9 procedures to monitor and record the average opacity for each six-minute period during the test.

(3) To remain in compliance, the opacity must be maintained such that the 6-minute average opacity for any 6-minute block period does not exceed 10 percent. If the average opacity for any 6-minute block period exceeds 10 percent, this shall constitute a violation of the standard.

(e) The owner or operator of a raw mill or finish mill shall monitor opacity by conducting daily visual emissions observations of the mill sweep and air separator PMCDs of these affected sources, in accordance with the procedures of Method 22 of appendix A of part 60 of this chapter. The Method 22 test shall be conducted while the affected source is operating at the highest load or capacity level reasonably expected to occur within the day. The duration of the Method 22 test shall be six minutes. If visible emissions are observed during any Method 22 visible emissions test, the owner or operator must:

(1) Initiate, within one-hour, the corrective actions specified in the site specific operating and maintenance plan developed in accordance with paragraphs (a)(1) and (a)(2) of this section; and

(2) Within 24 hours of the end of the Method 22 test in which visible emissions were observed, conduct a visual opacity test of each stack from which visible emissions were observed in accordance with Method 9 of

appendix A of part 60 of this chapter. The duration of the Method 9 test shall be thirty minutes.

(f) The owner or operator of an affected source subject to a limitation on D/F emissions shall monitor D/F emissions in accordance with paragraphs (f)(1) through (f)(6) of this section.

(1) The owner or operator shall install, calibrate, maintain, and continuously operate a continuous monitor to record the temperature of the exhaust gases from the kiln, in-line kiln/raw mill and alkali bypass, if applicable, at the inlet to, or upstream of, the kiln, in-line kiln/raw mill and/or alkali bypass PM control devices.

(i) The recorder response range must include zero and 1.5 times either of the average temperatures established according to the requirements in § 63.1349(b)(3)(iv).

(ii) The reference method must be a National Institute of Standards and Technology calibrated reference thermocouple-potentiometer system or alternate reference, subject to approval by the Administrator.

(2) The owner or operator shall monitor and continuously record the temperature of the exhaust gases from the kiln, in-line kiln/raw mill and alkali bypass, if applicable, at the inlet to the kiln, in-line kiln/raw mill and/or alkali bypass PMCD.

(3) The three-hour rolling average temperature shall be calculated as the average of 180 successive one-minute average temperatures.

(4) Periods of time when one-minute averages are not available shall be ignored when calculating three-hour rolling averages. When one-minute averages become available, the first one-minute average is added to the previous 179 values to calculate the three-hour rolling average.

(5) When the operating status of the raw mill of the in-line kiln/raw mill is changed from off to on, or from on to off the calculation of the three-hour rolling average temperature must begin anew, without considering previous recordings.

(6) The calibration of all thermocouples and other temperature sensors shall be verified at least once every three months.

(g) The owner or operator of an affected source subject to a limitation on D/F emissions that employs carbon injection as an emission control technique shall comply with the monitoring requirements of paragraphs (f)(1) through (f)(6) and (g)(1) through (g)(6) of this section to demonstrate continuous compliance with the D/F emission standard.

(1) Install, operate, calibrate and maintain a continuous monitor to record the rate of activated carbon injection. The accuracy of the rate measurement device must be ±1 percent of the rate being measured.

(2) Verify the calibration of the device at least once every three months.

(3) The three-hour rolling average activated carbon injection rate shall be calculated as the average of 180 successive one-minute average activated carbon injection rates.

(4) Periods of time when one-minute averages are not available shall be ignored when calculating three-hour rolling averages. When one-minute averages become available, the first one-minute average is added to the previous 179 values to calculate the three-hour rolling average.

(5) When the operating status of the raw mill of the in-line kiln/raw mill is changed from off to on, or from on to off the calculation of the three-hour rolling average activated carbon injection rate must begin anew, without considering previous recordings.

(6) The owner or operator must install, operate, calibrate and maintain a continuous monitor to record the activated carbon injection system carrier gas parameter (either the carrier gas flow rate or the carrier gas pressure drop) established during the D/F performance test in accordance with paragraphs (g)(6)(i) through (g)(6)(iii) of this section.

(i) The owner or operator shall install, calibrate, operate and maintain a device to continuously monitor and record the parameter value.

(ii) The owner or operator must calculate and record three-hour rolling averages of the parameter value.

(iii) Periods of time when one-minute averages are not available shall be ignored when calculating three-hour rolling averages. When one-minute averages become available, the first one-minute average shall be added to the previous 179 values to calculate the three-hour rolling average.

(h) The owner or operator of an affected source subject to a limitation on THC emissions under this subpart shall comply with the monitoring requirements of paragraphs (h)(1) through (h)(3) of this section to demonstrate continuous compliance with the THC emission standard:

(1) The owner or operator shall install, operate and maintain a THC continuous emission monitoring system in accordance with Performance Specification 8A, of appendix B to part 60 of this chapter and comply with all of the requirements for continuous monitoring systems found in the general provisions, subpart A of this part.

(2) The owner or operator is not required to calculate hourly rolling averages in accordance with section 4.9 of Performance Specification 8A.

(3) Any thirty-day block average THC concentration in any gas discharged from a greenfield raw material dryer, the main exhaust of a greenfield kiln, or the main exhaust of a greenfield in-line kiln/raw mill, exceeding 50 ppmvd, reported as propane, corrected to seven percent oxygen, is a violation of the standard.

(i) The owner or operator of any kiln or in-line kiln/raw mill subject to a D/F emission limit under this subpart shall conduct an inspection of the components of the combustion system of each kiln or in-line kiln raw mill at least once per year.

(j) The owner or operator of an affected source subject to a limitation on opacity under § 63.1346 or § 63.1348 shall monitor opacity in accordance with the operation and maintenance plan developed in accordance with paragraph (a) of this section.

(k) The owner or operator of an affected source subject to a particulate matter standard under § 63.1343 shall install, calibrate, maintain and operate a particulate matter continuous emission monitoring system (PM CEMS) to measure the particulate matter discharged to the atmosphere. The compliance deadline for installing the PM CEMS and all requirements relating to performance of the PM CEMS and implementation of the PM CEMS requirement is deferred pending further rulemaking.

(l) An owner or operator may submit an application to the Administrator for approval of alternate monitoring requirements to demonstrate compliance with the emission standards of this subpart, except for emission standards for THC, subject to the provisions of paragraphs (l)(1) through (l)(6) of this section.

(1) The Administrator will not approve averaging periods other than those specified in this section, unless the owner or operator documents, using data or information, that the longer averaging period will ensure that emissions do not exceed levels achieved during the performance test over any increment of time equivalent to the time required to conduct three runs of the performance test.

(2) If the application to use an alternate monitoring requirement is approved, the owner or operator must continue to use the original monitoring requirement until approval is received to use another monitoring requirement.

(3) The owner or operator shall submit the application for approval of alternate monitoring requirements no later than the notification of performance test. The application must contain the information specified in paragraphs (l)(3)(i) through (l)(3)(iii) of this section:

(i) Data or information justifying the request, such as the technical or economic infeasibility, or the impracticality of using the required approach;

(ii) A description of the proposed alternative monitoring requirement, including the operating parameter to be monitored, the monitoring approach and technique, the averaging period for the limit, and how the limit is to be calculated; and

(iii) Data or information documenting that the alternative monitoring requirement would provide equivalent or better assurance of compliance with the relevant emission standard.

(4) The Administrator will notify the owner or operator of the approval or denial of the application within 90 calendar days after receipt of the original request, or within 60 calendar days of the receipt of any supplementary information, whichever is later. The Administrator will not approve an alternate monitoring application unless it would provide equivalent or better assurance of compliance with the relevant emission standard. Before disapproving any alternate monitoring application, the Administrator will provide:

(i) Notice of the information and findings upon which the intended disapproval is based; and

(ii) Notice of opportunity for the owner or operator to present additional supporting information before final action is taken on the application. This notice will specify how much additional time is allowed for the owner or operator to provide additional supporting information.

(5) The owner or operator is responsible for submitting any supporting information in a timely manner to enable the Administrator to consider the application prior to the performance test. Neither submittal of an application, nor the Administrator's failure to approve or disapprove the application relieves the owner or operator of the responsibility to comply with any provision of this subpart.

(6) The Administrator may decide at any time, on a case-by-case basis that additional or alternative operating limits, or alternative approaches to establishing operating limits, are necessary to demonstrate compliance with the emission standards of this subpart.

(m) A summary of the monitoring requirements of this subpart is given in Table 1 to this section.

TABLE 1 TO § 63.1350.—MONITORING REQUIREMENTS

Affected source/pollutant or opacity	Monitor type/operation/process	Monitoring requirements
All affected sources	Operations and maintenance plan	Prepare written plan for all affected sources and control devices.
All kilns and in-line kiln raw mills at major sources (including alkali bypass)/opacity.	Continuous opacity monitor, if applicable	Install, calibrate, maintain and operate in accordance with general provisions and with PS–1.
	Method 9 opacity test, if applicable	Daily test of at least 30-minutes, while kiln is at highest load or capacity level.
Kilns and in-line kiln raw mills at major sources (including alkali bypass)/particulate matter.	Particulate matter continuous emission monitoring system.	Deferred.
Kilns and in-line kiln raw mills at major and area sources (including alkali bypass)/ D/F.	Combustion system inspection	Conduct annual inspection of components of combustion system.
	Continuous temperature monitoring at PMCD inlet.	Install, operate, calibrate and maintain continuous temperature monitoring and recording system; calculate three-hour rolling averages; verify temperature sensor calibration at least quarterly.
Kilns and in-line kiln raw mills at major and area sources (including alkali bypass)/ D/F (continued).	Activated carbon injection rate monitor, if applicable.	Install, operate, calibrate and maintain continuous activated carbon injection rate monitor; calculate three-hour rolling averages; verify calibration at least quarterly; install, operate, calibrate and maintain carrier gas flow rate monitor or carrier gas pressure drop monitor; calculate three-hour rolling averages; document carbon specifications.
New greenfield kilns and in-line kiln raw mills at major and area sources/THC.	Total hydrocarbon continuous emission monitor.	Install, operate, and maintain THC CEM in accordance with PS–8A; calculate 30-day block average THC concentration.
Clinker coolers at major sources/opacity	Continuous opacity monitor, if applicable	Install, calibrate, maintain and operate in accordance with general provisions and with PS–1.
	Method 9 opacity test, if applicable	Daily test of at least 30-minutes, while kiln is at highest load or capacity level.
Raw mills and finish mills at major sources/ opacity.	Method 22 visible emissions test	Conduct daily 6-minute Method 22 visible emissions test while mill is operating at highest load or capacity level; if visible emissions are observed, initiate corrective action within one hour and conduct 30-minute Method 9 test within 24 hours.
New greenfield raw material dryers at major and area sources/THC.	Total hydrocarbon continuous emission monitor.	Install, operate, and maintain THC CEM in accordance with PS–8A; calculate 30-day block average THC concentration.
Raw material dryers; raw material, clinker, finished product storage bins; conveying system transfer points; bagging systems; and bulk loading and unloading systems at major sources/opacity.	Method 22 visible emissions test	As specified in operation and maintenance plan.

§ 63.1351   Compliance dates.

(a) The compliance date for an owner or operator of an existing affected source subject to the provisions of this subpart is June 10, 2002.

(b) The compliance date for an owner or operator of an affected source subject to the provisions of this subpart that commences new construction or reconstruction after March 24, 1998 is June 9, 1999 or immediately upon startup of operations, whichever is later.

6§ 3.1352   Additional test methods.

(a) Owners or operators conducting tests to determine the rates of emission of hydrogen chloride (HCl) from kilns,

in-line kiln/raw mills and associated bypass stacks at portland cement manufacturing facilities, for use in applicability determinations under § 63.1340 are permitted to use Method 320 or Method 321 of appendix A of this part.

(b) Owners or operators conducting tests to determine the rates of emission of hydrogen chloride (HCl) from kilns, in-line kiln/raw mills and associated bypass stacks at portland cement manufacturing facilities, for use in applicability determinations under § 63.1340 are permitted to use Methods 26 or 26A of appendix A to part 60 of this chapter, except that the results of

these tests shall not be used to establish status as an area source.

(c) Owners or operators conducting tests to determine the rates of emission of specific organic HAP from raw material dryers, kilns and in-line kiln/raw mills at portland cement manufacturing facilities, for use in applicability determinations under § 63.1340 of this subpart are permitted to use Method 320 of appendix A to this part, or Method 18 of appendix A to part 60 of this chapter.

Notification, Reporting and Recordkeeping

§ 63.1353 Notification requirements.

(a) The notification provisions of 40 CFR part 63, subpart A that apply and those that do not apply to owners and operators of affected sources subject to this subpart are listed in Table 1 of this subpart. If any State requires a notice that contains all of the information required in a notification listed in this section, the owner or operator may send the Administrator a copy of the notice sent to the State to satisfy the requirements of this section for that notification.

(b) Each owner or operator subject to the requirements of this subpart shall comply with the notification requirements in § 63.9 as follows:

(1) Initial notifications as required by § 63.9(b) through (d). For the purposes of this subpart, a Title V or 40 CFR part 70 permit application may be used in lieu of the initial notification required under § 63.9(b), provided the same information is contained in the permit application as required by § 63.9(b), and the State to which the permit application has been submitted has an approved operating permit program under part 70 of this chapter and has received delegation of authority from the EPA. Permit applications shall be submitted by the same due dates as those specified for the initial notification.

(2) Notification of performance tests, as required by §§ 63.7 and 63.9(e).

(3) Notification of opacity and visible emission observations required by § 63.1349 in accordance with §§ 63.6(h)(5) and 63.9(f).

(4) Notification, as required by § 63.9(g), of the date that the continuous emission monitor performance evaluation required by § 63.8(e) is scheduled to begin.

(5) Notification of compliance status, as required by § 63.9(h).

§ 63.1354 Reporting requirements.

(a) The reporting provisions of subpart A of this part that apply and those that do not apply to owners or operators of affected sources subject to this subpart are listed in Table 1 of this subpart. If any State requires a report that contains all of the information required in a report listed in this section, the owner or operator may send the Administrator a copy of the report sent to the State to satisfy the requirements of this section for that report.

(b) The owner or operator of an affected source shall comply with the reporting requirements specified in § 63.10 of the general provisions of this part 63, subpart A as follows:

(1) As required by § 63.10(d)(2), the owner or operator shall report the results of performance tests as part of the notification of compliance status.

(2) As required by § 63.10(d)(3), the owner or operator of an affected source shall report the opacity results from tests required by § 63.1349.

(3) As required by § 63.10(d)(4), the owner or operator of an affected source who is required to submit progress reports as a condition of receiving an extension of compliance under § 63.6(i) shall submit such reports by the dates specified in the written extension of compliance.

(4) As required by § 63.10(d)(5), if actions taken by an owner or operator during a startup, shutdown, or malfunction of an affected source (including actions taken to correct a malfunction) are consistent with the procedures specified in the source's startup, shutdown, and malfunction plan specified in § 63.6(e)(3), the owner or operator shall state such information in a semiannual report. Reports shall only be required if a startup, shutdown, or malfunction occurred during the reporting period. The startup, shutdown, and malfunction report may be submitted simultaneously with the excess emissions and continuous monitoring system performance reports; and

(5) Any time an action taken by an owner or operator during a startup, shutdown, or malfunction (including actions taken to correct a malfunction) is not consistent with the procedures in the startup, shutdown, and malfunction plan, the owner or operator shall make an immediate report of the actions taken for that event within 2 working days, by telephone call or facsimile (FAX) transmission. The immediate report shall be followed by a letter, certified by the owner or operator or other responsible official, explaining the circumstances of the event, the reasons for not following the startup, shutdown, and malfunction plan, and whether any excess emissions and/or parameter monitoring exceedances are believed to have occurred.

(6) As required by § 63.10(e)(2), the owner or operator shall submit a written report of the results of the performance evaluation for the continuous monitoring system required by § 63.8(e). The owner or operator shall submit the report simultaneously with the results of the performance test.

(7) As required by § 63.10(e)(2), the owner or operator of an affected source using a continuous opacity monitoring system to determine opacity compliance during any performance test required under § 63.7 and described in § 63.6(d)(6) shall report the results of the continuous opacity monitoring system performance evaluation conducted under § 63.8(e).

(8) As required by § 63.10(e)(3), the owner or operator of an affected source equipped with a continuous emission monitor shall submit an excess emissions and continuous monitoring system performance report for any event when the continuous monitoring system data indicate the source is not in compliance with the applicable emission limitation or operating parameter limit.

(9) The owner or operator shall submit a summary report semiannually which contains the information specified in § 63.10(e)(3)(vi). In addition, the summary report shall include:

(i) All exceedences of maximum control device inlet gas temperature limits specified in § 63.1344(a) and (b);

(ii) All failures to calibrate thermocouples and other temperature sensors as required under § 63.1350(f)(7) of this subpart; and

(iii) All failures to maintain the activated carbon injection rate, and the activated carbon injection carrier gas flow rate or pressure drop, as applicable, as required under § 63.1344(c).

(iv) The results of any combustion system component inspections conducted within the reporting period as required under § 63.1350(i).

(v) All failures to comply with any provision of the operation and maintenance plan developed in accordance with § 63.1350(a).

(10) If the total continuous monitoring system downtime for any CEM or any continuous monitoring system (CMS) for the reporting period is ten percent or greater of the total operating time for the reporting period, the owner or operator shall submit an excess emissions and continuous monitoring system performance report along with the summary report.

§ 63.1355 Recordkeeping requirements.

(a) The owner or operator shall maintain files of all information (including all reports and notifications) required by this section recorded in a form suitable and readily available for inspection and review as required by § 63.10(b)(1). The files shall be retained for at least five years following the date of each occurrence, measurement, maintenance, corrective action, report, or record. At a minimum, the most recent two years of data shall be retained on site. The remaining three

years of data may be retained off site. The files may be maintained on microfilm, on a computer, on floppy disks, on magnetic tape, or on microfiche.

(b) The owner or operator shall maintain records for each affected source as required by § 63.10(b)(2) and (b)(3) of this part; and

(1) All documentation supporting initial notifications and notifications of compliance status under § 63.9;

(2) All records of applicability determination, including supporting analyses; and

(3) If the owner or operator has been granted a waiver under § 63.8(f)(6), any information demonstrating whether a source is meeting the requirements for a waiver of recordkeeping or reporting requirements.

(c) In addition to the recordkeeping requirements in paragraph (b) of this section, the owner or operator of an affected source equipped with a continuous monitoring system shall maintain all records required by § 63.10(c).

Other

§ 63.1356 **Exemption from new source performance standards.**

(a) Except as provided in paragraphs (a)(1) and (a)(2) of this section, any affected source subject to the provisions of this subpart is exempted from any otherwise applicable new source performance standard contained in 40 CFR part 60, subpart F.

(1) Kilns and in-line kiln/raw mills, as applicable under 40 CFR 60.60(b), located at area sources are subject to PM and opacity limits and associated reporting and recordkeeping, under 40 CFR part 60, subpart F.

(2) Greenfield raw material dryers, as applicable under 40 CFR 60.60(b), located at area sources are subject to opacity limits and associated reporting and recordkeeping under 40 CFR part 60, subpart F.

§ 63.1357 **Temporary, conditioned exemption from particulate matter and opacity standards.**

(a) Subject to the limitations of paragraphs (b) through (f) of this section, an owner or operator conducting PM CEMS correlation tests (that is, correlation with manual stack methods) is exempt from:

(1) Any particulate matter and opacity standards of part 60 or part 63 of this chapter that are applicable to cement kilns and in-line kiln/raw mills.

(2) Any permit or other emissions or operating parameter or other limitation on workplace practices that are applicable to cement kilns and in-line kiln raw mills to ensure compliance with any particulate matter and opacity standards of this part or part 60 of this chapter.

(b) The owner or operator must develop a PM CEMS correlation test plan. The plan must be submitted to the Administrator for approval at least 90 days before the correlation test is scheduled to be conducted. The plan must include:

(1) The number of test conditions and the number of runs for each test condition;

(2) The target particulate matter emission level for each test condition;

(3) How the operation of the affected source will be modified to attain the desired particulate matter emission rate; and

(4) The anticipated normal particulate matter emission level.

(c) The Administrator will review and approve or disapprove the correlation test plan in accordance with § 63.7(c)(3)(i) and (iii). If the Administrator fails to approve or disapprove the correlation test plan within the time period specified in § 63.7(c)(3)(iii), the plan shall be considered approved, unless the Administrator has requested additional information.

(d) The stack sampling team must be on-site and prepared to perform correlation testing no later than 24 hours after operations are modified to attain the desired particulate matter

emissions concentrations, unless the correlation test plan documents that a longer period is appropriate.

(e) The particulate matter and opacity standards and associated operating limits and conditions will not be waived for more than 96 hours, in the aggregate, for a correlation test, including all runs and conditions.

(f) The owner or operator must return the affected source to operating conditions indicative of compliance with the applicable particulate matter and opacity standards as soon as possible after correlation testing is completed.

§ 63.1358 **Delegation of authority.**

(a) In delegating implementation and enforcement authority to a State under subpart E of this part, the authorities contained in paragraph (b) of this section shall be retained by the Administrator and not transferred to a State.

(b) Authority which will not be delegated to States:

(1) Approval of alternative non-opacity emission standards under § 63.6(g).

(2) Approval of alternative opacity standards under § 63.6(h)(9).

(3) Approval of major changes to test methods under §§ 63.7(e)(2)(ii) and 63.7(f). A major change to a test method is a modification to a federally enforceable test method that uses unproven technology or procedures or is an entirely new method (sometimes necessary when the required test method is unsuitable).

(4) Approval of major changes to monitoring under § 63.8(f). A major change to monitoring is a modification to federally enforceable monitoring that uses unproven technology or procedures, is an entirely new method (sometimes necessary when the required monitoring is unsuitable), or is a change in the averaging period.

(5) Waiver of recordkeeping under § 63.10(f).

§ 63.1359 **[Reserved]**

TABLE 1 TO SUBPART LLL.—APPLICABILITY OF GENERAL PROVISIONS

General Provisions 40 CFR Citation	Requirement	Applies to Subpart LLL	Comment
63.1(a)(1) through (4)	Applicability	Yes.	
63.1(a)(5)		No	[Reserved].
63.1(a)(6) through (a)(8)	Applicability	Yes.	
63.1(a)(9)		No	[Reserved].
63.1(a)(10) through (14)	Applicability	Yes.	
63.1(b)(1)	Initial Applicability Determination	No	§ 63.1340 specifies applicability.
63.1(b)(2) and (3)	Initial Applicability Determination	Yes.	
63.1(c)(1)	Applicability After Standard Established	Yes.	
63.1(c)(2)	Permit Requirements	Yes	Area sources must obtain Title V permits.

