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## Title 40—Protection of Environment

CHAPTER I—ENVIRONMENTAL  
PROTECTION AGENCY

## SUBCHAPTER C—AIR PROGRAMS

## PART 60—STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES

## Additions and Miscellaneous Amendments

On June 11, 1973 (38 FR 15406), pursuant to section 111 of the Clean Air Act, as amended, the Administrator proposed standards of performance for new and modified stationary sources within seven categories of stationary sources: (1) Asphalt concrete plants, (2) petroleum refineries, (3) storage vessels for petroleum liquids, (4) secondary lead smelters, (5) secondary brass and bronze ingot production plants, (6) iron and steel plants, and (7) sewage treatment plants. In the same publication, the Administrator also proposed amendments to subpart A, General Provisions, and to the Appendix, Test Methods, of 40 CFR Part 60.

Interested parties participated in the rulemaking by sending comments to EPA. Some 253 letters, many with multiple comments, were received from commentators, and about 152 were received from Congressmen making inquiries on behalf of their constituents. Copies of the comments received directly are available from public inspection at the EPA Office of Public Affairs, 401 M Street SW., Washington, D.C. 20460. The comments have been considered, additional data have been collected and assessed, and the standards have been reevaluated. Where determined by the Administrator to be appropriate, revisions have been made to the proposed standards. The promulgated standards, the principal revisions to the proposed standards, and the Agency's responses to major comments are summarized below. More detail may be found in *Background Information for New Source Performance Standards: Asphalt Concrete Plants, Petroleum Refineries, Storage Vessels, Secondary Lead Smelters and Refineries, Brass and Bronze Ingot Production Plants, Iron and Steel Plants, and Sewage Treatment Plants, Volume 3, Promulgated Standards*, (APTD-1352c) which is available on request from the Emission Standards and Engineering Division, Research Triangle Park, North Carolina 27711, Attention: Mr. Don R. Goodwin.

Discussions of the environmental impact of these standards of performance for new sources are contained in Volume 1, Main Text (APTD-1352a), of the background document. This volume and Volume 2, *Appendix: Summaries of Test Data* (APTD-1352b), are still available on request from the office noted above.

In accordance with section 111 of the Act, these regulations prescribing standards of performance for the selected stationary sources are effective on February 28, 1974 and apply to sources the construction or modification of which was commenced after June 11, 1973.

## GENERAL PROVISIONS

These promulgated regulations include changes to subpart A, General Pro-

visions, which applies to all new sources. The general provisions were published on December 23, 1971 (36 FR 24876). The definition of "commenced" has been altered to exclude the act of entering into a binding agreement to construct or modify a source from among the specified acts which, if taken by an owner or operator of a source on or after the date on which an applicable new source performance standard is proposed, cause the source to be subject to the promulgated standard. The phrase "binding agreement" was duplicate terminology for the phrase "contractual obligation" but was being construed incorrectly to apply to other arrangements. Deletion of the first phrase and retention of the second phrase eliminates the problem. The definition of "standard conditions" replaces the definition of "standard or normal conditions" to avoid the confusion, noted by commentators, created by the duplicate terminology. The promulgated definition also expresses the temperature and pressure in commonly used metric units to be consistent with the Administrator's policy of converting to the metric system. Four definitions are added: "Reference method," "equivalent method," "alternative method," and "run," to clarify the terms used in changes to § 60.8, Performance Tests, discussed below. The definition of "particulate matter" is added here and removed from each of the subparts specific to this group of new sources to avoid repetition. The word "run," as used in the sections pertinent to performance tests, is defined as the net time required to collect an adequate sample of a pollutant, and may be either intermittent or continuous. Section 60.3, Abbreviations, is revised to include new abbreviations, to accord more closely with standard usage, and to alphabetize the listing. Section 60.4, Address, is revised to change the address to which all requests, reports, applications, submittals, and other communications will be submitted to the Administrator pursuant to any regulatory provision. Such communications are now to be addressed to the Director of the Enforcement Division in the appropriate EPA regional office rather than to the Office of General Enforcement in Washington, D.C. The addresses of all 10 regional offices are included, and the "in triplicate" requirement is changed to "in duplicate." Some of the wording is changed in § 60.6, Review of Plans, to require that owners or operators requesting review of plans for construction or modification make a separate request for each project rather than for each affected facility as previously required; each such facility, however, must be identified and appropriately described. A paragraph is added to § 60.7, Notification and Recordkeeping, to require owners and operators to maintain a file of all recorded information required by the regulations for at least 2 years after the dates of such information, and this requirement is removed from the subparts specific to each of the new sources in this group to avoid repetition. Section 60.8, Performance Tests, is amended (1) to re-