TABLE 1 TO SUBPART LLL.—APPLICABILITY OF GENERAL PROVISIONS—Continued

General Provisions 40 CFR Citation	Requirement	Applies to Subpart LLL	Comment
63.1(c)(3)		No	[Reserved].
63.1(c)(4) and (5)	Extensions, Notifications	Yes.	
63.1(d)		No	[Reserved].
63.1(e)	Applicability of Permit Program	Yes.	
63.2	Definitions	Yes.	Additional definitions in § 63.1341.
63.3(a) through (c)	Units and Abbreviations	Yes.	
63.4(a)(1) through (a)(3)	Prohibited Activities	Yes.	
63.4(a)(4)		No	[Reserved].
63.4(a)(5)	Compliance date	Yes.	
63.4(b) and (c)	Circumvention, Severability	Yes.	
63.5(a)(1) and (2)	Construction/Reconstruction	Yes.	
63.5(b)(1)	Compliance Dates	Yes.	
63.5(b)(2)		No	[Reserved].
63.5(b)(3) through (6)	Construction Approval, Applicability	Yes.	
63.5(c)		No	[Reserved].
63.5(d)(1) through (4)	Approval of Construction/Reconstruction	Yes.	
63.5(e)	Approval of Construction/Reconstruction	Yes.	
63.5(f)(1) and (2)	Approval of Construction/Reconstruction	Yes.	
63.6(a)	Compliance for Standards and Maintenance.	Yes.	
63.6(b)(1) through (5)	Compliance Dates	Yes.	
63.6(b)(6)		No	[Reserved].
63.6(b)(7)	Compliance Dates	Yes.	
63.6(c)(1) and (2)	Compliance Dates	Yes.	
63.6(c)(3) and (c)(4)	....	No	[Reserved].
63.6(c)(5)	Compliance Dates	Yes.	
63.6(d)		No	[Reserved].
63.6(e)(1) and (e)(2)	Operation & Maintenance	Yes.	
63.6(e)(3)	Startup, Shutdown Malfunction Plan	Yes.	
63.6(f)(1) through (3)	Compliance with Emission Standards	Yes.	
63.6(g)(1) through (g)(3)	Alternative Standard	Yes.	
63.6(h)(1) and (2)	Opacity/VE Standards	Yes.	
63.6(h)(3)		No	Reserved
63.6(h)(4) and (h)(5)(i)	Opacity/VE Standards	Yes.	
63.6(h)(5)(ii) through (iv)	Opacity/VE Standards	No	Test duration specified in Subpart LLL.
63.6(h)(6)	Opacity/VE Standards	Yes.	
63.6(i)(1) through (i)(14)	Extension of Compliance	Yes.	
63.6(i)(15)		No	[Reserved].
63.6(i)(16)	Extension of Compliance	Yes.	
63.6(j)	Exemption from Compliance	Yes.	
63.7(a)(1) through (a)(3)	Performance Testing Requirements	Yes	§ 63.1349 has specific requirements.
63.7(b)	Notification	Yes.	
63.7(c)	Quality Assurance/Test Plan	Yes.	
63.7(d)	Testing Facilities	Yes.	
63.7(e)(1) through (4)	Conduct of Tests	Yes.	
63.7(f)	Alternative Test Method	Yes.	
63.7(g)	Data Analysis	Yes.	
63.7(h)	Waiver of Tests	Yes.	
63.8(a)(1)	Monitoring Requirements	Yes.	
63.8(a)(2)	Monitoring	No	§ 63.1350 includes CEM requirements.
63.8(a)(3)		No	[Reserved].
63.8(a)(4)	Monitoring	No	Flares not applicable.
63.8(b)(1) through (3)	Conduct of Monitoring	Yes.	
63.8(c)(1) through (8)	CMS Operation/ Maintenance	Yes.	Performance specification supersedes requirements for THC CEM. Temperature and activated carbon injection monitoring data reduction requirements given in subpart LLL.
63.8(d)	Quality Control	Yes.	
63.8(e)	Performance Evaluation for CMS	Yes	Performance specification supersedes requirements for THC CEM.
63.8(f)(1) through (f)(5)	Alternative Monitoring Method	Yes	Additional requirements in § 1350(l).
63.8(f)(6)	Alternative to RATA Test	Yes.	
63.8(g)	Data Reduction	Yes.	
63.9(a)	Notification Requirements	Yes.	
63.9(b)(1) through (5)	Initial Notifications	Yes.	
63.9(c)	Request for Compliance Extension	Yes.	
63.9(d)	New Source Notification for Special Compliance Requirements.	Yes.	
63.9(e)	Notification of Performance Test	Yes.	
63.9(f)	Notification of VE/Opacity Test	Yes	Notification not required for VE/ opacity test under § 63.1350(e) and (j).

TABLE 1 TO SUBPART LLL.—APPLICABILITY OF GENERAL PROVISIONS—Continued

General Provisions 40 CFR Citation	Requirement	Applies to Subpart LLL	Comment
63.9(g)	Additional CMS Notifications	Yes.	
63.9(h)(1) through (h)(3)	Notification of Compliance Status	Yes.	
63.9(h)(4)		No	[Reserved].
63.9(h)(5) and (h)(6)	Notification of Compliance Status	Yes.	
63.9(i)	Adjustment of Deadlines	Yes.	
63.9(j)	Change in Previous Information	Yes.	
63.10(a)	Recordkeeping/Reporting	Yes	Yes.
63.10(b)	General Requirements	Yes.	
63.10(c)(1)	Additional CMS Recordkeeping	Yes	PS–8A applies.
63.10(c)(2) through (c)(4)		No	Reserved]
63.10(c)(5) through (c)(8)	Additional CMS Recordkeeping	Yes	PS–8A applies instead of requirements for THC CEM.
63.10(c)(9)		No	[Reserved]
63.10(c)(10) through (15)	Additional CMS Recordkeeping	Yes	PS–8A applies instead of requirements for THC CEM.
63.10(d)(1)	General Reporting Requirements	Yes.	
63.10(d)(2)	Performance Test Results	Yes.	
63.10(d)(3)	Opacity or VE Observations	Yes.	
63.10(d)(4)	Progress Reports	Yes.	
63.10(d)(5)	Startup, Shutdown, Malfunction Reports	Yes.	
63.10(e)(1) and (e)(2)	Additional CMS Reports	Yes.	
63.10(e)(3)	Excess Emissions and CMS Performance Reports.	Yes	Exceedences are defined in subpart LLL.
63.10(f)	Waiver for Recordkeeping/ Reporting	Yes.	
63.11(a) and (b)	Control Device Requirements	No	Flares not applicable.
63.12(a)–(c	)State Authority and Delegations	Yes.	
63.13(a)–(c)	State/Regional Addresses	Yes.	
63.14(a) and (b)	Incorporation by Reference	Yes.	
63.15(a) and (b)	Availability of Information	Yes.	

3. Appendix A of part 63 is amended by adding, in numerical order, Methods 320 and 321 to read as follows:

Appendix A to Part 63—Test Methods

* * * * *

Test Method 320—Measurement of Vapor Phase Organic and Inorganic Emissions by Extractive Fourier Transform Infrared (FTIR) Spectroscopy

1.0 *Introduction.*

Persons unfamiliar with basic elements of FTIR spectroscopy should not attempt to use this method. This method describes sampling and analytical procedures for extractive emission measurements using Fourier transform infrared (FTIR) spectroscopy. Detailed analytical procedures for interpreting infrared spectra are described in the ''Protocol for the Use of Extractive Fourier Transform Infrared (FTIR) Spectrometry in Analyses of Gaseous Emissions from Stationary Sources,'' hereafter referred to as the ''Protocol.'' Definitions not given in this method are given in appendix A of the Protocol. References to specific sections in the Protocol are made throughout this Method. For additional information refer to references 1 and 2, and other EPA reports, which describe the use of FTIR spectrometry in specific field measurement applications and validation tests. The sampling procedure described here is extractive. Flue gas is extracted through a heated gas transport and handling system. For some sources, sample conditioning systems may be applicable. Some examples are given in this method.

Note: sample conditioning systems may be used providing the method validation requirements in Sections 9.2 and 13.0 of this method are met.

1.1 *Scope and Applicability.*

1.1.1 *Analytes.* Analytes include hazardous air pollutants (HAPs) for which EPA reference spectra have been developed. Other compounds can also be measured with this method if reference spectra are prepared according to section 4.6 of the protocol.

1.1.2 *Applicability.* This method applies to the analysis of vapor phase organic or inorganic compounds which absorb energy in the mid-infrared spectral region, about 400 to 4000 cm^{-1} (25 to 2.5 μm). This method is used to determine compound-specific concentrations in a multi-component vapor phase sample, which is contained in a closed-path gas cell. Spectra of samples are collected using double beam infrared absorption spectroscopy. A computer program is used to analyze spectra and report compound concentrations.

1.2 *Method Range and Sensitivity.* Analytical range and sensitivity depend on the frequency-dependent analyte absorptivity, instrument configuration, data collection parameters, and gas stream composition. Instrument factors include: (a) spectral resolution, (b) interferometer signal averaging time, (c) detector sensitivity and response, and (d) absorption path length.

1.2.1 For any optical configuration the analytical range is between the absorbance values of about .01 (infrared transmittance relative to the background = 0.98) and 1.0

(T = 0.1). (For absorbance > 1.0 the relation between absorbance and concentration may not be linear.)

1.2.2 The concentrations associated with this absorbance range depend primarily on the cell path length and the sample temperature. An analyte absorbance greater than 1.0, can be lowered by decreasing the optical path length. Analyte absorbance increases with a longer path length. Analyte detection also depends on the presence of other species exhibiting absorbance in the same analytical region. Additionally, the estimated lower absorbance (A) limit (A = 0.01) depends on the root mean square deviation (RMSD) noise in the analytical region.

1.2.3 The concentration range of this method is determined by the choice of optical configuration.

1.2.3.1 The absorbance for a given concentration can be decreased by decreasing the path length or by diluting the sample. There is no practical upper limit to the measurement range.

1.2.3.2 The analyte absorbance for a given concentration may be increased by increasing the cell path length or (to some extent) using a higher resolution. Both modifications also cause a corresponding increased absorbance for all compounds in the sample, and a decrease in the signal throughput. For this reason the practical lower detection range (quantitation limit) usually depends on sample characteristics such as moisture content of the gas, the presence of other interferants, and losses in the sampling system.

1.3 *Sensitivity.* The limit of sensitivity for an optical configuration and integration time is determined using appendix D of the Protocol: Minimum Analyte Uncertainty, (MAU). The MAU depends on the RMSD noise in an analytical region, and on the absorptivity of the analyte in the same region.

1.4 *Data Quality.* Data quality shall be determined by executing Protocol pre-test procedures in appendices B to H of the protocol and post-test procedures in appendices I and J of the protocol.

1.4.1 Measurement objectives shall be established by the choice of detection limit (DL$_i$) and analytical uncertainty (AU$_i$) for each analyte.

1.4.2 An instrumental configuration shall be selected. An estimate of gas composition shall be made based on previous test data, data from a similar source or information gathered in a pre-test site survey. Spectral interferants shall be identified using the selected DL$_i$ and AU$_i$ and band areas from reference spectra and interferant spectra. The baseline noise of the system shall be measured in each analytical region to determine the MAU of the instrument configuration for each analyte and interferant (MIU$_i$).

1.4.3 Data quality for the application shall be determined, in part, by measuring the RMS (root mean square) noise level in each analytical spectral region (appendix C of the Protocol). The RMS noise is defined as the RMSD of the absorbance values in an analytical region from the mean absorbance value in the region.

1.4.4 The MAU is the minimum analyte concentration for which the AU$_i$ can be maintained; if the measured analyte concentration is less than MAU$_i$, then data quality are unacceptable.

2.0 *Summary of Method*

2.1 *Principle.* References 4 through 7 provide background material on infrared spectroscopy and quantitative analysis. A summary is given in this section.

2.1.1 Infrared absorption spectroscopy is performed by directing an infrared beam through a sample to a detector. The frequency-dependent infrared absorbance of the sample is measured by comparing this detector signal (single beam spectrum) to a signal obtained without a sample in the beam path (background).

2.1.2 Most molecules absorb infrared radiation and the absorbance occurs in a characteristic and reproducible pattern. The infrared spectrum measures fundamental molecular properties and a compound can be identified from its infrared spectrum alone.

2.1.3 Within constraints, there is a linear relationship between infrared absorption and compound concentration. If this frequency dependent relationship (absorptivity) is known (measured), it can be used to determine compound concentration in a sample mixture.

2.1.4 Absorptivity is measured by preparing, in the laboratory, standard samples of compounds at known concentrations and measuring the FTIR ''reference spectra'' of these standard samples. These ''reference spectra'' are then used in sample analysis: (1) Compounds are detected by matching sample absorbance

bands with bands in reference spectra, and (2) concentrations are measured by comparing sample band intensities with reference band intensities.

2.1.5 This method is self-validating provided that the results meet the performance requirement of the QA spike in sections 8.6.2 and 9.0 of this method, and results from a previous method validation study support the use of this method in the application.

2.2 *Sampling and Analysis.* In extractive sampling a probe assembly and pump are used to extract gas from the exhaust of the affected source and transport the sample to the FTIR gas cell. Typically, the sampling apparatus is similar to that used for single-component continuous emission monitor (CEM) measurements.

2.2.1 The digitized infrared spectrum of the sample in the FTIR gas cell is measured and stored on a computer. Absorbance band intensities in the spectrum are related to sample concentrations by what is commonly referred to as Beer's Law.

$$A_i = a_i b c_i \qquad (1)$$

Where:

A_i = absorbance at a given frequency of the ith sample component.

a_i = absorption coefficient (absorptivity) of the ith sample component.

b = path length of the cell.

c_i = concentration of the ith sample component.

2.2.2 Analyte spiking is used for quality assurance (QA). In this procedure (section 8.6.2 of this method) an analyte is spiked into the gas stream at the back end of the sample probe. Analyte concentrations in the spiked samples are compared to analyte concentrations in unspiked samples. Since the concentration of the spike is known, this procedure can be used to determine if the sampling system is removing the spiked analyte(s) from the sample stream.

2.3 *Reference Spectra Availability.* Reference spectra of over 100 HAPs are available in the EPA FTIR spectral library on the EMTIC (Emission Measurement Technical Information Center) computer bulletin board service and at internet address http://info.arnold.af.mil/epa/welcome.htm. Reference spectra for HAPs, or other analytes, may also be prepared according to section 4.6 of the Protocol.

2.4 *Operator Requirements.* The FTIR analyst shall be trained in setting up the instrumentation, verifying the instrument is functioning properly, and performing routine maintenance. The analyst must evaluate the initial sample spectra to determine if the sample matrix is consistent with pre-test assumptions and if the instrument configuration is suitable. The analyst must be able to modify the instrument configuration, if necessary.

2.4.1 The spectral analysis shall be supervised by someone familiar with EPA FTIR Protocol procedures.

2.4.2 A technician trained in instrumental test methods is qualified to install and operate the sampling system. This includes installing the probe and heated line assembly, operating the analyte spike system,

and performing moisture and flow measurements.

3.0 *Definitions*

See appendix A of the Protocol for definitions relating to infrared spectroscopy. Additional definitions are given in sections 3.1 through 3.29.

3.1 *Analyte.* A compound that this method is used to measure. The term ''target analyte'' is also used. This method is multi-component and a number of analytes can be targeted for a test.

3.2 *Reference Spectrum.* Infrared spectrum of an analyte prepared under controlled, documented, and reproducible laboratory conditions according to procedures in section 4.6 of the Protocol. A library of reference spectra is used to measure analytes in gas samples.

3.3 *Standard Spectrum.* A spectrum that has been prepared from a reference spectrum through a (documented) mathematical operation. A common example is de-resolving of reference spectra to lower-resolution standard spectra (Protocol, appendix K to the addendum of this method). Standard spectra, prepared by approved, and documented, procedures can be used as reference spectra for analysis.

3.4 *Concentration.* In this method concentration is expressed as a molar concentration, in ppm-meters, or in (ppm-meters)/K, where K is the absolute temperature (Kelvin). The latter units allow the direct comparison of concentrations from systems using different optical configurations or sampling temperatures.

3.5 *Interferant.* A compound in the sample matrix whose infrared spectrum overlaps with part of an analyte spectrum. The most accurate analyte measurements are achieved when reference spectra of interferants are used in the quantitative analysis with the analyte reference spectra. The presence of an interferant can increase the analytical uncertainty in the measured analyte concentration.

3.6 *Gas Cell.* A gas containment cell that can be evacuated. It is equipped with the optical components to pass the infrared beam through the sample to the detector. Important cell features include: path length (or range if variable), temperature range, materials of construction, and total gas volume.

3.7 *Sampling System.* Equipment used to extract the sample from the test location and transport the sample gas to the FTIR analyzer. This includes sample conditioning systems.

3.8 *Sample Analysis.* The process of interpreting the infrared spectra to obtain sample analyte concentrations. This process is usually automated using a software routine employing a classical least squares (cls), partial least squares (pls), or K- or P-matrix method.

3.9 *One hundred percent line.* A double beam transmittance spectrum obtained by combining two background single beam spectra. Ideally, this line is equal to 100 percent transmittance (or zero absorbance) at every frequency in the spectrum. Practically, a zero absorbance line is used to measure the baseline noise in the spectrum.

3.10 *Background Deviation.* A deviation from 100 percent transmittance in any region

of the 100 percent line. Deviations greater than ±5 percent in an analytical region are unacceptable (absorbance of 0.021 to −0.022). Such deviations indicate a change in the instrument throughput relative to the background single beam.

3.11 *Batch Sampling.* A procedure where spectra of discreet, static samples are collected. The gas cell is filled with sample and the cell is isolated. The spectrum is collected. Finally, the cell is evacuated to prepare for the next sample.

3.12 *Continuous Sampling.* A procedure where spectra are collected while sample gas is flowing through the cell at a measured rate.

3.13 *Sampling resolution.* The spectral resolution used to collect sample spectra.

3.14 *Truncation.* Limiting the number of interferogram data points by deleting points farthest from the center burst (zero path difference, ZPD).

3.15 *Zero filling.* The addition of points to the interferogram. The position of each added point is interpolated from neighboring real data points. Zero filling adds no information to the interferogram, but affects line shapes in the absorbance spectrum (and possibly analytical results).

3.16 *Reference CTS.* Calibration Transfer Standard spectra that were collected with reference spectra.

3.17 *CTS Standard.* CTS spectrum produced by applying a de-resolution procedure to a reference CTS.

3.18 *Test CTS.* CTS spectra collected at the sampling resolution using the same optical configuration as for sample spectra. Test spectra help verify the resolution, temperature and path length of the FTIR system.

3.19 *RMSD.* Root Mean Square Difference, defined in EPA FTIR Protocol, appendix A.

3.20 *Sensitivity.* The noise-limited compound-dependent detection limit for the FTIR system configuration. This is estimated by the MAU. It depends on the RMSD in an analytical region of a zero absorbance line.

3.21 *Quantitation Limit.* The lower limit of detection for the FTIR system configuration in the sample spectra. This is estimated by mathematically subtracting scaled reference spectra of analytes and interferences from sample spectra, then measuring the RMSD in an analytical region of the subtracted spectrum. Since the noise in subtracted sample spectra may be much greater than in a zero absorbance spectrum, the quantitation limit is generally much higher than the sensitivity. Removing spectral interferences from the sample or improving the spectral subtraction can lower the quantitation limit toward (but not below) the sensitivity.

3.22 *Independent Sample.* A unique volume of sample gas; there is no mixing of gas between two consecutive independent samples. In continuous sampling two independent samples are separated by at least 5 cell volumes. The interval between independent measurements depends on the cell volume and the sample flow rate (through the cell).

3.23 *Measurement.* A single spectrum of flue gas contained in the FTIR cell.

3.24 *Run.* A run consists of a series of measurements. At a minimum a run includes 8 independent measurements spaced over 1 hour.

3.25 *Validation.* Validation of FTIR measurements is described in sections 13.0 through 13.4 of this method. Validation is used to verify the test procedures for measuring specific analytes at a source. Validation provides proof that the method works under certain test conditions.

3.26 *Validation Run.* A validation run consists of at least 24 measurements of independent samples. Half of the samples are spiked and half are not spiked. The length of the run is determined by the interval between independent samples.

3.27 *Screening.* Screening is used when there is little or no available information about a source. The purpose of screening is to determine what analytes are emitted and to obtain information about important sample characteristics such as moisture, temperature, and interferences. Screening results are semi-quantitative (estimated concentrations) or qualitative (identification only). Various optical and sampling configurations may be used. Sample conditioning systems may be evaluated for their effectiveness in removing interferences. It is unnecessary to perform a complete run under any set of sampling conditions. Spiking is not necessary, but spiking can be a useful screening tool for evaluating the sampling system, especially if a reactive or soluble analyte is used for the spike.

3.28 *Emissions Test.* An FTIR emissions test is performed according specific sampling and analytical procedures. These procedures, for the target analytes and the source, are based on previous screening and validation results. Emission results are quantitative. A QA spike (sections 8.6.2 and 9.2 of this method) is performed under each set of sampling conditions using a representative analyte. Flow, gas temperature and diluent data are recorded concurrently with the FTIR measurements to provide mass emission rates for detected compounds.

3.29 *Surrogate.* A surrogate is a compound that is used in a QA spike procedure (section 8.6.2 of this method) to represent other compounds. The chemical and physical properties of a surrogate shall be similar to the compounds it is chosen to represent. Under given sampling conditions, usually a single sampling factor is of primary concern for measuring the target analytes: for example, the surrogate spike results can be representative for analytes that are more reactive, more soluble, have a lower absorptivity, or have a lower vapor pressure than the surrogate itself.

4.0 Interferences

Interferences are divided into two classifications: analytical and sampling.

4.1 *Analytical Interferences.* An analytical interference is a spectral feature that complicates (in extreme cases may prevent) the analysis of an analyte. Analytical interferences are classified as background or spectral interference.

4.1.1 *Background Interference.* This results from a change in throughput relative to the single beam background. It is corrected by collecting a new background and proceeding with the test. In severe instances the cause must be identified and corrected.

Potential causes include: (1) Deposits on reflective surfaces or transmitting windows, (2) changes in detector sensitivity, (3) a change in the infrared source output, or (4) failure in the instrument electronics. In routine sampling throughput may degrade over several hours. Periodically a new background must be collected, but no other corrective action will be required.

4.1.2 *Spectral Interference.* This results from the presence of interfering compound(s) (interferant) in the sample. Interferant spectral features overlap analyte spectral features. Any compound with an infrared spectrum, including analytes, can potentially be an interferant. The Protocol measures absorbance band overlap in each analytical region to determine if potential interferants shall be classified as known interferants (FTIR Protocol, section 4.9 and appendix B). Water vapor and CO_2 are common spectral interferants. Both of these compounds have strong infrared spectra and are present in many sample matrices at high concentrations relative to analytes. The extent of interference depends on the (1) interferant concentration, (2) analyte concentration, and (3) the degree of band overlap. Choosing an alternate analytical region can minimize or avoid the spectral interference. For example, CO_2 interferes with the analysis of the 670 cm_{-1} benzene band. However, benzene can also be measured near 3000 cm⁻¹ (with less sensitivity).

4.2 *Sampling System Interferences.* These prevent analytes from reaching the instrument. The analyte spike procedure is designed to measure sampling system interference, if any.

4.2.1 *Temperature.* A temperature that is too low causes condensation of analytes or water vapor. The materials of the sampling system and the FTIR gas cell usually set the upper limit of temperature.

4.2.2 *Reactive Species.* Anything that reacts with analytes. Some analytes, like formaldehyde, polymerize at lower temperatures.

4.2.3 *Materials.* Poor choice of material for probe, or sampling line may remove some analytes. For example, HF reacts with glass components.

4.2.4 *Moisture.* In addition to being a spectral interferant, condensed moisture removes soluble compounds.

5.0 Safety

The hazards of performing this method are those associated with any stack sampling method and the same precautions shall be followed. Many HAPs are suspected carcinogens or present other serious health risks. Exposure to these compounds should be avoided in all circumstances. For instructions on the safe handling of any particular compound, refer to its material safety data sheet. When using analyte standards, always ensure that gases are properly vented and that the gas handling system is leak free. (Always perform a leak check with the system under maximum vacuum and, again, with the system at greater than ambient pressure.) Refer to section 8.2 of this method for leak check procedures. This method does not address all of the potential safety risks associated with its use. Anyone performing this method must follow

safety and health practices consistent with applicable legal requirements and with prudent practice for each application.

6.0 Equipment and Supplies

Note: Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

The equipment and supplies are based on the schematic of a sampling system shown in Figure 1. Either the batch or continuous sampling procedures may be used with this sampling system. Alternative sampling configurations may also be used, provided that the data quality objectives are met as determined in the post-analysis evaluation. Other equipment or supplies may be necessary, depending on the design of the sampling system or the specific target analytes.

6.1 *Sampling Probe.* Glass, stainless steel, or other appropriate material of sufficient length and physical integrity to sustain heating, prevent adsorption of analytes, and to transport analytes to the infrared gas cell. Special materials or configurations may be required in some applications. For instance, high stack sample temperatures may require special steel or cooling the probe. For very high moisture sources it may be desirable to use a dilution probe.

6.2 *Particulate Filters.* A glass wool plug (optional) inserted at the probe tip (for large particulate removal) and a filter (required) rated for 99 percent removal efficiency at 1-micron (e.g., Balston'') connected at the outlet of the heated probe.

6.3 *Sampling Line/Heating System.* Heated (sufficient to prevent condensation) stainless steel, polytetrafluoroethane, or other material inert to the analytes.

6.4 *Gas Distribution Manifold.* A heated manifold allowing the operator to control flows of gas standards and samples directly to the FTIR system or through sample conditioning systems. Usually includes heated flow meter, heated valve for selecting and sending sample to the analyzer, and a by-pass vent. This is typically constructed of stainless steel tubing and fittings, and high-temperature valves.

6.5 *Stainless Steel Tubing.* Type 316, appropriate diameter (e.g., 3/8 in.) and length for heated connections. Higher grade stainless may be desirable in some applications.

6.6 *Calibration/Analyte Spike Assembly.* A three way valve assembly (or equivalent) to introduce analyte or surrogate spikes into the sampling system at the outlet of the probe upstream of the out-of-stack particulate filter and the FTIR analytical system.

6.7 *Mass Flow Meter (MFM).* These are used for measuring analyte spike flow. The MFM shall be calibrated in the range of 0 to 5 L/min and be accurate to ± 2 percent (or better) of the flow meter span.

6.8 *Gas Regulators.* Appropriate for individual gas standards.

6.9 *Polytetrafluoroethane Tubing.* Diameter (e.g., 3/8 in.) and length suitable to connect cylinder regulators to gas standard manifold.

6.10 *Sample Pump.* A leak-free pump (e.g., KNF™), with by-pass valve, capable of producing a sample flow rate of at least 10

L/min through 100 ft of sample line. If the pump is positioned upstream of the distribution manifold and FTIR system, use a heated pump that is constructed from materials non-reactive to the analytes. If the pump is located downstream of the FTIR system, the gas cell sample pressure will be lower than ambient pressure and it must be recorded at regular intervals.

6.11 *Gas Sample Manifold.* Secondary manifold to control sample flow at the inlet to the FTIR manifold. This is optional, but includes a by-pass vent and heated rotameter.

6.12 *Rotameter.* A 0 to 20 L/min rotameter. This meter need not be calibrated.

6.13 *FTIR Analytical System.* Spectrometer and detector, capable of measuring the analytes to the chosen detection limit. The system shall include a personal computer with compatible software allowing automated collection of spectra.

6.14 *FTIR Cell Pump.* Required for the batch sampling technique, capable of evacuating the FTIR cell volume within 2 minutes. The pumping speed shall allow the operator to obtain 8 sample spectra in 1 hour.

6.15 *Absolute Pressure Gauge.* Capable of measuring pressure from 0 to 1000 mmHg to within ± 2.5 mmHg (e.g., Baratron™).

6.16 *Temperature Gauge.* Capable of measuring the cell temperature to within ± 2 °C.

6.17 *Sample Conditioning.* One option is a condenser system, which is used for moisture removal. This can be helpful in the measurement of some analytes. Other sample conditioning procedures may be devised for the removal of moisture or other interfering species.

6.17.1 The analyte spike procedure of section 9.2 of this method, the QA spike procedure of section 8.6.2 of this method, and the validation procedure of section 13 of this method demonstrate whether the sample conditioning affects analyte concentrations. Alternatively, measurements can be made with two parallel FTIR systems; one measuring conditioned sample, the other measuring unconditioned sample.

6.17.2 Another option is sample dilution. The dilution factor measurement must be documented and accounted for in the reported concentrations. An alternative to dilution is to lower the sensitivity of the FTIR system by decreasing the cell path length, or to use a short-path cell in conjunction with a long path cell to measure more than one concentration range.

7.0 Reagents and Standards

7.1 *Analyte(s) and Tracer Gas.* Obtain a certified gas cylinder mixture containing all of the analyte(s) at concentrations within ± 2 percent of the emission source levels (expressed in ppm-meter/K). If practical, the analyte standard cylinder shall also contain the tracer gas at a concentration which gives a measurable absorbance at a dilution factor of at least 10:1. Two ppm SF_6 is sufficient for a path length of 22 meters at 250 °F.

7.2 *Calibration Transfer Standard(s).* Select the calibration transfer standards (CTS) according to section 4.5 of the FTIR Protocol. Obtain a National Institute of Standards and Technology (NIST) traceable gravimetric standard of the CTS (± 2 percent).

7.3 *Reference Spectra.* Obtain reference spectra for each analyte, interferant, surrogate, CTS, and tracer. If EPA reference spectra are not available, use reference spectra prepared according to procedures in section 4.6 of the EPA FTIR Protocol.

8.0 Sampling and Analysis Procedure

Three types of testing can be performed: (1) Screening, (2) emissions test, and (3) validation. Each is defined in section 3 of this method. Determine the purpose(s) of the FTIR test. Test requirements include: (a) AU_i, DL_i, overall fractional uncertainty, OFU_i, maximum expected concentration ($CMAX_i$), and t_{AN} for each, (b) potential interferants, (c) sampling system factors, e.g., minimum absolute cell pressure, (P_{min}), FTIR cell volume (V_{SS}), estimated sample absorption pathlength, L_S', estimated sample pressure, P_S', T_S', signal integration time (t_{SS}), minimum instrumental linewidth, MIL, fractional error, and (d) analytical regions, e.g., $m = 1$ to M, lower wavenumber position, FL_m, center wavenumber position, FC_m, and upper wavenumber position, FU_m, plus interferants, upper wavenumber position of the CTS absorption band, FFU_m, lower wavenumber position of the CTS absorption band, FFL_m, wavenumber range FNU to FNL. If necessary, sample and acquire an initial spectrum. From analysis of this preliminary spectrum determine a suitable operational path length. Set up the sampling train as shown in Figure 1 or use an appropriate alternative configuration. Sections 8.1 through 8.11 of this method provide guidance on pre-test calculations in the EPA protocol, sampling and analytical procedures, and post-test protocol calculations.

8.1 *Pretest Preparations and Evaluations.* Using the procedure in section 4.0 of the FTIR Protocol, determine the optimum sampling system configuration for measuring the target analytes. Use available information to make reasonable assumptions about moisture content and other interferences.

8.1.1 *Analytes.* Select the required detection limit (DL_i) and the maximum permissible analytical uncertainty (AU_i) for each analyte (labeled from 1 to i). Estimate, if possible, the maximum expected concentration for each analyte, $CMAX_i$. The expected measurement range is fixed by DL_i and $CMAX_i$ for each analyte (i).

8.1.2 *Potential Interferants.* List the potential interferants. This usually includes water vapor and CO_2, but may also include some analytes and other compounds.

8.1.3 *Optical Configuration.* Choose an optical configuration that can measure all of the analytes within the absorbance range of .01 to 1.0 (this may require more than one path length). Use Protocol sections 4.3 to 4.8 for guidance in choosing a configuration and measuring CTS.

8.1.4 *Fractional Reproducibility Uncertainty (FRU_i).* The FRU is determined for each analyte by comparing CTS spectra taken before and after the reference spectra were measured. The EPA para-xylene reference spectra were collected on 10/31/91 and 11/01/91 with corresponding CTS spectra ''cts1031a,'' and

"cts1101b." The CTS spectra are used to estimate the reproducibility (FRU) in the system that was used to collect the references. The FRU must be < AU. Appendix E of the protocol is used to calculate the FRU from CTS spectra. Figure 2 plots results for 0.25 cm^{-1} CTS spectra in EPA reference library: S_3 (cts1101b − cts1031a), and S_4 [(cts1101b+cts1031a)/2]. The RMSD (SRMS) is calculated in the subtracted baseline, S_3, in the corresponding CTS region from 850 to 1065 cm^{-1}. The area (BAV) is calculated in the same region of the averaged CTS spectrum, S_4.

8.1.5 *Known Interferants.* Use appendix B of the EPA FTIR Protocol.

8.1.6 Calculate the Minimum Analyte Uncertainty, MAU (section 1.3 of this method discusses MAU and protocol appendix D gives the MAU procedure). The MAU for each analyte, i, and each analytical region, m, depends on the RMS noise.

8.1.7 *Analytical Program.* See FTIR Protocol, section 4.10. Prepare computer program based on the chosen analytical technique. Use as input reference spectra of all target analytes and expected interferants. Reference spectra of additional compounds shall also be included in the program if their presence (even if transient) in the samples is considered possible. The program output shall be in ppm (or ppb) and shall be corrected for differences between the reference path length, L_R, temperature, T_R, and pressure, P_R, and the conditions used for collecting the sample spectra. If sampling is performed at ambient pressure, then any pressure correction is usually small relative to corrections for path length and temperature, and may be neglected.

8.2 *Leak-Check*

8.2.1 *Sampling System.* A typical FTIR extractive sampling train is shown in Figure 1. Leak check from the probe tip to pump outlet as follows: Connect a 0-to 250-mL/min rate meter (rotameter or bubble meter) to the outlet of the pump. Close off the inlet to the probe, and record the leak rate. The leak rate shall be ≤200 mL/min.

8.2.2 *Analytical System Leak check.* Leak check the FTIR cell under vacuum and under pressure (greater than ambient). Leak check connecting tubing and inlet manifold under pressure.

8.2.2.1 For the evacuated sample technique, close the valve to the FTIR cell, and evacuate the absorption cell to the minimum absolute pressure P_{min}. Close the valve to the pump, and determine the change in pressure $\triangle P_v$ after 2 minutes.

8.2.2.2 For both the evacuated sample and purging techniques, pressurize the system to about 100 mmHg above atmospheric pressure. Isolate the pump and determine the change in pressure $\triangle P_p$ after 2 minutes.

8.2.2.3 Measure the barometric pressure, P_b in mmHg.

8.2.2.4 Determine the percent leak volume %V_L for the signal integration time t_{SS} and for $\triangle P_{max}$, i.e., the larger of $\triangle P_v$ or $\triangle P_p$, as follows:

$$\%V_L = 50t_{SS}\frac{\triangle P_{max}}{P_{SS}} \qquad (2)$$

where 50 = 100% divided by the leak-check time of 2 minutes. 8.2.2.5 Leak volumes in excess of 4 percent of the FTIR system volume V_{SS} are unacceptable.

8.3 *Detector Linearity.* Once an optical configuration is chosen, use one of the procedures of sections 8.3.1 through 8.3.3 to verify that the detector response is linear. If the detector response is not linear, decrease the aperture, or attenuate the infrared beam. After a change in the instrument configuration perform a linearity check until it is demonstrated that the detector response is linear.

8.3.1 Vary the power incident on the detector by modifying the aperture setting. Measure the background and CTS at three instrument aperture settings: (1) at the aperture setting to be used in the testing, (2) at one half this aperture and (3) at twice the proposed testing aperture. Compare the three CTS spectra. CTS band areas shall agree to within the uncertainty of the cylinder standard and the RMSD noise in the system. If test aperture is the maximum aperture, collect CTS spectrum at maximum aperture, then close the aperture to reduce the IR throughput by half. Collect a second background and CTS at the smaller aperture setting and compare the spectra again.

8.3.2 Use neutral density filters to attenuate the infrared beam. Set up the FTIR system as it will be used in the test measurements. Collect a CTS spectrum. Use a neutral density filter to attenuate the infrared beam (either immediately after the source or the interferometer) to approximately ½ its original intensity. Collect a second CTS spectrum. Use another filter to attenuate the infrared beam to approximately ¼ its original intensity. Collect a third background and CTS spectrum. Compare the CTS spectra. CTS band areas shall agree to within the uncertainty of the cylinder standard and the RMSD noise in the system.

8.3.3 Observe the single beam instrument response in a frequency region where the detector response is known to be zero. Verify that the detector response is "flat" and equal to zero in these regions.

8.4 *Data Storage Requirements.* All field test spectra shall be stored on a computer disk and a second backup copy must stored on a separate disk. The stored information includes sample interferograms, processed absorbance spectra, background interferograms, CTS sample interferograms and CTS absorbance spectra. Additionally, documentation of all sample conditions, instrument settings, and test records must be recorded on hard copy or on computer medium. Table 1 gives a sample presentation of documentation.

8.5 *Background Spectrum.* Evacuate the gas cell to ≤5 mmHg, and fill with dry nitrogen gas to ambient pressure (or purge the cell with 10 volumes of dry nitrogen). Verify that no significant amounts of absorbing species (for example water vapor and CO_2) are present. Collect a background spectrum, using a signal averaging period equal to or greater than the averaging period for the sample spectra. Assign a unique file name to the background spectrum. Store two copies of the background interferogram and

processed single-beam spectrum on separate computer disks (one copy is the back-up).

8.5.1 *Interference Spectra.* If possible, collect spectra of known and suspected major interferences using the same optical system that will be used in the field measurements. This can be done on-site or earlier. A number of gases, e.g. CO_2, SO_2, CO, NH_3, are readily available from cylinder gas suppliers.

8.5.2 Water vapor spectra can be prepared by the following procedure. Fill a sample tube with distilled water. Evacuate above the sample and remove dissolved gasses by alternately freezing and thawing the water while evacuating. Allow water vapor into the FTIR cell, then dilute to atmospheric pressure with nitrogen or dry air. If quantitative water spectra are required, follow the reference spectrum procedure for neat samples (protocol, section 4.6). Often, interference spectra need not be quantitative, but for best results the absorbance must be comparable to the interference absorbance in the sample spectra.

8.6 *Pre-Test Calibrations.*

8.6.1 *Calibration Transfer Standard.* Evacuate the gas cell to ≤ 5 mmHg absolute pressure, and fill the FTIR cell to atmospheric pressure with the CTS gas. Alternatively, purge the cell with 10 cell volumes of CTS gas. (If purge is used, verify that the CTS concentration in the cell is stable by collecting two spectra 2 minutes apart as the CTS gas continues to flow. If the absorbance in the second spectrum is no greater than in the first, within the uncertainty of the gas standard, then this can be used as the CTS spectrum.) Record the spectrum.

8.6.2 *QA Spike.* This procedure assumes that the method has been validated for at least some of the target analytes at the source. For emissions testing perform a QA spike. Use a certified standard, if possible, of an analyte, which has been validated at the source. One analyte standard can serve as a QA surrogate for other analytes which are less reactive or less soluble than the standard. Perform the spike procedure of section 9.2 of this method. Record spectra of at least three independent (section 3.22 of this method) spiked samples. Calculate the spiked component of the analyte concentration. If the average spiked concentration is within 0.7 to 1.3 times the expected concentration, then proceed with the testing. If applicable, apply the correction factor from the Method 301 of this appendix validation test (not the result from the QA spike).

8.7 *Sampling.* If analyte concentrations vary rapidly with time, continuous sampling is preferable using the smallest cell volume, fastest sampling rate and fastest spectra collection rate possible. Continuous sampling requires the least operator intervention even without an automated sampling system. For continuous monitoring at one location over long periods, Continuous sampling is preferred. Batch sampling and continuous static sampling are used for screening and performing test runs of finite duration. Either technique is preferred for sampling several

locations in a matter of days. Batch sampling gives reasonably good time resolution and ensures that each spectrum measures a discreet (and unique) sample volume. Continuous static (and continuous) sampling provide a very stable background over long periods. Like batch sampling, continuous static sampling also ensures that each spectrum measures a unique sample volume. It is essential that the leak check procedure under vacuum (section 8.2 of this method) is passed if the batch sampling procedure is used. It is essential that the leak check procedure under positive pressure is passed if the continuous static or continuous sampling procedures are used. The sampling techniques are described in sections 8.7.1 through 8.7.2 of this method.

8.7.1 *Batch Sampling.* Evacuate the absorbance cell to ≤5 mmHg absolute pressure. Fill the cell with exhaust gas to ambient pressure, isolate the cell, and record the spectrum. Before taking the next sample, evacuate the cell until no spectral evidence of sample absorption remains. Repeat this procedure to collect eight spectra of separate samples in 1 hour.

8.7.2 *Continuous Static Sampling.* Purge the FTIR cell with 10 cell volumes of sample gas. Isolate the cell, collect the spectrum of the static sample and record the pressure. Before measuring the next sample, purge the cell with 10 more cell volumes of sample gas.

8.8 *Sampling QA and Reporting*

8.8.1 Sample integration times shall be sufficient to achieve the required signal-to-noise ratio. Obtain an absorbance spectrum by filling the cell with N2. Measure the RMSD in each analytical region in this absorbance spectrum. Verify that the number of scans used is sufficient to achieve the target MAU.

8.8.2 Assign a unique file name to each spectrum.

8.8.3 Store two copies of sample interferograms and processed spectra on separate computer disks.

8.8.4 For each sample spectrum, document the sampling conditions, the sampling time (while the cell was being filled), the time the spectrum was recorded, the instrumental conditions (path length,

temperature, pressure, resolution, signal integration time), and the spectral file name. Keep a hard copy of these data sheets.

8.9 *Signal Transmittance.* While sampling, monitor the signal transmittance. If signal transmittance (relative to the background) changes by 5 percent or more (absorbance = -.02 to .02) in any analytical spectral region, obtain a new background spectrum.

8.10 *Post-test CTS.* After the sampling run, record another CTS spectrum.

8.11 *Post-test QA*

8.11.1 Inspect the sample spectra immediately after the run to verify that the gas matrix composition was close to the expected (assumed) gas matrix.

8.11.2 Verify that the sampling and instrumental parameters were appropriate for the conditions encountered. For example, if the moisture is much greater than anticipated, it may be necessary to use a shorter path length or dilute the sample.

8.11.3 Compare the pre- and post-test CTS spectra. The peak absorbance in pre- and post-test CTS must be ±5 περψεντ οφ τηε μεαν ωαλθε. Σεε αππενδιχ Ε οφ τηε ΦΤΙΡ Προτοψολ.

9.0 *Quality Control.*

Use analyte spiking (sections 8.6.2, 9.2 and 13.0 of this method) to verify that the sampling system can transport the analytes from the probe to the FTIR system.

9.1 *Spike Materials.* Use a certified standard (accurate to ±2 percent) of the target analyte, if one can be obtained. If a certified standard cannot be obtained, follow the procedures in section 4.6.2.2 of the FTIR Protocol.

9.2 *Spiking Procedure.* QA spiking (section 8.6.2 of this method) is a calibration procedure used before testing. QA spiking involves following the spike procedure of sections 9.2.1 through 9.2.3 of this method to obtain at least three spiked samples. The analyte concentrations in the spiked samples shall be compared to the expected spike concentration to verify that the sampling/analytical system is working properly. Usually, when QA spiking is used, the method has already been validated at a similar source for the analyte in question. The QA spike demonstrates that the

validated sampling/analytical conditions are being duplicated. If the QA spike fails then the sampling/analytical system shall be repaired before testing proceeds. The method validation procedure (section 13.0 of this method) involves a more extensive use of the analyte spike procedure of sections 9.2.1 through 9.2.3 of this method. Spectra of at least 12 independent spiked and 12 independent unspiked samples are recorded. The concentration results are analyzed statistically to determine if there is a systematic bias in the method for measuring a particular analyte. If there is a systematic bias, within the limits allowed by Method 301 of this appendix, then a correction factor shall be applied to the analytical results. If the systematic bias is greater than the allowed limits, this method is not valid and cannot be used.