quire owners and operators to give the Administrator 30 days' advance notice, instead of 10 days', of performance testing to demonstrate compliance with standards in order to provide the Administrator with a better opportunity to have an observer present, (2) to specify the Administrator's authority to permit, in specific cases, the use of minor changes to reference methods, the use of equivalent or alternative methods, or the waiver of the performance test requirement, and (3) to specify that each performance test shall consist of three runs except where the Administrator approves the use of two runs because of circumstances beyond the control of the owner or operator. These amendments give the Administrator needed flexibility for making judgments for determining compliance with standards. Section 60.12, Circumvention, is added to clearly prohibit owners and operators from using devices or techniques which conceal, rather than control, emissions to comply with standards of performance for new sources. The standards proposed on June 11, 1973, contained provisions which required compliance to be based on undiluted gases. Many commentators pointed out the inequities of these provisions and the vagueness of the language used. Because many processes require the addition of air in various quantities for cooling, for enhancing combustion, and for other useful purposes, no single definition of excess dilution air can be sensibly applied. It is considered preferable to state clearly what is prohibited and to use the Administrator's authority to specify the conditions for compliance testing in each case to ensure that the prohibited concealment is not used.

## OPACITY

It is evident from comments received that an inadequate explanation was given for applying both an enforceable opacity standard and an enforceable concentration standard to the same source and that the relationship between the concentration standard and the opacity standard was not clearly presented. Because all but one of the regulations include these dual standards, this subject is dealt with here from the general viewpoint. Specific changes made to the regulations proposed for a specific source are described in the discussions of each source.

A discussion of the major points raised by the comments on the opacity standard follows:

1. Several commentators felt that opacity limits should be only guidelines for determining when to conduct the stack tests needed to determine compliance with concentration/mass standards. Several other commentators expressed the opinion that the opacity standard was more stringent than the concentration/mass standard.

As promulgated below, the opacity standards are regulatory requirements, just like the concentration/mass standards. It is not necessary to show that the concentration/mass standard is being violated in order to support enforcement of the opacity standard. Where opacity and concentration/mass standards are

applicable to the same source, the opacity standard is not more restrictive than the concentration/mass standard. The concentration/mass standard is established at a level which will result in the design, installation, and operation of the best adequately demonstrated system of emission reduction (taking costs into account) for each source. The opacity standard is established at a level which will require proper operation and maintenance of such control systems on a day-to-day basis, but not require the design and installation of a control system more efficient or expensive than that required by the concentration/mass standard.

Opacity standards are a necessary supplement to concentration/mass standards. Opacity standards help ensure that sources and emission control systems continue to be properly maintained and operated so as to comply with concentration/mass standards. Particulate testing by EPA method 5 and most other techniques requires an expenditure of \$3,000 to \$10,000 per test including about 300 man-hours of technical and semi-technical personnel. Furthermore, scheduling and preparation are required such that it is seldom possible to conduct a test with less than 2 weeks notice. Therefore, method 5 particulate tests can be conducted only on an infrequent basis.

If there were no standards other than concentration/mass standards, it would be possible to inadequately operate or maintain pollution control equipment at all times except during periods of performance testing. It takes 2 weeks or longer to schedule a typical stack test. If only small repairs were required, e.g., pump or fan repair or replacement of fabric filter bags, such remedial action could be delayed until shortly before the test is conducted. For some types of equipment such as scrubbers, the energy input could be reduced (the pressure drop through the system) when stack tests weren't being conducted, which would result in the release of significantly more particulate matter than normal. Therefore, EPA has required that operators properly maintain air pollution control equipment at all times (40 CFR 60.11(d)) and meet opacity standards at all times except during periods of startup, shutdown, and malfunction (40 CFR 60.11(c)), and during other periods of exemption as specified in individual regulations.