9.2.1 Introduce the spike/tracer gas at a constant flow rate of ≤10 percent of the total sample flow, when possible.

Note: Use the rotameter at the end of the sampling train to estimate the required spike/tracer gas flow rate.

Use a flow device, e.g., mass flow meter (± 2 percent), to monitor the spike flow rate. Record the spike flow rate every 10 minutes.

9.2.2 Determine the response time (RT) of the system by continuously collecting spectra of the spiked effluent until the spectrum of the spiked component is constant for 5 minutes. The RT is the interval from the first measurement until the spike becomes constant. Wait for twice the duration of the RT, then collect spectra of two independent spiked gas samples. Duplicate analyses of the spiked concentration shall be within 5 percent of the mean of the two measurements.

9.2.3 Calculate the dilution ratio using the tracer gas as follows: where:

$$DF = \frac{SF_{6(spk)}}{SF_{6(dir)}} \qquad (3)$$

Where:

$$CS = DF * Spike_{dir} + Unspike\,(1 - DF) \qquad (4)$$

DF=Dilution factor of the spike gas; this value shall be ≥10.
$SF_{6(dir)}$=SF_6 (or tracer gas) concentration measured directly in undiluted spike gas.
$SF_{6(spk)}$=Diluted SF_6 (or tracer gas) concentration measured in a spiked sample.
Spike^{dir}=Concentration of the analyte in the spike standard measured by filling the FTIR cell directly.
CS=Expected concentration of the spiked samples.
Unspike=Native concentration of analytes in unspiked samples.

10.0 Calibration and Standardization

10.1 *Signal-to-Noise Ratio (S/N).* The RMSD in the noise must be less than one

tenth of the minimum analyte peak absorbance in each analytical region. For example if the minimum peak absorbance is 0.01 at the required DL, then RMSD measured over the entire analytical region must be ≤0.001.

10.2 *Absorbance Path length.* Verify the absorbance path length by comparing reference CTS spectra to test CTS spectra. See appendix E of the FTIR Protocol.

10.3 *Instrument Resolution.* Measure the line width of appropriate test CTS band(s) to verify instrument resolution. Alternatively, compare CTS spectra to a reference CTS spectrum, if available, measured at the nominal resolution.

10.4 *Apodization Function.* In transforming the sample interferograms to absorbance spectra use the same apodization

function that was used in transforming the reference spectra.

10.5 *FTIR Cell Volume.* Evacuate the cell to ≤5 mmHg. Measure the initial absolute temperature (T_i) and absolute pressure (P_i). Connect a wet test meter (or a calibrated dry gas meter), and slowly draw room air into the cell. Measure the meter volume (V_m), meter absolute temperature (T_m), and meter absolute pressure (P_m); and the cell final absolute temperature (T_f) and absolute pressure (P_f). Calculate the FTIR cell volume VSS, including that of the connecting tubing, as follows:

$$V_{SS} = \frac{V_m \dfrac{P_m}{T_m}}{\left[\dfrac{P_f}{T_f} - \dfrac{P_i}{T_i}\right]} \qquad (5)$$

11.0 Data Analysis and Calculations

Analyte concentrations shall be measured using reference spectra from the EPA FTIR spectral library. When EPA library spectra are not available, the procedures in section 4.6 of the Protocol shall be followed to prepare reference spectra of all the target analytes.

11.1 *Spectral De-resolution.* Reference spectra can be converted to lower resolution standard spectra (section 3.3 of this method) by truncating the original reference sample and background interferograms. Appendix K of the FTIR Protocol gives specific deresolution procedures. Deresolved spectra shall be transformed using the same apodization function and level of zero filling as the sample spectra. Additionally, pre-test FTIR protocol calculations (e.g., FRU, MAU, FCU) shall be performed using the de-resolved standard spectra.

11.2 *Data Analysis.* Various analytical programs are available for relating sample absorbance to a concentration standard. Calculated concentrations shall be verified by analyzing residual baselines after mathematically subtracting scaled reference spectra from the sample spectra. A full description of the data analysis and calculations is contained in the FTIR Protocol (sections 4.0, 5.0, 6.0 and appendices). Correct the calculated concentrations in the sample spectra for differences in absorption path length and temperature between the reference and sample spectra using equation 6,

$$C_{corr} = \left(\frac{L_r}{L_s}\right)\left(\frac{T_s}{T_r}\right)\left(\frac{P_r}{P_s}\right) C_{calc} \qquad (6)$$

Where:

C_{corr}=Concentration, corrected for path length.
C_{calc}=Concentration, initial calculation (output of the analytical program designed for the compound).
L_r=Reference spectra path length.
L_s=Sample spectra path length.
T_s=Absolute temperature of the sample gas, K.
T_r=Absolute gas temperature of reference spectra, K.
P_s=Sample cell pressure.
P_r=Reference spectrum sample pressure.

12.0 Method Performance

12.1 *Spectral Quality.* Refer to the FTIR Protocol appendices for analytical requirements, evaluation of data quality, and analysis of uncertainty.

12.2 *Sampling QA/QC.* The analyte spike procedure of section 9 of this method, the QA spike of section 8.6.2 of this method, and the validation procedure of section 13 of this method are used to evaluate the performance of the sampling system and to quantify sampling system effects, if any, on the measured concentrations. This method is self-validating provided that the results meet the performance requirement of the QA spike in sections 9.0 and 8.6.2 of this method and results from a previous method validation study support the use of this method in the application. Several factors can contribute to uncertainty in the measurement of spiked samples. Factors which can be controlled to provide better accuracy in the spiking procedure are listed in sections 12.2.1 through 12.2.4 of this method.

12.2.1 *Flow meter.* An accurate mass flow meter is accurate to ±1 percent of its span. If a flow of 1 L/min is monitored with such a MFM, which is calibrated in the range of 0–5 L/min, the flow measurement has an uncertainty of 5 percent. This may be improved by re-calibrating the meter at the specific flow rate to be used.

12.2.2 *Calibration gas.* Usually the calibration standard is certified to within ± 2 percent. With reactive analytes, such as HCl, the certified accuracy in a commercially available standard may be no better than ± 5 percent.

12.2.3 *Temperature.* Temperature measurements of the cell shall be quite accurate. If practical, it is preferable to measure sample temperature directly, by inserting a thermocouple into the cell chamber instead of monitoring the cell outer wall temperature.

12.2.4 *Pressure.* Accuracy depends on the accuracy of the barometer, but fluctuations in pressure throughout a day may be as much as 2.5 percent due to weather variations.

13.0 *Method Validation Procedure*

This validation procedure, which is based on EPA Method 301 (40 CFR part 63, appendix (A), may be used to validate this method for the analytes in a gas matrix. Validation at one source may also apply to another type of source, if it can be shown that the exhaust gas characteristics are similar at both sources.

13.1 Section 5.3 of Method 301 (40 CFR part 63, appendix A), the Analyte Spike procedure, is used with these modifications. The statistical analysis of the results follows section 6.3 of EPA Method 301. Section 3 of this method defines terms that are not defined in Method 301.

13.1.1 The analyte spike is performed dynamically. This means the spike flow is continuous and constant as spiked samples are measured.

13.1.2 The spike gas is introduced at the back of the sample probe.

13.1.3 Spiked effluent is carried through all sampling components downstream of the probe.

13.1.4 A single FTIR system (or more) may be used to collect and analyze spectra (not quadruplicate integrated sampling trains).

13.1.5 All of the validation measurements are performed sequentially in a single "run" (section 3.26 of this method).

13.1.6 The measurements analyzed statistically are each independent (section 3.22 of this method).

13.1.7 A validation data set can consist of more than 12 spiked and 12 unspiked measurements.

13.2 *Batch Sampling.* The procedure in sections 13.2.1 through 13.2.2 may be used for stable processes. If process emissions are highly variable, the procedure in section 13.2.3 shall be used.

13.2.1 With a single FTIR instrument and sampling system, begin by collecting spectra of two unspiked samples. Introduce the spike flow into the sampling system and allow 10 cell volumes to purge the sampling system and FTIR cell. Collect spectra of two spiked samples. Turn off the spike and allow 10 cell volumes of unspiked sample to purge the FTIR cell. Repeat this procedure until the 24 (or more) samples are collected.

13.2.2 In batch sampling, collect spectra of 24 distinct samples. (Each distinct sample consists of filling the cell to ambient pressure after the cell has been evacuated.)

13.2.3 Alternatively, a separate probe assembly, line, and sample pump can be used for spiked sample. Verify and document that sampling conditions are the same in both the spiked and the unspiked sampling systems. This can be done by wrapping both sample lines in the same heated bundle. Keep the same flow rate in both sample lines. Measure samples in sequence in pairs. After two spiked samples are measured, evacuate the FTIR cell, and turn the manifold valve so that spiked sample flows to the FTIR cell. Allow the connecting line from the manifold to the FTIR cell to purge thoroughly (the time depends on the line length and flow rate). Collect a pair of spiked samples. Repeat the procedure until at least 24 measurements are completed.

13.3 Simultaneous Measurements With Two FTIR Systems. If unspiked effluent concentrations of the target analyte(s) vary significantly with time, it may be desirable to perform synchronized measurements of spiked and unspiked sample. Use two FTIR systems, each with its own cell and sampling system to perform simultaneous spiked and unspiked measurements. The optical configurations shall be similar, if possible. The sampling configurations shall be the same. One sampling system and FTIR analyzer shall be used to measure spiked effluent. The other sampling system and FTIR analyzer shall be used to measure unspiked flue gas. Both systems shall use the same sampling procedure (i.e., batch or continuous).

13.3.1 If batch sampling is used, synchronize the cell evacuation, cell filling, and collection of spectra. Fill both cells at the same rate (in cell volumes per unit time).

13.3.2 If continuous sampling is used, adjust the sample flow through each gas cell so that the same number of cell volumes pass through each cell in a given time (i.e. $TC_1 = TC_2$).

13.4 *Statistical Treatment.* The statistical procedure of EPA Method 301 of this appendix, section 6.3 is used to evaluate the bias and precision. For FTIR testing a validation "run" is defined as spectra of 24 independent samples, 12 of which are spiked with the analyte(s) and 12 of which are not spiked.

13.4.1 *Bias.* Determine the bias (defined by EPA Method 301 of this appendix, section 6.3.2) using equation 7:

$$B = S_m - CS \qquad (7)$$

Where:

B = Bias at spike level.
S_m = Mean concentration of the analyte spiked samples.
CS = Expected concentration of the spiked samples.

13.4.2 *Correction Factor.* Use section 6.3.2.2 of Method 301 of this appendix to evaluate the statistical significance of the bias. If it is determined that the bias is significant, then use section 6.3.3 of Method 301 to calculate a correction factor (CF). Analytical results of the test method are multiplied by the correction factor, if $0.7 \leq CF \leq 1.3$. If is determined that the bias is significant and CF $>\pm30$ percent, then the test method is considered to "not valid."

13.4.3 If measurements do not pass validation, evaluate the sampling system, instrument configuration, and analytical system to determine if improper set-up or a malfunction was the cause. If so, repair the system and repeat the validation.

14.0 *Pollution Prevention.*

The extracted sample gas is vented outside the enclosure containing the FTIR system and gas manifold after the analysis. In typical method applications the vented sample volume is a small fraction of the source volumetric flow and its composition is identical to that emitted from the source. When analyte spiking is used, spiked pollutants are vented with the extracted sample gas. Approximately 1.6×10^{-4} to 3.2×10^{-4} lbs of a single HAP may be vented to the atmosphere in a typical validation run of 3 hours. (This assumes a molar mass of 50 to 100 g, spike rate of 1.0 L/min, and a standard concentration of 100 ppm). Minimize emissions by keeping the spike flow off when not in use.

15.0 *Waste Management.*

Small volumes of laboratory gas standards can be vented through a laboratory hood. Neat samples must be packed and disposed according to applicable regulations. Surplus materials may be returned to supplier for disposal.

16.0 *References.*

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4. "Molecular Vibrations; The Theory of Infrared and Raman Vibrational Spectra," E. Bright Wilson, J. C. Decius, and P. C. Cross, Dover Publications, Inc., 1980. For a less intensive treatment of molecular rotational-vibrational spectra see, for example, "Physical Chemistry," G. M. Barrow, chapters 12, 13, and 14, McGraw Hill, Inc., 1979.

5. "Fourier Transform Infrared Spectrometry," Peter R. Griffiths and James de Haseth, Chemical Analysis, 83, 16–25,(1986), P. J. Elving, J. D. Winefordner and I. M. Kolthoff (ed.), John Wiley and Sons.

6. "Computer-Assisted Quantitative Infrared Spectroscopy," Gregory L. McClure (ed.), ASTM Special Publication 934 (ASTM), 1987.

7. "Multivariate Least-Squares Methods Applied to the Quantitative Spectral Analysis of Multicomponent Mixtures," Applied Spectroscopy, 39(10), 73–84, 1985.

TABLE 1.—EXAMPLE PRESENTATION OF SAMPLING DOCUMENTATION.

Sample time	Spectrum file name	Background file name	Sample conditioning	Process condition

Sample time	Spectrum file	Interferogram	Resolution	Scans	Apodization	Gain	CTS Spectrum
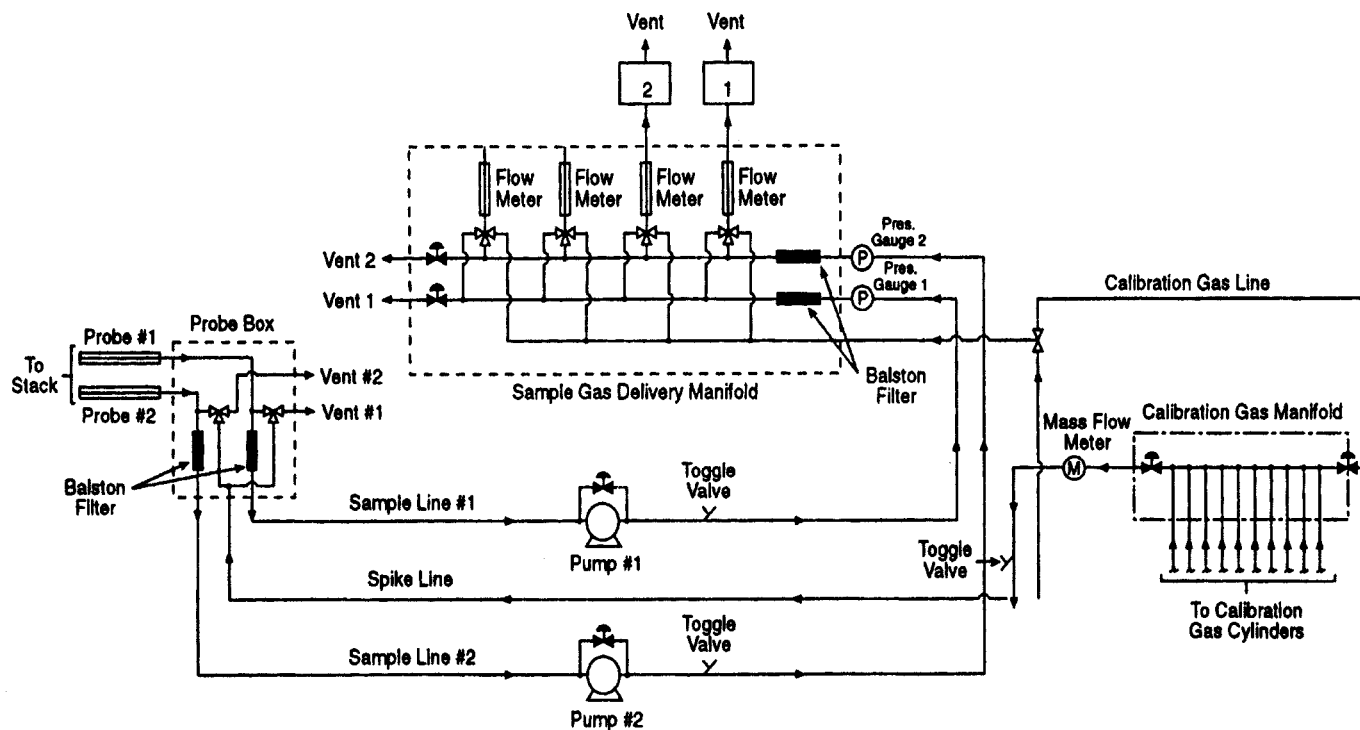
Figure 1. Extractive FTIR sampling system.

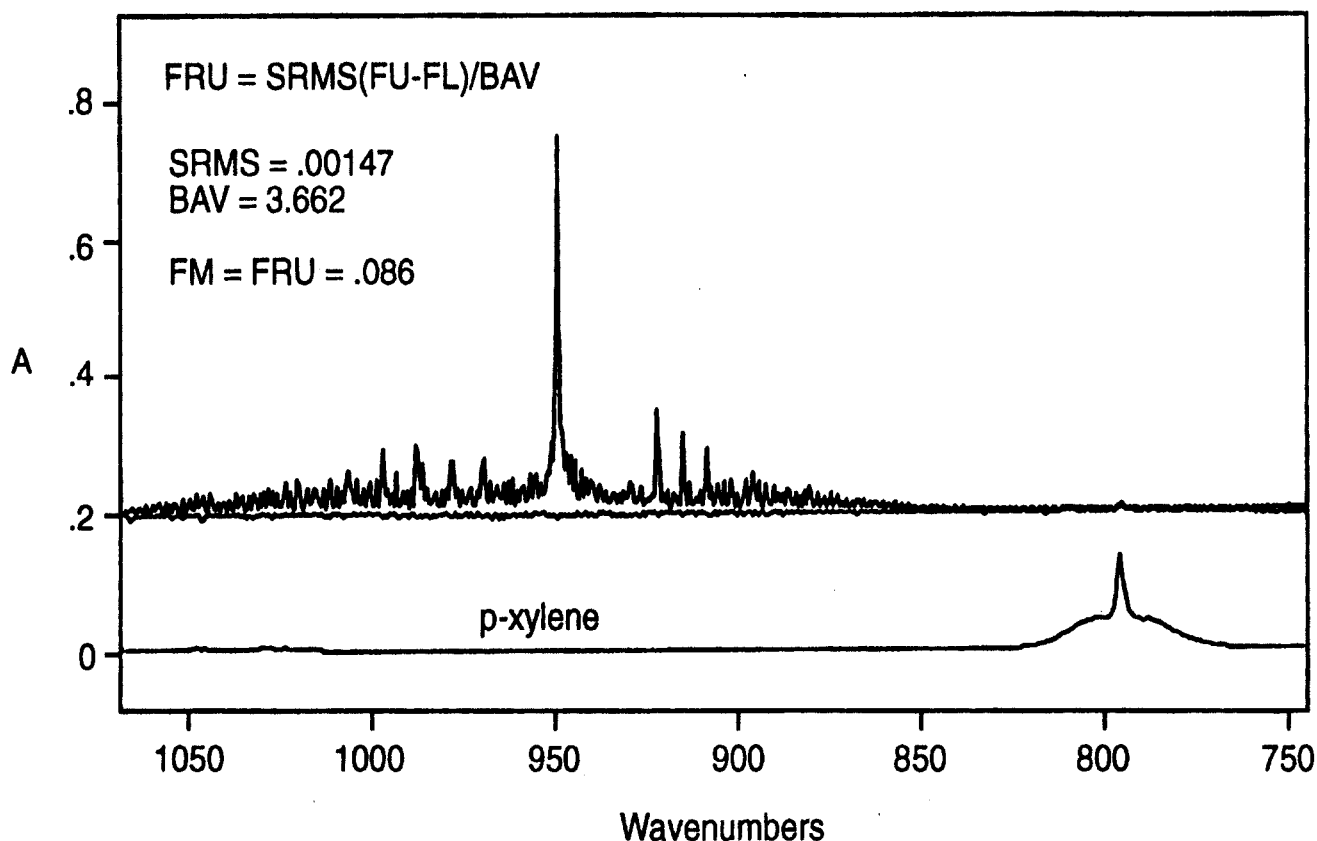FRU = SRMS(FU-FL)/BAV

SRMS = .00147
BAV = 3.662

FM = FRU = .086

p-xylene

Figure 2. Fractional Reproducibility. Top: average of cts1031a and cts1101b. Bottom: Reference spectrum of p-xylene.

Addendum to Test Method 320—Protocol for the Use of Extractive Fourier Transform Infrared (FTIR) Spectrometry for the Analyses of Gaseous Emissions from Stationary Sources

1.0 *Introduction*

The purpose of this addendum is to set general guidelines for the use of modern FTIR spectroscopic methods for the analysis of gas samples extracted from the effluent of stationary emission sources. This addendum outlines techniques for developing and evaluating such methods and sets basic requirements for reporting and quality assurance procedures.

1.1 *Nomenclature*

1.1.1 Appendix A to this addendum lists definitions of the symbols and terms used in this Protocol, many of which have been taken directly from American Society for Testing and Materials (ASTM) publication E 131–90a, entitled "Terminology Relating to Molecular Spectroscopy."

1.1.2 Except in the case of background spectra or where otherwise noted, the term "spectrum" refers to a double-beam spectrum in units of absorbance vs. wavenumber (cm^{-1}).

1.1.3 The term "Study" in this addendum refers to a publication that has been subjected to EPA- or peer-review.

2.0 *Applicability and Analytical Principle*

2.1 *Applicability.* This Protocol applies to the determination of compound-specific concentrations in single- and multiple-component gas phase samples using double-beam absorption spectroscopy in the mid-infrared band. It does not specifically address other FTIR applications, such as single-beam spectroscopy, analysis of open-path (non-enclosed) samples, and continuous measurement techniques. If multiple spectrometers, absorption cells, or instrumental linewidths are used in such analyses, each distinct operational configuration of the system must be evaluated separately according to this Protocol.

2.2 *Analytical Principle*

2.2.1 In the mid-infrared band, most molecules exhibit characteristic gas phase absorption spectra that may be recorded by FTIR systems. Such systems consist of a source of mid-infrared radiation, an interferometer, an enclosed sample cell of known absorption pathlength, an infrared detector, optical elements for the transfer of infrared radiation between components, and gas flow control and measurement

components. Adjunct and integral computer systems are used for controlling the instrument, processing the signal, and for performing both Fourier transforms and quantitative analyses of spectral data.

2.2.2 The absorption spectra of pure gases and of mixtures of gases are described by a linear absorbance theory referred to as Beer's Law. Using this law, modern FTIR systems use computerized analytical programs to quantify compounds by comparing the absorption spectra of known (reference) gas samples to the absorption spectrum of the sample gas. Some standard mathematical techniques used for comparisons are classical least squares, inverse least squares, cross-correlation, factor analysis, and partial least squares. Reference A describes several of these techniques, as well as additional techniques, such as differentiation methods, linear baseline corrections, and non-linear absorbance corrections.

3.0 *General Principles of Protocol Requirements*

The characteristics that distinguish FTIR systems from gas analyzers used in instrumental gas analysis methods (e.g., Methods 6C and 7E of appendix A to part 60 of this chapter) are: (1) Computers are necessary to obtain and analyze data; (2) chemical concentrations can be quantified using previously recorded infrared reference

spectra; and (3) analytical assumptions and results, including possible effects of interfering compounds, can be evaluated after the quantitative analysis. The following general principles and requirements of this Protocol are based on these characteristics.

3.1 *Verifiability and Reproducibility of Results.* Store all data and document data analysis techniques sufficient to allow an independent agent to reproduce the analytical results from the raw interferometric data.

3.2 *Transfer of Reference Spectra.* To determine whether reference spectra recorded under one set of conditions (e.g., optical bench, instrumental linewidth, absorption pathlength, detector performance, pressure, and temperature) can be used to analyze sample spectra taken under a different set of conditions, quantitatively compare "calibration transfer standards" (CTS) and reference spectra as described in this Protocol. (**Note:** The CTS may, but need not, include analytes of interest). To effect this, record the absorption spectra of the CTS (a) immediately before and immediately after recording reference spectra and (b) immediately after recording sample spectra.

3.3 *Evaluation of FTIR Analyses.* The applicability, accuracy, and precision of FTIR measurements are influenced by a number of interrelated factors, which may be divided into two classes:

3.3.1 *Sample-Independent Factors.* Examples are system configuration and performance (e.g., detector sensitivity and infrared source output), quality and applicability of reference absorption spectra, and type of mathematical analyses of the spectra. These factors define the fundamental limitations of FTIR measurements for a given system configuration. These limitations may be estimated from evaluations of the system before samples are available. For example, the detection limit for the absorbing compound under a given set of conditions may be estimated from the system noise level and the strength of a particular absorption band. Similarly, the accuracy of measurements may be estimated from the analysis of the reference spectra.

3.3.2 *Sample-Dependent Factors.* Examples are spectral interferants (e.g., water vapor and CO_2) or the overlap of spectral features of different compounds and contamination deposits on reflective surfaces or transmitting windows. To maximize the effectiveness of the mathematical techniques used in spectral analysis, identification of interferants (a standard initial step) and analysis of samples (includes effect of other analytical errors) are necessary. Thus, the Protocol requires post-analysis calculation of measurement concentration uncertainties for the detection of these potential sources of measurement error.

4.0 *Pre-Test Preparations and Evaluations*

Before testing, demonstrate the suitability of FTIR spectrometry for the desired application according to the procedures of this section.

4.1 *Identify Test Requirements.* Identify and record the test requirements described in sections 4.1.1 through 4.1.4 of this addendum. These values set the desired or required goals of the proposed analysis; the

description of methods for determining whether these goals are actually met during the analysis comprises the majority of this Protocol.

4.1.1 Analytes (specific chemical species) of interest. Label the analytes from i = 1 to I.

4.1.2 Analytical uncertainty limit (AU_i). The AU_i is the maximum permissible fractional uncertainty of analysis for the ith analyte concentration, expressed as a fraction of the analyte concentration in the sample.

4.1.3 Required detection limit for each analyte (DL_i, ppm). The detection limit is the lowest concentration of an analyte for which its overall fractional uncertainty (OFU_i) is required to be less than its analytical uncertainty limit (AU_i).

4.1.4 Maximum expected concentration of each analyte ($CMAX_i$, ppm).

4.2 *Identify Potential Interferants.* Considering the chemistry of the process or results of previous studies, identify potential interferants, i.e., the major effluent constituents and any relatively minor effluent constituents that possess either strong absorption characteristics or strong structural similarities to any analyte of interest. Label them 1 through N_j, where the subscript "j" pertains to potential interferants. Estimate the concentrations of these compounds in the effluent ($CPOT_j$, ppm).

4.3 *Select and Evaluate the Sampling System.* Considering the source, e.g., temperature and pressure profiles, moisture content, analyte characteristics, and particulate concentration), select the equipment for extracting gas samples. Recommended are a particulate filter, heating system to maintain sample temperature above the dew point for all sample constituents at all points within the sampling system (including the filter), and sample conditioning system (e.g., coolers, water-permeable membranes that remove water or other compounds from the sample, and dilution devices) to remove spectral interferants or to protect the sampling and analytical components. Determine the minimum absolute sample system pressure (P_{min}, mmHg) and the infrared absorption cell volume (V_{SS}, liter). Select the techniques and/or equipment for the measurement of sample pressures and temperatures.

4.4 *Select Spectroscopic System.* Select a spectroscopic configuration for the application. Approximate the absorption pathlength (L_S', meter), sample pressure (P_S', kPa), absolute sample temperature T_S', and signal integration period (t_{SS}, seconds) for the analysis. Specify the nominal minimum instrumental linewidth (MIL) of the system. Verify that the fractional error at the approximate values P_S' and T_S' is less than one half the smallest value AU_i (see section 4.1.2 of this addendum).

4.5 Select Calibration Transfer Standards (CTS's). Select CTS's that meet the criteria listed in sections 4.5.1, 4.5.2, and 4.5.3 of this addendum.

Note: It may be necessary to choose preliminary analytical regions (see section 4.7 of this addendum), identify the minimum analyte linewidths, or estimate the system noise level (see section 4.12 of this

addendum) before selecting the CTS. More than one compound may be needed to meet the criteria; if so, obtain separate cylinders for each compound.

4.5.1 The central wavenumber position of each analytical region shall lie within 25 percent of the wavenumber position of at least one CTS absorption band.

4.5.2 The absorption bands in section 4.5.1 of this addendum shall exhibit peak absorbances greater than ten times the value RMS_{EST} (see section 4.12 of this addendum) but less than 1.5 absorbance units.

4.5.3 At least one absorption CTS band within the operating range of the FTIR instrument shall have an instrument-independent linewidth no greater than the narrowest analyte absorption band. Perform and document measurements or cite Studies to determine analyte and CTS compound linewidths.

4.5.4 For each analytical region, specify the upper and lower wavenumber positions (FFU_m and FFL_m, respectively) that bracket the CTS absorption band or bands for the associated analytical region. Specify the wavenumber range, FNU to FNL, containing the absorption band that meets the criterion of section 4.5.3 of this addendum.

4.5.5 Associate, whenever possible, a single set of CTS gas cylinders with a set of reference spectra. Replacement CTS gas cylinders shall contain the same compounds at concentrations within 5 percent of that of the original CTS cylinders; the entire absorption spectra (not individual spectral segments) of the replacement gas shall be scaled by a factor between 0.95 and 1.05 to match the original CTS spectra.

4.6 *Prepare Reference Spectra*

Note: Reference spectra are available in a permanent soft copy from the EPA spectral library on the EMTIC (Emission Measurement Technical Information Center) computer bulletin board; they may be used if applicable.

4.6.1 Select the reference absorption pathlength (L_R) of the cell.

4.6.2 Obtain or prepare a set of chemical standards for each analyte, potential and known spectral interferants, and CTS. Select the concentrations of the chemical standards to correspond to the top of the desired range.

4.6.2.1 *Commercially-Prepared Chemical Standards.* Chemical standards for many compounds may be obtained from independent sources, such as a specialty gas manufacturer, chemical company, or commercial laboratory. These standards (accurate to within ±2 percent) shall be prepared according to EPA Traceability Protocol (see Reference D) or shall be traceable to NIST standards. Obtain from the supplier an estimate of the stability of the analyte concentration. Obtain and follow all of the supplier's recommendations for recertifying the analyte concentration.

4.6.2.2 *Self-Prepared Chemical Standards.* Chemical standards may be prepared by diluting certified commercially prepared chemical gases or pure analytes with ultra-pure carrier (UPC) grade nitrogen according to the barometric and volumetric techniques generally described in Reference A, section A4.6.

4.6.3 Record a set of the absorption spectra of the CTS {R1}, then a set of the reference spectra at two or more concentrations in duplicate over the desired range (the top of the range must be less than 10 times that of the bottom), followed by a second set of CTS spectra {R2}. (If self-prepared standards are used, see section 4.6.5 of this addendum before disposing of any of the standards.) The maximum accepted standard concentration-pathlength product (ASCPP) for each compound shall be higher than the maximum estimated concentration-pathlength products for both analytes and known interferants in the effluent gas. For each analyte, the minimum ASCPP shall be no greater than ten times the concentration-pathlength product of that analyte at its required detection limit.

4.6.4 Permanently store the background and interferograms in digitized form. Document details of the mathematical process for generating the spectra from these interferograms. Record the sample pressure (P_R), sample temperature (T_R), reference absorption pathlength (L_R), and interferogram signal integration period (t_{SR}). Signal integration periods for the background interferograms shall be $\geq t_{SR}$. Values of P_R, L_R, and t_{SR} shall not deviate by more than ± 1 percent from the time of recording {R1} to that of recording {R2}.

4.6.5 If self-prepared chemical standards are employed and spectra of only two concentrations are recorded for one or more compounds, verify the accuracy of the dilution technique by analyzing the prepared standards for those compounds with a secondary (non-FTIR) technique in accordance with sections 4.6.5.1 through 4.6.5.4 of this addendum.

4.6.5.1 Record the response of the secondary technique to each of the four standards prepared.

4.6.5.2 Perform a linear regression of the response values (dependant variable) versus the accepted standard concentration (ASC) values (independent variable), with the regression constrained to pass through the zero-response, zero ASC point.

4.6.5.3 Calculate the average fractional difference between the actual response values and the regression-predicted values (those calculated from the regression line using the four ASC values as the independent variable).

4.6.5.4 If the average fractional difference value calculated in section 4.6.5.3 of this addendum is larger for any compound than the corresponding AU_i, the dilution technique is not sufficiently accurate and the reference spectra prepared are not valid for the analysis.

4.7 *Select Analytical Regions.* Using the general considerations in section 7 of Reference A and the spectral characteristics of the analytes and interferants, select the analytical regions for the application. Label them m = 1 to M. Specify the lower, center and upper wavenumber positions of each analytical region (FL_m, FC_m, and FU_m, respectively). Specify the analytes and interferants which exhibit absorption in each region.

4.8 *Determine Fractional Reproducibility Uncertainties.* Using appendix E of this

addendum, calculate the fractional reproducibility uncertainty for each analyte (FRU_i) from a comparison of {R1} and {R2}. If $FRU_i > AU_i$ for any analyte, the reference spectra generated in accordance with section 4.6 of this addendum are not valid for the application.

4.9 *Identify Known Interferants.* Using appendix B of this addendum, determine which potential interferants affect the analyte concentration determinations. Relabel these potential interferant as "known" interferants, and designate these compounds from k = 1 to K. Appendix B to this addendum also provides criteria for determining whether the selected analytical regions are suitable.

4.10 *Prepare Computerized Analytical Programs*

4.10.1 Choose or devise mathematical techniques (e.g, classical least squares, inverse least squares, cross-correlation, and factor analysis) based on equation 4 of Reference A that are appropriate for analyzing spectral data by comparison with reference spectra.

4.10.2 Following the general recommendations of Reference A, prepare a computer program or set of programs that analyzes all of the analytes and known interferants, based on the selected analytical regions (section 4.7 of this addendum) and the prepared reference spectra (section 4.6 of this addendum). Specify the baseline correction technique (e.g., determining the slope and intercept of a linear baseline contribution in each analytical region) for each analytical region, including all relevant wavenumber positions.

4.10.3 Use programs that provide as output [at the reference absorption pathlength (L_R), reference gas temperature (T_R), and reference gas pressure (P_R)] the analyte concentrations, the known interferant concentrations, and the baseline slope and intercept values. If the sample absorption pathlength (L_S), sample gas temperature (T_S), or sample gas pressure (P_S) during the actual sample analyses differ from L_R, T_R, and P_R, use a program or set of programs that applies multiplicative corrections to the derived concentrations to account for these variations, and that provides as output both the corrected and uncorrected values. Include in the report of the analysis (see section 7.0 of this addendum) the details of any transformations applied to the original reference spectra (e.g., differentiation), in such a fashion that all analytical results may be verified by an independent agent from the reference spectra and data spectra alone.

4.11 *Determine the Fractional Calibration Uncertainty.* Calculate the fractional calibration uncertainty for each analyte ($FCUi$) according to appendix F of this addendum, and compare these values to the fractional uncertainty limits (AU_i; see section 4.1.2 of this addendum). If $FCU_i > AU_i$, either the reference spectra or analytical programs for that analyte are unsuitable.

4.12 *Verify System Configuration Suitability.* Using appendix C of this addendum, measure or obtain estimates of the noise level (RMS_{EST}, absorbance) of the FTIR system. Alternatively, construct the complete spectrometer system and determine the values RMS_{Sm} using appendix G of this

addendum. Estimate the minimum measurement uncertainty for each analyte (MAU_i, ppm) and known interferant (MIU_k, ppm) using appendix D of this addendum. Verify that (a) $MAU_i < (AU_i)(DL_i)$, $FRUi < AU_i$, and $FCUi < AU_i$ for each analyte and that (b) the CTS chosen meets the requirements listed in sections 4.5.1 through 4.5.5 of this addendum.

5.0 *Sampling and Analysis Procedure*

5.1 *Analysis System Assembly and Leak-Test.* Assemble the analysis system. Allow sufficient time for all system components to reach the desired temperature. Then, determine the leak-rate (L_R) and leak volume (V_L), where $V_L = L_R\, t_{SS}$. Leak volumes shall be ≤ 4 percent of V_{SS}.

5.2 *Verify Instrumental Performance.* Measure the noise level of the system in each analytical region using the procedure of appendix G of this addendum. If any noise level is higher than that estimated for the system in section 4.12 of this addendum, repeat the calculations of appendix D of this addendum and verify that the requirements of section 4.12 of this addendum are met; if they are not, adjust or repair the instrument and repeat this section.

5.3 *Determine the Sample Absorption Pathlength*

Record a background spectrum. Then, fill the absorption cell with CTS at the pressure P_R and record a set of CTS spectra {R3}. Store the background and unscaled CTS single beam interferograms and spectra. Using appendix H of this addendum, calculate the sample absorption pathlength (L_S) for each analytical region. The values L_S shall not differ from the approximated sample pathlength L_S' (see section 4.4 of this addendum) by more than 5 percent.

5.4 *Record Sample Spectrum.* Connect the sample line to the source. Either evacuate the absorption cell to an absolute pressure below 5 mmHg before extracting a sample from the effluent stream into the absorption cell, or pump at least ten cell volumes of sample through the cell before obtaining a sample. Record the sample pressure P_S. Generate the absorbance spectrum of the sample. Store the background and sample single beam interferograms, and document the process by which the absorbance spectra are generated from these data. (If necessary, apply the spectral transformations developed in section 5.6.2 of this addendum). The resulting sample spectrum is referred to below as S_S.

Note: Multiple sample spectra may be recorded according to the procedures of section 5.4 of this addendum before performing sections 5.5 and 5.6 of this addendum.

5.5 *Quantify Analyte Concentrations.* Calculate the unscaled analyte concentrations RUA_i and unscaled interferant concentrations RUI_K using the programs developed in section 4 of this addendum. To correct for pathlength and pressure variations between the reference and sample spectra, calculate the scaling factor, R_{LPS} using equation A.1,

$$R_{LPS} = \left(L_R P_R T_S\right) / \left(L_S P_S T_R\right) \quad \text{(A.1)}$$

Calculate the final analyte and interferant concentrations RSA$_i$ and RSI$_k$ using equations A.2 and A.3,

$$RSA_i = R_{LPS}RUA_i \quad (A.2)$$

$$RSI_k = R_{LPS}RUI_k \quad (A.3)$$

5.6 *Determine Fractional Analysis Uncertainty.* Fill the absorption cell with CTS at the pressure P$_S$. Record a set of CTS spectra {R4}. Store the background and CTS single beam interferograms. Using appendix H of this addendum, calculate the fractional analysis uncertainty (FAU) for each analytical region. If the FAU indicated for any analytical region is greater than the required accuracy requirements determined in sections 4.1.1 through 4.1.4 of this addendum, then comparisons to previously recorded reference spectra are invalid in that analytical region, and the analyst shall perform one or both of the procedures of sections 5.6.1 through 5.6.2 of this addendum.

5.6.1 Perform instrumental checks and adjust the instrument to restore its performance to acceptable levels. If adjustments are made, repeat sections 5.3, 5.4 (except for the recording of a sample spectrum), and 5.5 of this addendum to demonstrate that acceptable uncertainties are obtained in all analytical regions.

5.6.2 Apply appropriate mathematical transformations (e.g., frequency shifting, zero-filling, apodization, smoothing) to the spectra (or to the interferograms upon which the spectra are based) generated during the performance of the procedures of section 5.3 of this addendum. Document these transformations and their reproducibility. Do not apply multiplicative scaling of the spectra, or any set of transformations that is mathematically equivalent to multiplicative scaling. Different transformations may be applied to different analytical regions. Frequency shifts shall be less than one-half the minimum instrumental linewidth, and must be applied to all spectral data points in an analytical region. The mathematical transformations may be retained for the analysis if they are also applied to the appropriate analytical regions of all sample spectra recorded, and if all original sample spectra are digitally stored. Repeat sections 5.3, 5.4 (except the recording of a sample spectrum), and 5.5 of this addendum to demonstrate that these transformations lead to acceptable calculated concentration uncertainties in all analytical regions.

6.0 Post-Analysis Evaluations

Estimate the overall accuracy of the analyses performed in accordance with sections 5.1 through 5.6 of this addendum using the procedures of sections 6.1 through 6.3 of this addendum.

6.1 *Qualitatively Confirm the Assumed Matrix.* Examine each analytical region of the sample spectrum for spectral evidence of unexpected or unidentified interferants. If found, identify the interfering compounds (see Reference C for guidance) and add them to the list of known interferants. Repeat the procedures of section 4 of this addendum to

include the interferants in the uncertainty calculations and analysis procedures. Verify that the MAU and FCU values do not increase beyond acceptable levels for the application requirements. Re-calculate the analyte concentrations (section 5.5 of this addendum) in the affected analytical regions.

6.2 *Quantitatively Evaluate Fractional Model Uncertainty (FMU).* Perform the procedures of either section 6.2.1 or 6.2.2 of this addendum:

6.2.1 Using appendix I of this addendum, determine the fractional model error (FMU) for each analyte.

6.2.2 Provide statistically determined uncertainties FMU for each analyte which are equivalent to two standard deviations at the 95 percent confidence level. Such determinations, if employed, must be based on mathematical examinations of the pertinent sample spectra (not the reference spectra alone). Include in the report of the analysis (see section 7.0 of this addendum) a complete description of the determination of the concentration uncertainties.

6.3 *Estimate Overall Concentration Uncertainty (OCU).* Using appendix J of this addendum, determine the overall concentration uncertainty (OCU) for each analyte. If the OCU is larger than the required accuracy for any analyte, repeat sections 4 and 6 of this addendum.

7.0 *Reporting Requirements*

[Documentation pertaining to virtually all the procedures of sections 4, 5, and 6 will be required. Software copies of reference spectra and sample spectra will be retained for some minimum time following the actual testing.]

8.0 *References*

(A) Standard Practices for General Techniques of Infrared Quantitative Analysis (American Society for Testing and Materials, Designation E 168–88).

(B) The Coblentz Society Specifications for Evaluation of Research Quality Analytical Infrared Reference Spectra (Class II); Anal. Chemistry 47, 945A (1975); Appl. Spectroscopy 444, pp. 211–215, 1990.

(C) Standard Practices for General Techniques for Qualitative Infrared Analysis, American Society for Testing and Materials, Designation E 1252–88.

(D) ''EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards,'' U.S. Environmental Protection Agency Publication No. EPA/600/R–93/224, December 1993.

Appendix A to Addendum to Method 320—Definitions of Terms and Symbols

A.1 *Definitions of Terms.* All terms used in this method that are not defined below have the meaning given to them in the CAA and in subpart A of this part.

Absorption band means a contiguous wavenumber region of a spectrum (equivalently, a contiguous set of absorbance spectrum data points) in which the absorbance passes through a maximum or a series of maxima.

Absorption pathlength means the distance in a spectrophotometer, measured in the direction of propagation of the beam of radiant energy, between the surface of the specimen on which the radiant energy is

incident and the surface of the specimen from which it is emergent.

Analytical region means a contiguous wavenumber region (equivalently, a contiguous set of absorbance spectrum data points) used in the quantitative analysis for one or more analytes.

Note: The quantitative result for a single analyte may be based on data from more than one analytical region.

Apodization means modification of the ILS function by multiplying the interferogram by a weighing function whose magnitude varies with retardation.

Background spectrum means the single beam spectrum obtained with all system components without sample present.

Baseline means any line drawn on an absorption spectrum to establish a reference point that represents a function of the radiant power incident on a sample at a given wavelength.

Beers's law means the direct proportionality of the absorbance of a compound in a homogeneous sample to its concentration.

Calibration transfer standard (CTS) gas means a gas standard of a compound used to achieve and/or demonstrate suitable quantitative agreement between sample spectra and the reference spectra; see section 4.5.1 of this addendum.

Compound means a substance possessing a distinct, unique molecular structure.

Concentration (c) means the quantity of a compound contained in a unit quantity of sample. The unit ''ppm'' (number, or mole, basis) is recommended.

Concentration-pathlength product means the mathematical product of concentration of the species and absorption pathlength. For reference spectra, this is a known quantity; for sample spectra, it is the quantity directly determined from Beer's law. The units ''centimeters-ppm'' or ''meters-ppm'' are recommended.

Derivative absorption spectrum means a plot of rate of change of absorbance or of any function of absorbance with respect to wavelength or any function of wavelength.

Double beam spectrum means a transmission or absorbance spectrum derived by dividing the sample single beam spectrum by the background spectrum.

Note: The term ''double-beam'' is used elsewhere to denote a spectrum in which the sample and background interferograms are collected simultaneously along physically distinct absorption paths. Here, the term denotes a spectrum in which the sample and background interferograms are collected at different times along the same absorption path.

Fast Fourier transform (FFT) means a method of speeding up the computation of a discrete FT by factoring the data into sparse matrices containing mostly zeros.

Flyback means interferometer motion during which no data are recorded.