Opacity of emissions is indicative of whether control equipment is properly maintained and operated. However, it is established as an independent enforceable standard, rather than an indicator of maintenance and operating conditions because information concerning the latter is peculiarly within the control of the plant operator. Furthermore, the time and expense required to prove that proper procedures have not been followed are so great that the provisions of 40 CFR 60.11(d) by themselves (without opacity standards) would not provide an economically sensible means of ensuring on a day-to-day basis that emissions of pollutants are within allowable limits. Opacity standards require nothing more

than a trained observer and can be performed with no prior notice. Normally, it is not even necessary for the observer to be admitted to the plant to determine properly the opacity of stack emissions. Where observed opacities are within allowable limits, it is not normally necessary for enforcement personnel to enter the plant or contact plant personnel. However, in some cases, including times when opacity standards may not be violated, a full investigation of operating and maintenance conditions will be desirable. Accordingly, EPA has requirements for both opacity limits and proper operating and maintenance procedures.

2. Some commentators suggested that the regulatory opacity limits should be lowered to be consistent with the opacity observed at existing plants; others felt that the opacity limits were too stringent. The regulatory opacity limits are sufficiently close to observed opacity to ensure proper operation and maintenance of control systems on a continuing basis but still allow some room for minor variations from the conditions existing at the time opacity readings were made.

3. There are specified periods during which opacity standards do not apply. Commentators questioned the rationale for these time exemptions, as proposed, some pointing out that the exemptions were not justified and some that they were inadequate. Time exemptions further reflect the stated purpose of opacity standards by providing relief from such standards during periods when acceptable systems of emission reduction are judged to be incapable of meeting prescribed opacity limits. Opacity standards do not apply to emissions during periods of startup, shutdown, and malfunction (see FEDERAL REGISTER of October 15, 1973, 38 FR 28564), nor do opacity standards apply during periods judged necessary to permit the observed excess emissions caused by soot-blowing and unstable process conditions. Some confusion resulted from the fact that the startup-shutdown-malfunction regulations were proposed separately (see FEDERAL REGISTER of May 2, 1973, 38 FR 10820) from the regulations for this group of new sources. Although this was pointed out in the preamble (see FEDERAL REGISTER of June 11, 1973, 38 FR 15406) to this group of new source performance standards, it appears to have escaped the notice of several commentators.

4. Other comments, along with re-study of sources and additional opacity observations, have led to definition of specific time exemptions, where needed, to account for excess emissions resulting from soot-blowing and process variations. These specific actions replace the generalized approach to time exemptions, 2 minutes per hour, contained in all but one of the proposed opacity standards. The intent of the 2 minutes was to prevent the opacity standards from being unfairly stringent and reflected an arbitrary selection of a time exemption to serve this purpose. Commentators noted that observed opacity and operating conditions did not support this approach. Some pointed out that these

exemptions were not warranted; others, that they were inadequate. The cyclical basic oxygen steel-making process, for example, does not operate in hourly cycles and the inappropriateness of 2 minutes per hour in this case would apply to other cyclical processes which exist both in sources now subject to standards of performance and sources for which standards will be developed in the future. The time exemptions now provide for circumstances specific to the sources and, coupled with the startup-shutdown-malfunction provisions and the higher-than-observed opacity limits, provide much better assurance that the opacity standards are not unfairly stringent.

#### ASPHALT CONCRETE PLANTS

The promulgated standards for asphalt concrete plants limit particulate matter emissions to 90 mg/dscm (0.04 gr/dscf) and 20 percent opacity.

The majority of the comments received on the seven proposed standards related to the proposed standards for asphalt concrete plants. Out of the 253 letters, over 65 percent related to the proposed standards for asphalt concrete plants. Each of the comments was reviewed and evaluated. The Agency's responses to the comments received are included in Appendix E of Volume 3 of the background information document. The Agency's rationale for the promulgated standards for asphalt concrete plants is summarized below. A more detailed statement is presented in Volume 3 of the background information document.

The major differences between the proposed standards and the promulgated standards are:

1. The concentration standard has been changed from 70 mg/dscm (0.031 gr/dscf) to 90 mg/dscm (0.04 gr/dscf).

2. The opacity standard has been changed from 10 percent with a 2-minute-per-hour exemption to 20 percent with no specified time exemption.

3. The definition of affected facility has been reworded to better define the applicability of the standards.

The preamble to the proposed standard (38 FR 15406) urged all interested parties to submit factual data during the comment period to ensure that the standard for asphalt concrete plants would, upon promulgation, be consistent with the requirements of section 111 of the Act. A substantial amount of information on emission tests was submitted in response to this request. The information is summarized and discussed in Volume 3 of the background information document.