Fourier transform (FT) means the mathematical process for converting an amplitude-time spectrum to an amplitude-frequency spectrum, or vice versa.

Fourier transform infrared (FTIR) spectrometer means an analytical system that

employs a source of mid-infrared radiation, an interferometer, an enclosed sample cell of known absorption pathlength, an infrared detector, optical elements that transfer infrared radiation between components, and a computer system. The time-domain detector response (interferogram) is processed by a Fourier transform to yield a representation of the detector response vs. infrared frequency.

Note: When FTIR spectrometers are interfaced with other instruments, a slash should be used to denote the interface; e.g., GC/FTIR; HPCL/FTIR, and the use of FTIR should be explicit; i.e., FTIR not IR.

Frequency, v means the number of cycles per unit time.

Infrared means the portion of the electromagnetic spectrum containing wavelengths from approximately 0.78 to 800 microns.

Interferogram, I(σ) means record of the modulated component of the interference signal measured as a function of retardation by the detector.

Interferometer means device that divides a beam of radiant energy into two or more paths, generates an optical path difference between the beams, and recombines them in order to produce repetitive interference maxima and minima as the optical retardation is varied.

Linewidth means the full width at half maximum of an absorption band in units of wavenumbers (cm^{-1}).

Mid-infrared means the region of the electromagnetic spectrum from approximately 400 to 5000 cm^{-1}.

Reference spectra means absorption spectra of gases with known chemical compositions, recorded at a known absorption pathlength, which are used in the quantitative analysis of gas samples.

Retardation, σ means optical path difference between two beams in an interferometer; also known as "optical path difference" or "optical retardation."

Scan means digital representation of the detector output obtained during one complete motion of the interferometer's moving assembly or assemblies.

Scaling means application of a multiplicative factor to the absorbance values in a spectrum.

Single beam spectrum means Fourier-transformed interferogram, representing the detector response vs. wavenumber.

Note: The term "single-beam" is used elsewhere to denote any spectrum in which the sample and background interferograms are recorded on the same physical absorption path; such usage differentiates such spectra from those generated using interferograms recorded along two physically distinct absorption paths (see "double-beam spectrum" above). Here, the term applies (for example) to the two spectra used directly in the calculation of transmission and absorbance spectra of a sample.

Standard reference material means a reference material, the composition or properties of which are certified by a recognized standardizing agency or group.

Note: The equivalent ISO term is "certified reference material."

Transmittance, T means the ratio of radiant power transmitted by the sample to the radiant power incident on the sample. Estimated in FTIR spectroscopy by forming the ratio of the single-beam sample and background spectra.

Wavenumber, $\bar{v}$ means the number of waves per unit length.

Note: The usual unit of wavenumber is the reciprocal centimeter, cm^{-1}. The wavenumber is the reciprocal of the wavelength, λ, when λ is expressed in centimeters.

Zero-filling means the addition of zero-valued points to the end of a measured interferogram.

Note: Performing the FT of a zero-filled interferogram results in correctly interpolated points in the computed spectrum.

A.2 *Definitions of Mathematical Symbols.* The symbols used in equations in this protocol are defined as follows:

(1) A, absorbance = the logarithm to the base 10 of the reciprocal of the transmittance (T).

$$ A = \log_{10}\left(\frac{1}{T}\right) = -\log_{10} T $$

(2) AAI_{im} = band area of the ith analyte in the mth analytical region, at the concentration (CL_i) corresponding to the product of its required detection limit (DL_i) and analytical uncertainty limit (AU_i).

(3) AAV_{im} = average absorbance of the ith analyte in the mth analytical region, at the concentration (CL_i) corresponding to the product of its required detection limit (DL_i) and analytical uncertainty limit (AU_i).

(4) ASC, accepted standard concentration = the concentration value assigned to a chemical standard.

(5) ASCPP, accepted standard concentration-pathlength product = for a chemical standard, the product of the ASC and the sample absorption pathlength. The units "centimeters-ppm" or "meters-ppm" are recommended.

(6) AU_i, analytical uncertainty limit = the maximum permissible fractional uncertainty of analysis for the ith analyte concentration, expressed as a fraction of the analyte concentration determined in the analysis.

(7) AVT_m = average estimated total absorbance in the mth analytical region.

(8) $CKWN_k$ = estimated concentration of the kth known interferant.

(9) $CMAX_i$ = estimated maximum concentration of the ith analyte.

(10) $CPOT_j$ = estimated concentration of the jth potential interferant.

(11) DL_i, required detection limit = for the ith analyte, the lowest concentration of the analyte for which its overall fractional uncertainty (OFU_i) is required to be less than the analytical uncertainty limit (AU_i).

(12) FC_m = center wavenumber position of the mth analytical region.

(13) FAU_i, fractional analytical uncertainty = calculated uncertainty in the measured concentration of the ith analyte because of errors in the mathematical comparison of reference and sample spectra.

(14) FCU_i, fractional calibration uncertainty = calculated uncertainty in the measured concentration of the ith analyte because of errors in Beer's law modeling of the reference spectra concentrations.

(15) FFL_m = lower wavenumber position of the CTS absorption band associated with the mth analytical region.

(16) FFU_m = upper wavenumber position of the CTS absorption band associated with the mth analytical region.

(17) FL_m = lower wavenumber position of the mth analytical region.

(18) FMU_i, fractional model uncertainty = calculated uncertainty in the measured concentration of the ith analyte because of errors in the absorption model employed.

(19) FN_L = lower wavenumber position of the CTS spectrum containing an absorption band at least as narrow as the analyte absorption bands.

(20) FN_U = upper wavenumber position of the CTS spectrum containing an absorption band at least as narrow as the analyte absorption bands.

(21) FRU_i, fractional reproducibility uncertainty = calculated uncertainty in the measured concentration of the ith analyte because of errors in the reproducibility of spectra from the FTIR system.

(22) FU_m = upper wavenumber position of the mth analytical region.

(23) IAI_{jm} = band area of the jth potential interferant in the mth analytical region, at its expected concentration ($CPOT_j$).

(24) IAV_{im} = average absorbance of the ith analyte in the mth analytical region, at its expected concentration ($CPOT_j$).

(25) $ISC_{i \text{ or } k}$, indicated standard concentration = the concentration from the computerized analytical program for a single-compound reference spectrum for the ith analyte or k^{th} known interferant.

(26) kPa = kilo-Pascal (see Pascal).

(27) L_S' = estimated sample absorption pathlength.

(28) L_R = reference absorption pathlength.

(29) L_S = actual sample absorption pathlength.

(30) MAU_i = mean of the MAU_{im} over the appropriate analytical regions.

(31) MAU_{im}, minimum analyte uncertainty = the calculated minimum concentration for which the analytical uncertainty limit (AU_i) in the measurement of the ith analyte, based on spectral data in the mth analytical region, can be maintained.

(32) MIU_j = mean of the MIU_{jm} over the appropriate analytical regions.

(33) MIU_{jm}, minimum interferant uncertainty = the calculated minimum concentration for which the analytical uncertainty limit $CPOT_j/20$ in the measurement of the jth interferant, based on spectral data in the mth analytical region, can be maintained.

(34) MIL, minimum instrumental linewidth = the minimum linewidth from the FTIR system, in wavenumbers.

Note: The MIL of a system may be determined by observing an absorption band known (through higher resolution examinations) to be narrower than indicated by the system. The MIL is fundamentally limited by the retardation of the interferometer, but is also affected by other operational parameters (e.g., the choice of apodization).

(35) N_i = number of analytes.

(36) N_j = number of potential interferants.

(37) N_k = number of known interferants.

(38) N_{scan} = the number of scans averaged to obtain an interferogram.

(39) OFU_i = the overall fractional uncertainty in an analyte concentration determined in the analysis (OFU_i = $MAX\{FRU_i, FCU_i, FAU_i, FMU_i\}$).

(40) Pascal (Pa) = metric unit of static pressure, equal to one Newton per square meter; one atmosphere is equal to 101,325 Pa; 1/760 atmosphere (one Torr, or one millimeter Hg) is equal to 133.322 Pa.

(41) P_{min} = minimum pressure of the sampling system during the sampling procedure.

(42) P_S' = estimated sample pressure.

(43) P_R = reference pressure.

(44) P_S = actual sample pressure.

(45) RMS_{Sm} = measured noise level of the FTIR system in the mth analytical region.

(46) RMSD, root mean square difference = a measure of accuracy determined by the following equation:

$$RMSD = \sqrt{\left(\frac{1}{n}\right)\sum_{i=1}^{n} e_i^2}$$

Where:

n = the number of observations for which the accuracy is determined.

e_i = the difference between a measured value of a property and its mean value over the n observations.

Note: The RMSD value ''between a set of n contiguous absorbance values (A_i) and the mean of the values'' (A_M) is defined as

$$RMSD = \sqrt{\left(\frac{1}{n}\right)\sum_{i-1}^{n}\left(A_i - A_M\right)^2}$$

(47) RSA_i = the (calculated) final concentration of the ith analyte.

(48) RSI_k = the (calculated) final concentration of the kth known interferant.

(49) t_{scan}, scan time = time used to acquire a single scan, not including flyback.

(50) t_S, signal integration period = the period of time over which an interferogram is averaged by addition and scaling of individual scans. In terms of the number of scans N_{scan} and scan time t_{scan}, $t_S = N_{scan}t_{scan}$.

(51) t_{SR} = signal integration period used in recording reference spectra.

(52) t_{SS} = signal integration period used in recording sample spectra.

(53) T_R = absolute temperature of gases used in recording reference spectra.

(54) T_S = absolute temperature of sample gas as sample spectra are recorded.

(55) TP, Throughput = manufacturer's estimate of the fraction of the total infrared power transmitted by the absorption cell and transfer optics from the interferometer to the detector.

(56) V_{SS} = volume of the infrared absorption cell, including parts of attached tubing.

(57) W_{ik} = weight used to average over analytical regions k for quantities related to the analyte i; see appendix D of this addendum.

Appendix B to Addendum to Method 320— Identifying Spectral Interferants

B.1 *General*

B.1.1 Assume a fixed absorption pathlength equal to the value L_S'.

B.1.2 Use band area calculations to compare the relative absorption strengths of the analytes and potential interferants. In the mth analytical region (FL_m to FU_m), use either rectangular or trapezoidal approximations to determine the band areas described below (see Reference A, sections A.3.1 through A.3.3). Document any baseline corrections applied to the spectra.

B.1.3 Use the average total absorbance of the analytes and potential interferants in each analytical region to determine whether the analytical region is suitable for analyte concentration determinations.

Note: The average absorbance in an analytical region is the band area divided by the width of the analytical region in wavenumbers. The average total absorbance in an analytical region is the sum of the average absorbances of all analytes and potential interferants.

B.2 *Calculations*

B.2.1 Prepare spectral representations of each analyte at the concentration CL_i = $(DL_i)(AU_i)$, where DL_i is the required detection limit and AU_i is the maximum permissible analytical uncertainty. For the mth analytical region, calculate the band area (AAI_{im}) and average absorbance (AAV_{im}) from these scaled analyte spectra.

B.2.2 Prepare spectral representations of each potential interferant at its expected concentration ($CPOT_j$). For the mth analytical region, calculate the band area (IAI_{jm}) and average absorbance (IAV_{jm}) from these scaled potential interferant spectra.

B.2.3 Repeat the calculation for each analytical region, and record the band area results in matrix form as indicated in Figure B.1.

B.2.4 If the band area of any potential interferant in an analytical region is greater than the one-half the band area of any analyte (i.e., $IAI_{jm} > 0.5 \, AAI_{im}$ for any pair ij and any m), classify the potential interferant as a known interferant. Label the known interferants k = 1 to K. Record the results in matrix form as indicated in Figure B.2.

B.2.5 Calculate the average total absorbance (AVT_m) for each analytical region and record the values in the last row of the matrix described in Figure B.2. Any analytical region where $AVT_m > 2.0$ is unsuitable.

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FIGURE B.1 Presentation of Potential Interferant Calculations.

Analytical Regions
1 . . . . M

Analyte Labels

1	AAI_{11} . . . AAI_{1M}
.	. .
.	. .
I	AAI_{I1} . . . AAI_{IM}

Potential Interferant Labels

1	IAI_{11} . . . IAI_{1M}
.	. .
.	. .
J	IAI_{J1} . . . IAI_{JM}

FIGURE B.2 Presentation of Known Interferant Calculations

Analytical Regions
1 . . . . M

Analyte Labels

1	AAI_{11} . . . . AAI_{1M}
.	. .
.	. .
I	AAI_{I1} . . . . AAI_{IM}

Known Interferant Labels

1	IAI_{11} . . . . IAI_{1M}
.	. .
.	. .
K	IAI_{K1} . . . . IAI_{KM}

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Total Average Absorbance	AVT_1	AVT_M

Appendix C to Addendum to Method 320— Estimating Noise Levels

C.1 *General*

C.1.1 The root-mean-square (RMS) noise level is the standard measure of noise in this addendum. The RMS noise level of a contiguous segment of a spectrum is defined as the RMS difference (RMSD) between the absorbance values which form the segment and the mean value of that segment (see appendix A of this addendum).

C.1.2 The RMS noise value in double-beam absorbance spectra is assumed to be inversely proportional to: (a) the square root of the signal integration period of the sample single beam spectra from which it is formed,

and (b) the total infrared power transmitted through the interferometer and absorption cell.

C.1.3 Practically, the assumption of C.1.2 allows the RMS noise level of a complete system to be estimated from the quantities described in sections C.1.3.1 through C.1.3.4:

C.1.3.1 RMS_{MAN}, the noise level of the system (in absorbance units), without the absorption cell and transfer optics, *under those conditions necessary to yield the specified minimum instrumental linewidth,* e.g., Jacquinot stop size.

C.1.3.2 t_{MAN}, the manufacturer's signal integration time used to determine RMS_{MAN}.

C.1.3.3 t_{SS}, the signal integration time for the analyses.

C.1.3.4 TP, the manufacturer's estimate of the fraction of the total infrared power transmitted by the absorption cell and transfer optics from the interferometer to the detector.

C.2 *Calculations*

C.2.1 Obtain the values of RMS_{MAN}, t_{MAN}, and TP from the manufacturers of the equipment, or determine the noise level by direct measurements with the completely constructed system proposed in section 4 of this addendum.

C.2.2 Calculate the noise value of the system (RMS_{EST}) using equation C.1.

$$RMS_{EST} = RMS_{MAN} \ TP \sqrt{\frac{t_{ss}}{t_{MAN}}} \qquad (C.1)$$

Appendix D to Addendum to Method 320— Estimating Minimum Concentration Measurement Uncertainties (MAU and MIU)

D.1 *General*

Estimate the minimum concentration measurement uncertainties for the ith analyte (MAU_i) and j^{th} interferant (MIU_j) based on the spectral data in the mth analytical region by comparing the analyte band area in the analytical region (AAI_{im}) and estimating or

measuring the noise level of the system (RMS_{EST} or RMS_{SM}).

Note: For a single analytical region, the MAU or MIU value is the concentration of the analyte or interferant for which the band area is equal to the product of the analytical region width (in wavenumbers) and the noise level of the system (in absorbance units). If data from more than one analytical region are used in the determination of an analyte concentration, the MAU or MIU is the mean

of the separate MAU or MIU values calculated for each analytical region.

D.2 *Calculations*

D.2.1 For each analytical region, set $RMS = RMS_{SM}$ if measured (appendix G of this addendum), or set $RMS = RMS_{EST}$ if estimated (appendix C of this addendum).

D.2.2 For each analyte associated with the analytical region, calculate MAU_{im} using equation D.1,

$$MAU_{im} = (RMS) \left(DL_i\right) \left(AU_i\right) \frac{\left(FU_m - FL_m\right)}{AAI_{im}} \qquad (D.1)$$

D.2.3 If only the mth analytical region is used to calculate the concentration of the ith analyte, set $MAU_i = MAU_{im}$.

D.2.4 If more than one analytical region is used to calculate the concentration of the ith analyte, set MAU_i equal to the weighted mean of the appropriate MAU_{im} values calculated above; the weight for each term in

the mean is equal to the fraction of the total wavenumber range used for the calculation represented by each analytical region. Mathematically, if the set of analytical regions employed is $\{m'\}$, then the MAU for each analytical region is given by equation D.2.

$$MAU_i = \sum_{k \in \{m'\}} W_k MAU_{ik} \qquad (D.2)$$

where the weight W_{ik} is defined for each term in the sum as

$$W_{ik} = \left(FM_k - FL_k\right) \left(\sum_{p \in \{m'\}} \left[FM_p - FL_p \right] \right)^{-1} \qquad (D.3)$$

D.2.5 Repeat sections D.2.1 through D.2.4 of this appendix to calculate the analogous values MIU_j for the interferants $j = 1$ to J. Replace the value (AU_i) (DL_i) in equation D.1 with $CPOT_j/20$; replace the value AAI_{im} in equation D.1 with IAI_{jm}.

Appendix E to Addendum to Method 320— Determining Fractional Reproducibility Uncertainties (FRU)

E.1 *General*

To estimate the reproducibility of the spectroscopic results of the system, compare the CTS spectra recorded before and after preparing the reference spectra. Compare the difference between the spectra to their average band area. Perform the calculation

for each analytical region on the portions of the CTS spectra associated with that analytical region.

E.2 *Calculations*

E.2.1 The CTS spectra $\{R1\}$ consist of N spectra, denoted by S_{1i}, i=1, N. Similarly, the CTS spectra $\{R2\}$ consist of N spectra, denoted by S_{2i}, i=1, N. Each S_{ki} is the spectrum of a single compound, where i denotes the compound and k denotes the set $\{Rk\}$ of which S_{ki} is a member. Form the spectra S_3 according to $S_{3i} = S_{2i} - S_{1i}$ for each i. Form the spectra S_4 according to $S_{4i} = [S_{2i}+S_{1i}]/2$ for each i.

E.2.2 Each analytical region m is associated with a portion of the CTS spectra

S_{2i} and S_{1i}, for a particular i, with lower and upper wavenumber limits FFL_m and FFU_m, respectively.

E.2.3 For each m and the associated i, calculate the band area of S_{4i} in the wavenumber range FFU_m to FFL_m. Follow the guidelines of section B.1.2 of this addendum for this band area calculation. Denote the result by BAV_m.

E.2.4 For each m and the associated i, calculate the RMSD of S_{3i} between the absorbance values and their mean in the wavenumber range FFU_m to FFL_m. Denote the result by $SRMS_m$.

E.2.5 For each analytical region m, calculate FM_m using equation E.1,

$$FM_m = SRMS_m\left(FFU_m - FFL_m\right)/BAV_m \qquad (E.1)$$

E.2.6 If only the mth analytical region is used to calculate the concentration of the ith analyte, set $FRU_i = FM_m$.

E.2.7 If a number p_i of analytical regions are used to calculate the concentration of the ith analyte, set FRU_i equal to the weighted mean of the appropriate FM_m values calculated according to section E.2.5. Mathematically, if the set of analytical regions employed is $\{m'\}$, then FRU_i is given by equation E.2,

$$FRU_i = \sum_{k\in\{m'\}} W_{ik}FM_k \qquad (E.2)$$

where the W_{ik} are calculated as described in appendix D of this addendum.

Appendix F of Addendum to Method 320—Determining Fractional Calibration Uncertainties (FCU)

F.1 General

F.1.1 The concentrations yielded by the computerized analytical program applied to each single-compound reference spectrum are defined as the indicated standard concentrations (ISC's). The ISC values for a single compound spectrum should ideally equal the accepted standard concentration (ASC) for one analyte or interferant, and should ideally be zero for all other compounds. Variations from these results are caused by errors in the ASC values, variations from the Beer's law (or modified Beer's law) model used to determine the concentrations, and noise in the spectra. When the first two effects dominate, the systematic nature of the errors is often apparent and the analyst shall take steps to correct them.

F.1.2 When the calibration error appears non-systematic, apply the procedures of sections F.2.1 through F.2.3 of this appendix to estimate the fractional calibration uncertainty (FCU) for each compound. The FCU is defined as the mean fractional error between the ASC and the ISC for all reference spectra with non-zero ASC for that compound. The FCU for each compound shall be less than the required fractional uncertainty specified in section 4.1 of this addendum.

F.1.3 The computerized analytical programs shall also be required to yield acceptably low concentrations for compounds with ISC = 0 when applied to the reference spectra. The ISC of each reference spectrum for each analyte or interferant shall not exceed that compound's minimum measurement uncertainty (MAU or MIU).

F.2 Calculations

F.2.1 Apply each analytical program to each reference spectrum. Prepare a similar table to that in Figure F.1 to present the ISC and ASC values for each analyte and interferant in each reference spectrum. Maintain the order of reference file names and compounds employed in preparing Figure F.1.

F.2.2 For all reference spectra in Figure F.1, verify that the absolute values of the ISC's are less than the compound's MAU (for analytes) or MIU (for interferants).

F.2.3 For each analyte reference spectrum, calculate the quantity (ASC–ISC)/ASC. For each analyte, calculate the mean of these values (the FCU_i for the ith analyte) over all reference spectra. Prepare a similar table to that in Figure F.2 to present the FCU_i and analytical uncertainty limit (AU_i) for each analyte.

FIGURE F.1.—PRESENTATION OF ACCEPTED STANDARD CONCENTRATIONS (ASC'S) AND INDICATED STANDARD CONCENTRATIONS (ISC'S)

Compound name	Reference spectrum file name	ASC (ppm)	ISC (ppm)					
			Analytes i=1 j=1			Interferants I J		

FIGURE F.2—PRESENTATION OF FRACTIONAL CALIBRATION UNCERTAINTIES (FCU'S) AND ANALYTICAL UNCERTAINTIES (AU'S)

Analyte name	FCU (%)	AU (%)

Appendix G to Addendum to Method 320—Measuring Noise Levels

G.1 General

The root-mean-square (RMS) noise level is the standard measure of noise. The RMS noise level of a contiguous segment of a spectrum is the RMSD between the absorbance values that form the segment and the mean value of the segment (see appendix A of this addendum).

G.2 Calculations

G.2.1 Evacuate the absorption cell or fill it with UPC grade nitrogen at approximately one atmosphere total pressure.

G.2.2 Record two single beam spectra of signal integration period t_{SS}.

G.2.3 Form the double beam absorption spectrum from these two single beam spectra, and calculate the noise level RMS_{Sm} in the M analytical regions.

Appendix H of Addendum to Method 320—Determining Sample Absorption Pathlength (L_S) and Fractional Analytical Uncertainty (FAU)

H.1 General

Reference spectra recorded at absorption pathlength (L_R), gas pressure (P_R), and gas absolute temperature (T_R) may be used to determine analyte concentrations in samples whose spectra are recorded at conditions

different from that of the reference spectra, i.e., at absorption pathlength (L_S), absolute temperature (T_S), and pressure (P_S). This appendix describes the calculations for estimating the fractional uncertainty (FAU) of this practice. It also describes the calculations for determining the sample absorption pathlength from comparison of CTS spectra, and for preparing spectra for further instrumental and procedural checks.

H.1.1   Before sampling, determine the sample absorption pathlength using least squares analysis. Determine the ratio L_S/L_R by comparing the spectral sets {R1} and {R3}, which are recorded using the same CTS at L_S and L_R, and T_S and T_R, but both at P_R.

H.1.2   Determine the fractional analysis uncertainty (FAU) for each analyte by comparing a scaled CTS spectral set,

recorded at L_S, T_S, and P_S, to the CTS reference spectra of the same gas, recorded at L_R, T_R, and P_R. Perform the quantitative comparison after recording the sample spectra, based on band areas of the spectra in the CTS absorbance band associated with each analyte.

H.2   *Calculations*

H.2.1   *Absorption Pathlength Determination.* Perform and document separate linear baseline corrections to each analytical region in the spectral sets {R1} and {R3}. Form a one-dimensional array A_R containing the absorbance values from all segments of {R1} that are associated with the analytical regions; the members of the array are A_{Ri}, i = 1, n. Form a similar one-dimensional array A_S from the absorbance

values in the spectral set {R3}; the members of the array are A_{Si}, i = 1, n. Based on the model $A_S = rA_R + E$, determine the least-squares estimate of r′, the value of r which minimizes the square error E^2. Calculate the sample absorption pathlength, L_S, using equation H.1,

$$L_s = r'\left(T_s/T_R\right)L_R \qquad (H.1)$$

H.2.2   *Fractional Analysis Uncertainty.* Perform and document separate linear baseline corrections to each analytical region in the spectral sets {R1} and {R4}. Form the arrays A_S and A_R as described in section H.2.1 of this appendix, using values from {R1} to form A_R, and values from {R4} to form A_S. Calculate $NRMS_E$ and IA_{AV} using equations H.2 and H.3,

$$NRMS_E = \sqrt{\sum_{i=1}^{n}\left[A_{Si} - \left(\frac{T_R}{T_S}\right)\left(\frac{L_S}{L_R}\right)\left(\frac{P_S}{P_R}\right)A_{Ri}\right]} \qquad (H.2)$$

$$IA_{AV} = \frac{1}{2}\sum_{i=1}^{n}\left[A_{Si} + \left(\frac{T_R}{T_S}\right)\left(\frac{L_S}{L_R}\right)\left(\frac{P_S}{P_R}\right)A_{Ri}\right] \qquad (H.3)$$

The fractional analytical uncertainty, FAU, is given by equation H.4,

$$FAU = \frac{NRMS_E}{IA_{AV}} \qquad (H.4)$$

Appendix I to Addendum to Method 320—Determining Fractional Model Uncertainties (FMU)

I.1   *General*

To prepare analytical programs for FTIR analyses, the sample constituents must first be assumed. The calculations in this appendix, based upon a simulation of the sample spectrum, shall be used to verify the appropriateness of these assumptions. The simulated spectra consist of the sum of single compound reference spectra scaled to represent their contributions to the sample absorbance spectrum; scaling factors are based on the indicated standard concentrations (ISC) and measured (sample) analyte and interferant concentrations, the sample and reference absorption pathlengths, and the sample and reference gas pressures. No band-shape correction for differences in

the temperature of the sample and reference spectra gases is made; such errors are included in the FMU estimate. The actual and simulated sample spectra are quantitatively compared to determine the fractional model uncertainty; this comparison uses the reference spectra band areas and residuals in the difference spectrum formed from the actual and simulated sample spectra.

I.2   *Calculations*

I.2.1   For each analyte (with scaled concentration RSA_i), select a reference spectrum SA_i with indicated standard concentration ISC_i. Calculate the scaling factors, RA_i, using equation I.1,

$$RA_i = \frac{T_R\ L_S\ P_S\ RSA_i}{T_S\ L_R\ P_R\ ISC_i} \qquad (I.1)$$

Form the spectra SAC_i by scaling each SA_i by the factor RA_i.

I.2.2   For each interferant, select a reference spectrum SI_k with indicated standard concentration ISC_k. Calculate the scaling factors, RI_k, using equation I.2,

$$RI_k = \frac{T_R\ L_S\ P_S\ RSI_k}{T_S\ L_R\ P_R\ ISC_k} \qquad (I.2)$$

Form the spectra SIC_k by scaling each SI_k by the factor RI_k.

I.2.3   For each analytical region, determine by visual inspection which of the spectra SAC_i and SIC_k exhibit absorbance bands within the analytical region. Subtract each spectrum SAC_i and SIC_k exhibiting absorbance from the sample spectrum S_S to form the spectrum SUB_S. To save analysis time and to avoid the introduction of unwanted noise into the subtracted spectrum, it is recommended that the calculation be made (1) only for those spectral data points within the analytical regions, and (2) for each analytical region separately using the original spectrum S_S.

I.2.4   For each analytical region m, calculate the RMSD of SUB_S between the absorbance values and their mean in the region FFU_m to FFL_m. Denote the result by $RMSS_m$.

I.2.5   For each analyte i, calculate FM_m, using equation I.3,

$$FM_m = \frac{RMSS_m\left(FFU_m - FFL_m\right)AU_i DL_i}{AAI_i\ RSA_i} \qquad (I.3)$$

for each analytical region associated with the analyte.

I.2.6   If only the mth analytical region is used to calculate the concentration of the ith analyte, set $FMU_i = FM_m$.

I.2.7   If a number of analytical regions are used to calculate the concentration of the ith analyte, set FM_i equal to the weighted mean

of the appropriate FM_m values calculated using equation I–3. Mathematically, if the set of analytical regions employed is {m′}, then the fractional model uncertainty, FMU, is given by equation I.4,

$$FMU_i = \sum_{k\in\{m'\}} W_{ik} FM_k \qquad (I.4)$$

where W_{ik} is calculated as described in appendix D of this addendum.

Appendix J of Addendum to Method 320— Determining Overall Concentration Uncertainties (OCU)

The calculations in this addendum estimate the measurement uncertainties for various FTIR measurements. The lowest possible overall concentration uncertainty (OCU) for an analyte is its MAU value, which is an estimate of the absolute concentration uncertainty when spectral noise dominates the measurement error. However, if the product of the largest fractional concentration uncertainty (FRU, FCU, FAU, or FMU) and the measured concentration of an analyte exceeds the MAU for the analyte, then the OCU is this product. In mathematical terms, set $OFU_i = MAX\{FRU_i, FCU_i, FAU_i, FMU_i\}$ and $OCU_i = MAX\{RSA_i*OFU_i, MAU_i\}$.

Test Method 321—Measurement of Gaseous Hydrogen Chloride Emissions At Portland Cement Kilns by Fourier Transform Infrared (FTIR) Spectroscopy

1.0 Introduction

This method should be performed by those persons familiar with the operation of Fourier Transform Infrared (FTIR) instrumentation in the application to source sampling. This document describes the sampling procedures for use in the application of FTIR spectrometry for the determination of vapor phase hydrogen chloride (HCl) concentrations both before and after particulate matter control devices installed at portland cement kilns. A procedure for analyte spiking is included for quality assurance. This method is considered to be self validating provided that the requirements listed in section 9 of this method are followed. The analytical procedures for interpreting infrared spectra from emission measurements are described in the "Protocol For The Use of Extractive Fourier Transform Infrared (FTIR) Spectrometry in Analyses of Gaseous Emissions From Stationary Industrial Sources", included as an addendum to proposed Method 320 of this appendix (hereafter referred to as the "FTIR Protocol)". References 1 and 2 describe the use of FTIR spectrometry in field measurements. Sample transport presents the principal difficulty in directly measuring HCl emissions. This identical problem must be overcome by any extractive measurement method. HCl is reactive and water soluble. The sampling system must be adequately designed to prevent sample condensation in the system.

1.1 Scope and Application

This method is specifically designed for the application of FTIR Spectrometry in extractive measurements of gaseous HCl concentrations in portland cement kiln emissions.

1.2 Applicability

This method applies to the measurement of HCl [CAS No. 7647–01–0]. This method can be applied to the determination of HCl concentrations both before and after particulate matter control devices installed at portland cement manufacturing facilities. This method applies to either continuous flow through measurement (with isolated sample analysis) or grab sampling (batch analysis). HCl is measured using the mid-infrared spectral region for analysis (about 400 to 4000 cm^{-1} or 25 to 2.5 μm). Table 1 lists the suggested analytical region for quantification of HCl taking the interference from water vapor into consideration.

TABLE 1.—EXAMPLE ANALYTICAL REGION FOR HCL

Compound	Analytical region (cm^{-1})	Potential interferants
Hydrogen chloride	2679–2840	Water.

1.3 Method Range and Sensitivity

1.3.1 The analytical range is determined by the instrumental design and the composition of the gas stream. For practical purposes there is no upper limit to the range because the pathlength may be reduced or the sample may be diluted. The lower detection range depends on (1) the absorption coefficient of the compound in the analytical frequency region, (2) the spectral resolution, (3) the interferometer sampling time, (4) the detector sensitivity and response, and (5) the absorption pathlength.

1.3.2 The practical lower quantification range is usually higher than the instrument sensitivity allows and is dependent upon (1) the presence of interfering species in the exhaust gas including H_2O, CO_2, and SO_2, (2) analyte losses in the sampling system, (3) the optical alignment of the gas cell and transfer optics, and (4) the quality of the reflective surfaces in the cell (cell throughput). Under typical test conditions (moisture content of up to 30% and CO_2 concentrations from 1 to 15 percent), a 22 meter path length cell with a suitable sampling system may achieve a lower quantification range of from 1 to 5 ppm for HCl.

1.4 Data Quality Objectives

1.4.1 In designing or configuring the analytical system, data quality is determined by measuring of the root mean square deviation (RMSD) of the absorbance values within a chosen spectral (analytical) region. The RMSD provides an indication of the signal-to-noise ratio (S/N) of the spectral baseline. Appendix D of the FTIR Protocol (the addendum to Method 320 of this appendix) presents a discussion of the relationship between the RMSD, lower detection limit, DL_i, and analytical uncertainty, AU_i. It is important to consider the target analyte quantification limit when performing testing with FTIR instrumentation, and to optimize the system to achieve the desired detection limit.

1.4.2 Data quality is determined by measuring the root mean square (RMS) noise level in each analytical spectral region (appendix C of the FTIR Protocol). The RMS noise is defined as the root mean square deviation (RMSD) of the absorbance values in an analytical region from the mean absorbance value in the same region. Appendix D of the *FTIR Protocol* defines the minimum analyte uncertainty (MAU), and how the RMSD is used to calculate the MAU. The MAU_{im} is the minimum concentration of the ith analyte in the mth analytical region for which the analytical uncertainty limit can be maintained. Table 2 presents example values of AU and MAU using the analytical region presented in Table 1.

TABLE 2.—EXAMPLE PRE-TEST PRO-TOCOL CALCULATIONS FOR HYDRO-GEN CHLORIDE

	HCl
Reference concentration (ppm-meters)/K	11.2
Reference Band area	2.881

TABLE 2.—EXAMPLE PRE-TEST PRO-TOCOL CALCULATIONS FOR HYDRO-GEN CHLORIDE—Continued

	HCl
DL (ppm-meters)/K	0.1117
AU	0.2
CL (DL × AU)	0.02234
FL (cm^{-1})	2679.83
FU (cm^{-1})	2840.93
FC (cm^{-1})	2760.38
AAI (ppm-meters)/K	0.06435
RMSD	2.28E–03
MAU (ppm-meters)/K	1.28E–01
MAU ppm at 22 meters and 250 °F	.0.2284

2.0 Summary of Method

2.1 Principle

See Method 320 of this appendix. HCl can also undergo rotation transitions by absorbing energy in the far-infrared spectral region. The rotational transitions are superimposed on the vibrational fundamental to give a series of lines centered at the fundamental vibrational frequency, 2885 cm-1. The frequencies of absorbance and the pattern of rotational/vibrational lines are unique to HCl. When this distinct pattern is observed in an infrared spectrum of an unknown sample, it unequivocally identifies HCl as a component of the mixture. The infrared spectrum of HCl is very distinctive and cannot be confused with the spectrum of any other compound. See Reference 6.

2.2 *Sampling and Analysis.* See Method 320 of this appendix.

2.3 *Operator Requirements.* The analyst must have knowledge of spectral patterns to choose an appropriate absorption path length or determine if sample dilution is necessary. The analyst should also understand FTIR instrument operation well enough to choose instrument settings that are consistent with the objectives of the analysis.

3.0 *Definitions*

See appendix A of the FTIR Protocol.

4.0 *Interferences*

This method will not measure HCl under conditions: (1) where the sample gas stream can condense in the sampling system or the instrumentation, or (2) where a high moisture content sample relative to the analyte concentrations imparts spectral interference due to the water vapor absorbance bands. For measuring HCl the first (sampling) consideration is more critical. Spectral interference from water vapor is not a significant problem except at very high moisture levels and low HCl concentrations.

4.1 *Analytical Interferences.* See Method 320 of this appendix.

4.1.1 *Background Interferences.* See Method 320 of this appendix.

4.1.2 *Spectral interferences.* Water vapor can present spectral interference for FTIR gas analysis of HCl. Therefore, the water vapor in the spectra of kiln gas samples must be accounted for. This means preparing at least one spectrum of a water vapor sample where the moisture concentration is close to that in the kiln gas.

4.2 *Sampling System Interferences.* The principal sampling system interferant for measuring HCl is water vapor. Steps must be taken to ensure that no condensation forms anywhere in the probe assembly, sample lines, or analytical instrumentation. Cold spots anywhere in the sampling system must be avoided. The extent of sampling system bias in the FTIR analysis of HCl depends on concentrations of potential interferants, moisture content of the gas stream, temperature of the gas stream, temperature of sampling system components, sample flow rate, and reactivity of HCl with other species in the gas stream (e.g., ammonia). For measuring HCl in a wet gas stream the temperatures of the gas stream, sampling components, and the sample flow rate are of primary importance. Analyte spiking with HCl is performed to demonstrate the integrity of the sampling system for transporting HCl vapor in the flue gas to the FTIR instrument. See section 9 of this method for a complete description of analyte spiking.

5.0 *Safety*

5.1 Hydrogen chloride vapor is corrosive and can cause irritation or severe damage to respiratory system, eyes and skin. Exposure to this compound should be avoided.

5.2 This method may involve sampling at locations having high positive or negative pressures, or high concentrations of hazardous or toxic pollutants, and can not address all safety problems encountered under these diverse sampling conditions. It is the responsibility of the tester(s) to ensure proper safety and health practices, and to

determine the applicability of regulatory limitations before performing this test method. Leak-check procedures are outlined in section 8.2 of Method 320 of this appendix.

6.0 *Equipment and Supplies*

Note: Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

6.1 *FTIR Spectrometer and Detector.* An FTIR Spectrometer system (interferometer, transfer optics, gas cell and detector) having the capability of measuring HCl to the predetermined minimum detectable level required (see section 4.1.3 of the FTIR Protocol). The system must also include an accurate means to control and/or measure the temperature of the FTIR gas analysis cell, and a personal computer with compatible software that provides real-time updates of the spectral profile during sample and spectral collection.

6.2 *Pump.* Capable of evacuating the FTIR cell volume to 1 Torr (133.3 Pascals) within two minutes (for batch sample analysis).

6.3 *Mass Flow Meters/Controllers.* To accurately measure analyte spike flow rate, having the appropriate calibrated range and a stated accuracy of ±2 percent of the absolute measurement value. This device must be calibrated with the major component of the calibration/spike gas (e.g., nitrogen) using an NIST traceable bubble meter or equivalent. Single point calibration checks should be performed daily in the field. When spiking HCl, the mass flow meter/controller should be thoroughly purged before and after introduction of the gas to prevent corrosion of the interior parts.

6.4 *Polytetrafluoroethane tubing.* Diameter and length suitable to connect cylinder regulators.

6.5 *Stainless Steel tubing.* Type 316 of appropriate length and diameter for heated connections.

6.6 *Gas Regulators.* Purgeable HCl regulator.

6.7 *Pressure Gauge.* Capable of measuring pressure from 0 to 1000 Torr (133.3 Pa=1 Torr) within ±5 percent.

6.8 *Sampling Probe.* Glass, stainless steel or other appropriate material of sufficient length and physical integrity to sustain heating, prevent adsorption of analytes and capable of reaching gas sampling point.

6.9 *Sampling Line.* Heated 180 °C (360 °F) and fabricated of either stainless steel, polytetrafluoroethane or other material that prevents adsorption of HCl and transports effluent to analytical instrumentation. The extractive sample line must have the capability to transport sample gas to the analytical components as well as direct heated calibration spike gas to the calibration assembly located at the sample probe. It is important to minimize the length of heated sample line.

6.10 *Particulate Filters.* A sintered stainless steel filter rated at 20 microns or greater may be placed at the inlet of the probe (for removal of large particulate matter). A heated filter (Balston® or equivalent) rated at 1 micron is necessary for primary particulate matter removal, and shall be placed immediately after the heated probe. The

filter/filter holder temperature should be maintained at 180 °C (360 °F).

6.11 *Calibration/Analyte Spike Assembly.* A heated three-way valve assembly (or equivalent) to introduce surrogate spikes into the sampling system at the outlet of the probe before the primary particulate filter.

6.12 *Sample Extraction Pump.* A leak-free heated head pump (KNF® Neuberger or equivalent) capable of extracting sample effluent through entire sampling system at a rate which prevents analyte losses and minimizes analyzer response time. The pump should have a heated by-pass and may be placed either before the FTIR instrument or after. If the sample pump is located upstream of the FTIR instrument, it must be fabricated from materials non-reactive to HCl. The sampling system and FTIR measurement system shall allow the operator to obtain at least six sample spectra during a one-hour period.

6.13 *Barometer.* For measurement of barometric pressure.

6.14 *Gas Sample Manifold.* A distribution manifold having the capabilities listed in sections 6.14.1 through 6.14.4;

6.14.1 Delivery of calibration gas directly to the analytical instrumentation;

6.14.2 Delivery of calibration gas to the sample probe (system calibration or analyte spike) via a heated traced sample line;

6.14.3 Delivery of sample gas (kiln gas, spiked kiln gas, or system calibrations) to the analytical instrumentation;

6.14.4 Delivery (optional) of a humidified nitrogen sample stream.

6.15 *Flow Measurement Device.* Type S Pitot tube (or equivalent) and Magnahelic® set for measurement of volumetric flow rate.

7.0 *Reagents and Standards*

HCl can be purchased in a standard compressed gas cylinder. The most stable HCl cylinder mixture available has a concentration certified at ±5 percent. Such a cylinder is suitable for performing analyte spiking because it will provide reproducible samples. The stability of the cylinder can be monitored over time by periodically performing direct FTIR analysis of cylinder samples. It is recommended that a 10–50 ppm cylinder of HCl be prepared having from 2–5 ppm SF6 as a tracer compound. (See sections 7.1 through 7.3 of Method 320 of this appendix for a complete description of the use of existing HCl reference spectra. See section 9.1 of Method 320 of this appendix for a complete discussion of standard concentration selection.)

8.0 *Sample Collection, Preservation and Storage*

See also Method 320 of this appendix.

8.1 *Pretest.* A screening test is ideal for obtaining proper data that can be used for preparing analytical program files. Information from literature surveys and source personnel is also acceptable. Information about the sampling location and gas stream composition is required to determine the optimum sampling system configuration for measuring HCl. Determine the percent moisture of the kiln gas by Method 4 of appendix A to part 60 of this chapter or by performing a wet bulb/dry bulb measurement. Perform a preliminary traverse

of the sample duct or stack and select the sampling point(s). Acquire an initial spectrum and determine the optimum operational pathlength of the instrument.

8.2 *Leak-Check.* See Method 320 of this appendix, section 8.2 for direction on performing leak-checks.

8.3 *Background Spectrum.* See Method 320 of this appendix, section 8.5 for direction in background spectral acquisition.

8.4 *Pre-Test Calibration Transfer Standard (Direct Instrument Calibration).* See Method 320 of this appendix, section 8.3 for direction in CTS spectral acquisition.

8.5 *Pre-Test System Calibration.* See Method 320 of this appendix, sections 8.6.1 through 8.6.2 for direction in performing system calibration.

8.6 *Sampling*

8.6.1 *Extractive System.* An extractive system maintained at 180 °C (360 °F) or higher which is capable of directing a total flow of at least 12 L/min to the sample cell is required (References 1 and 2). Insert the probe into the duct or stack at a point representing the average volumetric flow rate and 25 percent of the cross sectional area. Co-locate an appropriate flow monitoring device with the sample probe so that the flow rate is recorded at specified time intervals during emission testing (e.g., differential pressure measurements taken every 10 minutes during each run).

8.6.2 *Batch Samples.* Evacuate the absorbance cell to 5 Torr (or less) absolute pressure before taking first sample. Fill the cell with kiln gas to ambient pressure and record the infrared spectrum, then evacuate the cell until there is no further evidence of infrared absorption. Repeat this procedure, collecting a total of six separate sample spectra within a 1-hour period.

8.6.3 *Continuous Flow Through Sampling.* Purge the FTIR cell with kiln gas for a time period sufficient to equilibrate the entire sampling system and FTIR gas cell. The time required is a function of the mechanical response time of the system (determined by performing the system calibration with the CTS gas or equivalent), and by the chemical reactivity of the target analytes. If the effluent target analyte concentration is not variable, observation of the spectral up-date of the flowing gas sample should be performed until equilibration of the sample is achieved. Isolate the gas cell from the sample flow by directing the purge flow to vent. Record the spectrum and pressure of the sample gas. After spectral acquisition, allow the sample gas to purge the cell with at least three volumes of kiln gas. The time required to adequately purge the cell with the required volume of gas is a function of (1) cell volume, (2) flow rate through the cell, and (3) cell design. It is important that the gas introduction and vent for the FTIR cell provides a complete purge through the cell.