The proposed concentration standard was based on the conclusion that the best demonstrated systems of emission reduction, considering costs, are well designed, operated, and maintained baghouses or venturi scrubbers. The emission test data available at the time of proposal indicated that such systems could attain an emission level of 70 mg/Nm<sup>3</sup>, or 0.031 gr/dscf. After considering comments on the proposed standard and new emission test data, a thorough eval-

ulation was made of the achievability of the proposed standard. As a result of this evaluation, the concentration standard was changed to 90 mg/dscm, or 0.04 gr/dscf.

With the exception of three cases, the acceptable data had shown that the proposed concentration standard, 0.031 gr/dscf, is achievable with a properly designed, installed, operated, and maintained baghouse or venturi scrubber. The three exceptions, two plants equipped with baghouses and one with a venturi scrubber, had emissions between 0.031 and 0.04 gr/dscf.

Some of the major comments received from the industry were (1) the proposed concentration standard of 0.031 gr/dscf cannot be attained either consistently or at all with currently available equipment; (2) the standard should be 0.06 gr/dscf; (3) the standard should allow higher emissions when heavy fuel oil is burned; (4) the type of aggregate used by a plant changes and affects the emissions; (5) EPA failed to consider the impact of the standard on mobile plants, continuous-mix plants, and drum-mixing plants; and (6) the EPA control cost estimates are too low. Responses to these comments and others are given in Appendix E to Volume 3 of the background information document. When considered as a whole, along with the new emission data, the comments justify revising the standard. The revision is merely a change in EPA's judgment about what emission limit is achievable using the best systems of emission reduction. The revision is in no way a change in what EPA considers to be the best systems of emission reduction which, taking into account the cost of achieving such reduction, have been adequately demonstrated; these are still considered to be well designed, operated, and maintained baghouses or venturi scrubbers.

In response to comments received on the proposed opacity standard, additional data were obtained on visible emissions from three well-controlled plants. The data are summarized in Volume 3 of the background information document. No visible emissions were observed from the control equipment on any of the plants. In addition, one plant showed no visible fugitive emissions. Inspection of the two plants having visible fugitive emissions, together with the fact that one plant had no visible emissions, shows that all of the fugitive emissions observed could have been prevented by proper design, operation, and maintenance of the asphalt plant and its control equipment. The data show no normal process variations that would cause visible emissions, either fugitive or from the control device, at a well-controlled plant.

As indicated above in the discussion on opacity, the opacity standards are set such that they are not more restrictive than the applicable concentration standard. In the case of asphalt concrete plants, it is the judgment of the Administrator that if a plant's emissions equal or exceed 20 percent opacity, the emis-

sions will also clearly exceed the concentration standard of 90 mg/dscm (0.04 gr/dscf). Therefore, the promulgated standard of 20 percent opacity is not more restrictive than the concentration standard and no specific time exemptions are considered necessary.

An additional relief from the opacity standard is provided by the regulation promulgated on October 15, 1973 (38 FR 28564), which exempts from opacity standards any emissions generated during startups, shutdowns, or malfunctions. A general discussion of the purpose of opacity standards and the issues involved in setting them is included in Chapter 2, Volume 3, of the background information document.

Section 60.90, applicability and designation of affected facility, is changed from that proposed in order to clarify how and when the standards apply to asphalt concrete plants. The proposed regulation was interpreted by some commentators as requiring existing plants to meet the standards of performance for new sources when equipment was normally replaced or modernized. The proposed regulation specified certain equipment, e.g., transfer and storage systems, as affected facilities, and, because of regulatory language, this could have been interpreted to mean that a new conveyor system installed to replace a worn-out conveyor system on an existing plant was a new source as defined in section 111(a)(2) of the Act. The promulgated regulation specifies the asphalt concrete plant as the affected facility in order to avoid this unwanted interpretation. An existing asphalt concrete plant is subject to the promulgated standards of performance for new sources only if a physical change to the plant or change in the method of operating the plant causes an increase in the amount of air pollutants emitted. Routine maintenance, repair and replacement; relocation of a portable plant; change of aggregate; and transfer of ownership are not considered modifications which would require an existing plant to comply with the standard.

Industry's comments on the cost estimates pertinent to the proposed standards pointed out some errors and oversights. The cost estimates have been revised to include: (1) An increase in the investment cost for baghouses, (2) a change of credit for mineral filler from \$9.00 to \$3.40 per ton, and (3) an increase in the disposal costs. The changes increased the estimated investment cost of the control equipment by approximately 20 percent. The revised cost estimates are presented in Volume 3 of the background information document. It is concluded after evaluating the revised estimates that a baghouse designed with a 6-to-1 air-to-cloth ratio or a venturi scrubber with a pressure drop of at least 20 inches water gauge can be installed, operated, and maintained at a reasonable cost. It should be noted that the cost estimates were revised because the original estimates contained some errors and oversights, not because the concentration standard was changed.