8.6.4 *Continuous Sampling.* In some cases it is possible to collect spectra continuously while the FTIR cell is purged with sample gas. The sample integration time, t_{ss}, the sample flow rate through the gas cell, and the sample integration time must be chosen so that the collected data consist of at least 10 spectra with each spectrum being

of a separate cell volume of flue gas. Sampling in this manner may only be performed if the native source analyte concentrations do not affect the test results.

8.7 *Sample Conditioning*

8.7.1 *High Moisture Sampling.* Kiln gas emitted from wet process cement kilns may contain 3- to 40 percent moisture. Zinc selenide windows or the equivalent should be used when attempting to analyze hot/wet kiln gas under these conditions to prevent dissolution of water soluble window materials (e.g., KBr).

8.7.2 *Sample Dilution.* The sample may be diluted using an in-stack dilution probe, or an external dilution device provided that the sample is not diluted below the instrument's quantification range. As an alternative to using a dilution probe, nitrogen may be dynamically spiked into the effluent stream in the same manner as analyte spiking. A constant dilution rate shall be maintained throughout the measurement process. It is critical to measure and verify the exact dilution ratio when using a dilution probe or the nitrogen spiking approach. Calibrating the system with a calibration gas containing an appropriate tracer compound will allow determination of the dilution ratio for most measurement systems. The tester shall specify the procedures used to determine the dilution ratio, and include these calibration results in the report.

8.8 *Sampling QA, Data Storage and Reporting.* See the FTIR Protocol. Sample integration times shall be sufficient to achieve the required signal-to-noise ratio, and all sample spectra should have unique file names. Two copies of sample interferograms and processed spectra will be stored on separate computer media. For each sample spectrum the analyst must document the sampling conditions, the sampling time (while the cell was being filled), the time the spectrum was recorded, the instrumental conditions (path length, temperature, pressure, resolution, integration time), and the spectral file name. A hard copy of these data must be maintained until the test results are accepted.

8.9 *Signal Transmittance.* Monitor the signal transmittance through the instrumental system. If signal transmittance (relative to the background) drops below 95 percent in any spectral region where the sample does not absorb infrared energy, then a new background spectrum must be obtained.

8.10 *Post-test CTS.* After the sampling run completion, record the CTS spectrum. Analysis of the spectral band area used for quantification from pre- and post-test CTS spectra should agree to within ±5 percent or corrective action must be taken.

8.11 *Post-test QA.* The sample spectra shall be inspected immediately after the run to verify that the gas matrix composition was close to the assumed gas matrix, (this is necessary to account for the concentrations of the interferants for use in the analytical analysis programs), and to confirm that the sampling and instrumental parameters were appropriate for the conditions encountered.

9.0 *Quality Control*

Use analyte spiking to verify the effectiveness of the sampling system for the

target compounds in the actual kiln gas matrix. QA spiking shall be performed before and after each sample run. QA spiking shall be performed after the pre- and post-test CTS direct and system calibrations. The system biases calculated from the pre- and post-test dynamic analyte spiking shall be within ±30 percent for the spiked surrogate analytes for the measurements to be considered valid. See sections 9.3.1 through 9.3.2 for the requisite calculations. Measurement of the undiluted spike (direct-to-cell measurement) involves sending dry, spike gas to the FTIR cell, filling the cell to 1 atmosphere and obtaining the spectrum of this sample. The direct-to-cell measurement should be performed before each analyte spike so that the recovery of the dynamically spiked analytes may be calculated. Analyte spiking is only effective for assessing the integrity of the sampling system when the concentration of HCl in the source does not vary substantially. Any attempt to quantify an analyte recovery in a variable concentration matrix will result in errors in the expected concentration of the spiked sample. If the kiln gas target analyte concentrations vary by more than ±5 percent (or 5 ppm, whichever is greater) in the time required to acquire a sample spectrum, it may be necessary to: (1) Use a dual sample probe approach, (2) use two independent FTIR measurement systems, (3) use alternate QA/QC procedures, or (4) postpone testing until stable emission concentrations are achieved. (See section 9.2.3 of this method). It is recommended that a laboratory evaluation be performed before attempting to employ this method under actual field conditions. The laboratory evaluation shall include (1) performance of all applicable calculations in section 4 of the FTIR Protocol; (2) simulated analyte spiking experiments in dry (ambient) and humidified sample matrices using HCl; and (3) performance of bias (recovery) calculations from analyte spiking experiments. It is not necessary to perform a laboratory evaluation before every field test. The purpose of the laboratory study is to demonstrate that the actual instrument and sampling system configuration used in field testing meets the requirements set forth in this method.

9.1 *Spike Materials.* Perform analyte spiking with an HCl standard to demonstrate the integrity of the sampling system.

9.1.1 An HCl standard of approximately 50 ppm in a balance of ultra pure nitrogen is recommended. The SF_6 (tracer) concentration shall be 2 to 5 ppm depending upon the measurement pathlength. The spike ratio (spike flow/total flow) shall be no greater than 1:10, and an ideal spike concentration should approximate the native effluent concentration.

9.1.2 The ideal spike concentration may not be achieved because the target concentration cannot be accurately predicted prior to the field test, and limited calibration standards will be available during testing. Therefore, practical constraints must be applied that allow the tester to spike at an anticipated concentration. For these tests, the analyte concentration contributed by the HCl standard spike should be 1 to 5 ppm or should more closely approximate the native concentration if it is greater.

9.2 *Spike Procedure*

9.2.1 A spiking/sampling apparatus is shown in Figure 2. Introduce the spike/tracer gas mixture at a constant flow ($\pm$2 percent) rate at approximately 10 percent of the total sample flow. (For example, introduce the surrogate spike at 1 L/min 20 cc/min, into a total sample flow rate of 10 L/min). The spike must be pre-heated before introduction into the sample matrix to prevent a localized condensation of the gas stream at the spike introduction point. A heated sample transport line(s) containing multiple transport tubes within the heated bundle may be used to spike gas up through the sampling system to the spike introduction point. Use a calibrated flow device (e.g., mass flow meter/controller), to monitor the spike flow as indicated by a calibrated flow meter or controller, or alternately, the SF_6 tracer ratio may be calculated from the direct measurement and the diluted measurement. It is often desirable to use the tracer approach in calculating the spike/total flow ratio because of the difficulty in accurately measuring hot/wet total flow. The tracer technique has been successfully used in past validation efforts (Reference 1).

9.2.2 Perform a direct-to-cell measurement of the dry, undiluted spike gas. Introduce the spike directly to the FTIR cell, bypassing the sampling system. Fill cell to 1 atmosphere and collect the spectrum of this sample. Ensure that the spike gas has equilibrated to the temperature of the measurement cell before acquisition of the spectra. Inspect the spectrum and verify that the gas is dry and contains negligible CO_2. Repeat the process to obtain a second direct-to-cell measurement. Analysis of spectral band areas for HCl from these duplicate measurements should agree to within $\pm$5 percent of the mean.

9.2.3 *Analyte Spiking*. Determine whether the kiln gas contains native concentrations of HCl by examination of preliminary spectra. Determine whether the concentration varies significantly with time by observing a continuously up-dated spectrum of sample gas in the flow-through sampling mode. If the concentration varies by more than $\pm$5 percent during the period of time required to acquire a spectra, then an alternate approach should be used. One alternate approach uses two sampling lines to convey sample to the gas distribution manifold. One of the sample lines is used to continuously extract unspiked kiln gas from the source. The other sample line serves as the analyte spike line. One FTIR system can be used in this arrangement. Spiked or unspiked sample gas may be directed to the FTIR system from the gas distribution manifold, with the need to purge only the components between the manifold and the FTIR system. This approach minimizes the time required to acquire an equilibrated sample of spiked or unspiked kiln gas. If the source varies by more than $\pm$5 percent (or 5 ppm, whichever is greater) in the time it takes to switch from the unspiked sample line to the spiked sample line, then analyte spiking may not be a feasible means to determine the effectiveness of the sampling system for the HCl in the sample matrix. A second alternative is to use two completely

independent FTIR measurement systems. One system would measure unspiked samples while the other system would measure the spiked samples. As a last option, (where no other alternatives can be used) a humidified nitrogen stream may be generated in the field which approximates the moisture content of the kiln gas. Analyte spiking into this humidified stream can be employed to assure that the sampling system is adequate for transporting the HCl to the FTIR instrumentation.

9.2.3.1 Adjust the spike flow rate to approximately 10 percent of the total flow by metering spike gas through a calibrated mass flowmeter or controller. Allow spike flow to equilibrate within the sampling system before analyzing the first spiked kiln gas samples. A minimum of two consecutive spikes are required. Analysis of the spectral band area used for quantification should agree to within $\pm$5 percent or corrective action must be taken.

9.2.3.2 After QA spiking is completed, the sampling system components shall be purged with nitrogen or dry air to eliminate traces of the HCl compound from the sampling system components. Acquire a sample spectra of the nitrogen purge to verify the absence of the calibration mixture.

9.2.3.3 Analyte spiking procedures must be carefully executed to ensure that meaningful measurements are achieved. The requirements of sections 9.2.3.3.1 through 9.2.3.3.4 shall be met.

9.2.3.3.1 The spike must be in the vapor phase, dry, and heated to (or above) the kiln gas temperature before it is introduced to the kiln gas stream.

9.2.3.3.2 The spike flow rate must be constant and accurately measured.

9.2.3.3.3 The total flow must also be measured continuously and reliably or the dilution ratio must otherwise be verified before and after a run by introducing a spike of a non-reactive, stable compound (i.e., tracer).

9.2.3.3.4 The tracer must be inert to the sampling system components, not contained in the effluent gas, and readily detected by the analytical instrumentation. Sulfur hexafluoride (SF_6) has been used successfully (References 1 and 2) for this purpose.

9.3 *Calculations*

9.3.1 *Recovery*. Calculate the percent recovery of the spiked analytes using equations 1 and 2.

$$\%R = 100 \times \frac{S_m - S_u(1-DF)}{DF \times C_s} \qquad (1)$$

S_m = Mean concentration of the analyte spiked effluent samples (observed).

$$C_e = DF \times C_s + S_u(1-DF) \qquad (2)$$

C_e = Expected concentration of the spiked samples (theoretical).
D_f = dilution Factor (Total flow/Spike flow). total flow = spike flow plus effluent flow.
C_s = cylinder concentration of spike gas.
S_u = native concentration of analytes in unspiked samples.

The spike dilution factor may be confirmed by measuring the total flow and the spike flow directly. Alternately, the spike dilution can be verified by comparing the concentration of the tracer compound in the spiked samples (diluted) to the tracer concentration in the direct (undiluted) measurement of the spike gas. If SF_6 is the tracer gas, then

$$D_f = [SF_6]_{spike} / [SF_6]_{direct} \qquad (3)$$

$[SF_6]_{spike}$ = the diluted SF_6 concentration measured in a spiked sample.
$[SF_6]_{direct}$ = the SF_6 concentration measured directly.

9.3.2 Bias. The bias may be determined by the difference between the observed spike value and the expected response (i.e., the equivalent concentration of the spiked material plus the analyte concentration adjusted for spike dilution). Bias is defined by section 6.3.1 of EPA Method 301 of this appendix (Reference 8) as,

$$B = S_m - C_e \qquad (4)$$

Where:
B = Bias at spike level.
S_m = Mean concentration of the analyte spiked samples.
C_e = Expected concentration of the analyte in spiked samples.

Acceptable recoveries for analyte spiking are $\pm$30 percent. Application of correction factors to the data based upon bias and recovery calculations is subject to the approval of the Administrator.

10.0 *Calibration and Standardization*

10.1 *Calibration transfer standards (CTS)*. The EPA Traceability Protocol gases or NIST traceable standards, with a minimum accuracy of $\pm$2 percent shall be used. For other requirements of the CTS, see the FTIR Protocol section 4.5.

10.2 *Signal-to-Noise Ratio (S/N)*. The S/N shall be less than the minimum acceptable measurement uncertainty in the analytical regions to be used for measuring HCl.

10.3 *Absorbance Pathlength*. Verify the absorbance path length by comparing CTS spectra to reference spectra of the calibration gas(es).

10.4 *Instrument Resolution*. Measure the line width of appropriate CTS band(s) to verify instrumental resolution.

10.5 *Apodization Function*. Choose the appropriate apodization function. Determine any appropriate mathematical transformations that are required to correct instrumental errors by measuring the CTS. Any mathematical transformations must be documented and reproducible. Reference 9 provides additional information about FTIR instrumentation.

11.0 *Analytical Procedure*

A full description of the analytical procedures is given in sections 4.6–4.11, sections 5, 6, and 7, and the appendices of the FTIR Protocol. Additional description of quantitative spectral analysis is provided in References 10 and 11.

12.0 *Data Analysis and Calculations*

Data analysis is performed using appropriate reference spectra whose concentrations can be verified using CTS spectra. Various analytical programs (References 10 and 11) are available to relate sample absorbance to a concentration standard. Calculated concentrations should be verified by analyzing spectral baselines after mathematically subtracting scaled reference spectra from the sample spectra. A full description of the data analysis and calculations may be found in the FTIR Protocol (sections 4.0, 5.0, 6.0 and appendices).

12.1 Calculated concentrations in sample spectra are corrected for differences in absorption pathlength between the reference and sample spectra by

$$C_{corr} = \left(L_r / L_s\right) \times \left(T_s / T_r\right) \times \left(C_{calc}\right) \qquad (5)$$

Where:

C_{corr} = The pathlength corrected concentration.

C_{calc} = The initial calculated concentration (output of the multicomponent analysis program designed for the compound).

L_r = The pathlength associated with the reference spectra.

L_s = The pathlength associated with the sample spectra.

T_s = The absolute temperature (K) of the sample gas.

T_r = The absolute temperature (K) at which reference spectra were recorded.

12.2 The temperature correction in equation 5 is a volumetric correction. It does not account for temperature dependence of rotational-vibrational relative line intensities. Whenever possible, the reference spectra used in the analysis should be collected at a temperature near the temperature of the FTIR cell used in the test to minimize the calculated error in the measurement (FTIR Protocol, appendix D). Additionally, the analytical region chosen for the analysis should be sufficiently broad to minimize errors caused by small differences in relative line intensities between reference spectra and the sample spectra.

13.0 *Method Performance*

A description of the method performance may be found in the FTIR Protocol. This method is self validating provided the results meet the performance specification of the QA spike in sections 9.0 through 9.3 of this method.

14.0 *Pollution Prevention*

This is a gas phase measurement. Gas is extracted from the source, analyzed by the instrumentation, and discharged through the instrument vent.

15.0 *Waste Management*

Gas standards of HCl are handled according to the instructions enclosed with the material safety data sheet.

16.0 *References*

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7. ''Laboratory and Field Evaluations of Ammonium Chloride Interference in Method 26,'' U.S. Environmental Protection Agency Report, Entropy, Inc., EPA Contract No. 68D20163, Work Assignment No. I–45.

8. 40 CFR 63, appendix A. Method 301—Field Validation of Pollutant Measurement Methods from Various Waste Media.

9. ''Fourier Transform Infrared Spectrometry,'' Peter R. Griffiths and James de Haseth, Chemical Analysis, 83, 16–25, (1986), P.J. Elving, J.D. Winefordner and I.M. Kolthoff (ed.), John Wiley and Sons.

10. ''Computer-Assisted Quantitative Infrared Spectroscopy,'' Gregory L. McClure (ed.), ASTM Special Publication 934 (ASTM), 1987.

11. ''Multivariate Least-Squares Methods Applied to the Quantitative Spectral Analysis of Multicomponent Mixtures,'' Applied Spectroscopy, 39(10), 73–84, 1985.

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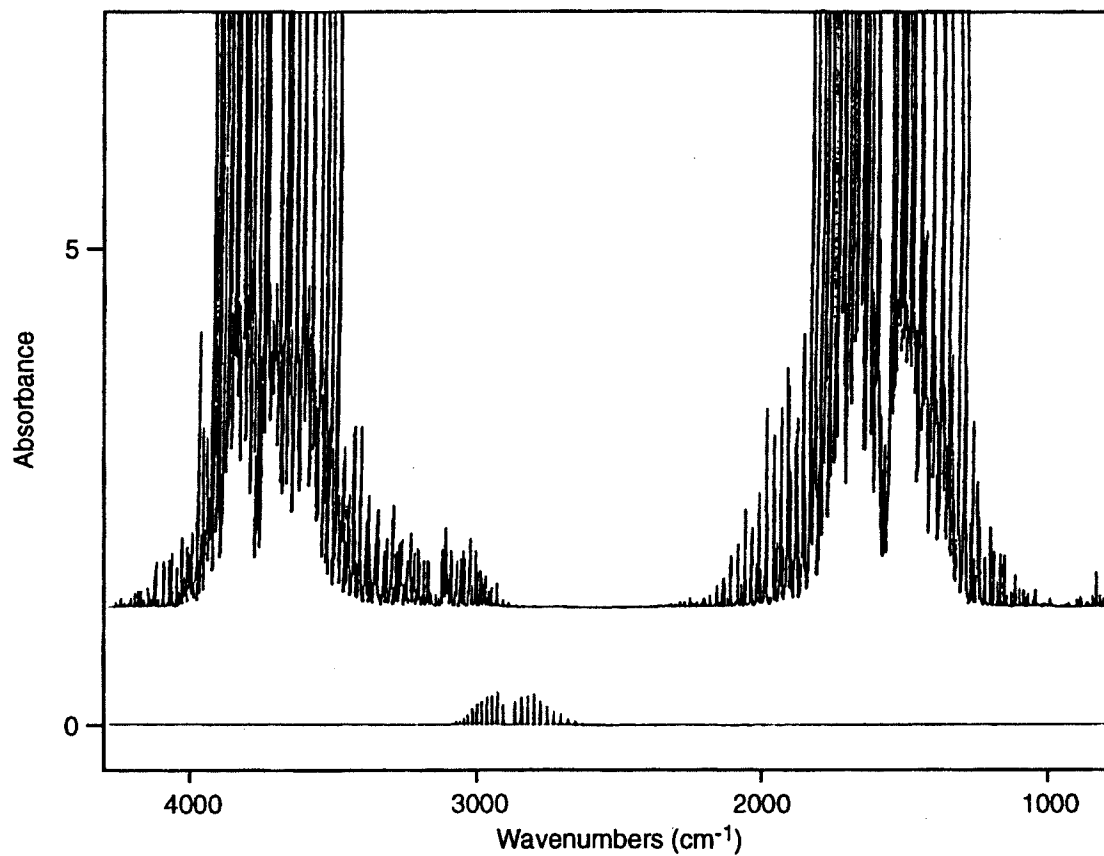
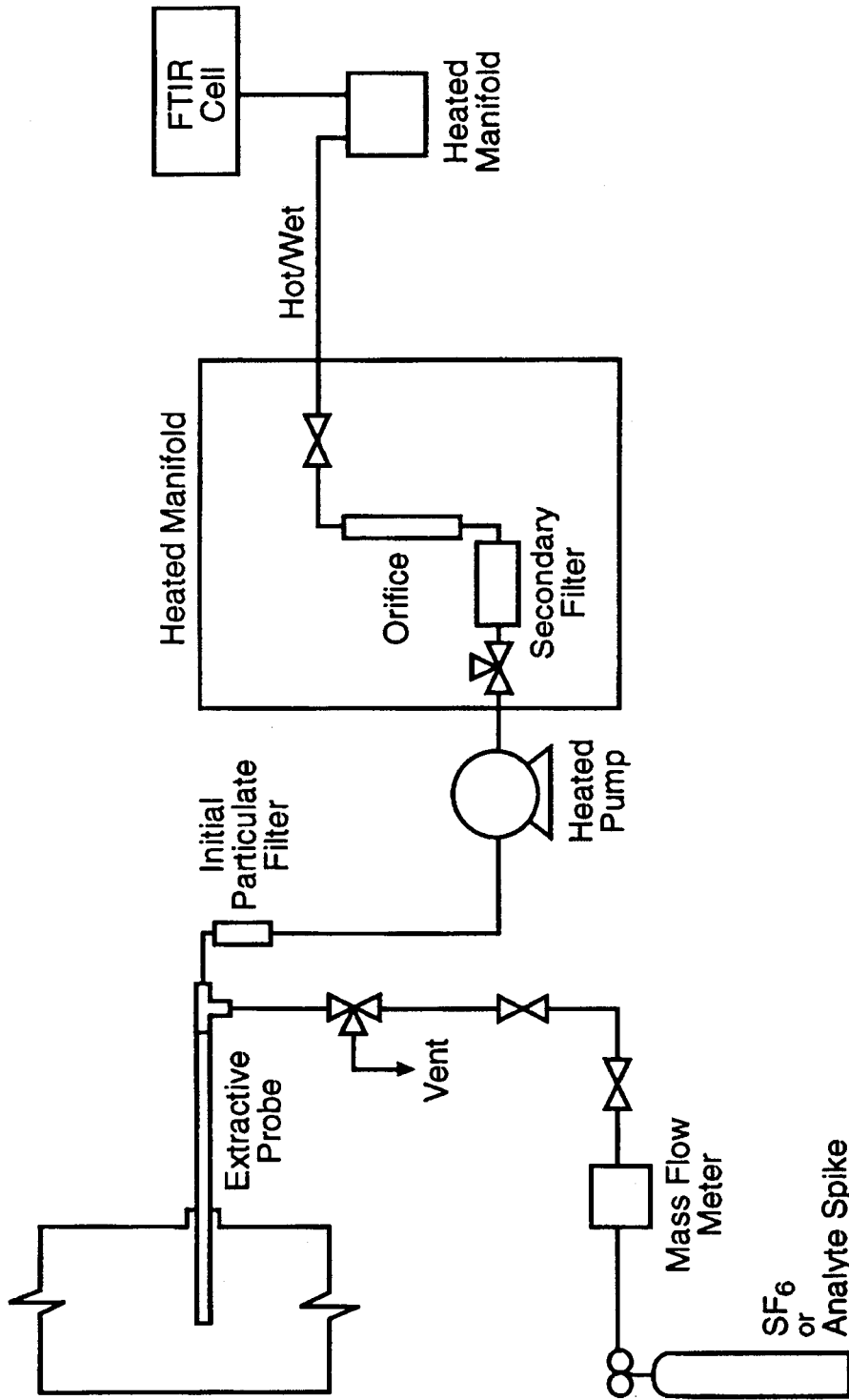Top, water vapor (194hsub); Bottom, HCl at 11.4 (ppm-M)/K.

Figure 1. FTIR Spectra of HCl and Water.

Figure 2. FTIR Sampling/Spiking System.

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