#### PETROLEUM REFINERIES

The promulgated standards for petroleum refineries limit emissions of sulfur dioxide from fuel gas combustion systems and limit emissions of particulate matter and carbon monoxide from fluid catalytic cracking unit catalyst regenerators.

Each of the comments received on the proposed standards was reviewed and evaluated. The Agency's responses to the comments received are included in Appendix E of Volume 3 of the background information document. The Agency's rationale for the promulgated standards for petroleum refineries is summarized below. A more detailed statement is presented in Volume 3 of the background information document.

The major differences between the promulgated standards and the proposed standards are:

1. The combustion of process upset gases in flare systems has been exempted.
2. Hydrogen sulfide in fuel gases combusted in any number of facilities may be monitored at one location if sampling at this location yields results representative of the hydrogen sulfide concentration in the fuel gas combusted in each facility.
3. The opacity standard for catalyst regenerators has been changed from the proposed level of less than 20 percent except for 3 minutes in any 1 hour to less than 30 percent except for 3 minutes in any 1 hour.
4. The standard for particulate matter has been changed from the proposed level of 50 mg/Nm<sup>3</sup> (0.022 gr/dscf) to 1.0 kilogram per 1,000 kilograms of coke burn-off in the catalyst regenerator (0.027 gr/dscf).

The two changes made to the proposed standard for fuel gas combustion systems do not represent any change in the Agency's original intent. It was evident from the comments received, however, that the intent of the regulation was not clear. Therefore, explicit provisions were incorporated into the promulgated standard to exempt the flaring of process upset gases and to permit monitoring at one location of the hydrogen sulfide content of fuel gases combusted in any number of combustion devices. Although hydrogen sulfide monitors are widely used by industry, the Agency has not evaluated the operating characteristics of such instruments. For this reason, calibration and zero specifications have been prescribed in only general terms. On the basis of evaluation programs currently underway, these requirements will be revised, or further guidance will be provided concerning the selection, operation, and maintenance of such instruments.

Commentators suggested that small petroleum refineries be exempt from the standard for fuel gas combustion systems since compliance with the standard would impose a severe economic penalty on small refineries. This problem was considered during the development of the proposed standard. It was concluded, however, that the proposed standard would have little or no adverse economic impact on petroleum refineries. In light

of the comments received, the Agency reexamined this point with particular attention to the small refiner.

The details of the analysis are presented in Appendix C to Volume 3 of the background information document. The domestic petroleum industry is extremely complex and highly sophisticated. Thus, any analysis of the petroleum refining industry will of necessity be based on a number of simplifying assumptions. Although the assumptions in the economic impact statement appear reasonable, the statement should not be viewed as definitively identifying specific costs; rather it identifies a range of costs and approximate impact points. The analysis examines more than the economic impact of the standard for fuel gas combustion systems. It also examines the combined economic impact of this standard for fuel gas combustion systems, the standards for fluid catalytic cracking units, the water quality effluent guidelines being developed for petroleum refineries, and EPA's regulations requiring the reduction of lead in gasoline. Essentially, the economic impact of 'pollution control' is reviewed in light of the petroleum import license-fee program being administered by the Oil and Gas Office of the Department of the Interior (38 FR 9645 and 38 FR 16195).

This program is designed to encourage expansion and construction of U.S. petroleum refining capacity and expansion of U.S. crude oil production by imposing a fee or tariff on imported petroleum products and crude oil. Although this program is currently being phased into practice with the full impact not to be felt until mid-1975, the central feature of the program is to impose a fee of 21¢ per barrel above world price on imported crude oil and a fee of 63¢ per barrel above world price on imported petroleum products such as gasoline, fuel oils, and 'unfinished' or intermediate petroleum products.

Under the conditions currently existing in the United States, which are forecast to continue throughout the remainder of this decade and most of the next decade, and with domestic demand for crude oil and petroleum products far outstripping domestic supply and petroleum refining capacity, the import license-fee program will encourage domestic prices of crude oil and petroleum products to increase to world levels plus the fee or tariff. Thus, an incentive of 42¢ per barrel (63¢ per barrel minus 21¢ per barrel) is provided to domestic refiners by this program. In cases where 'independent' refiners continue to enjoy a captive supply of domestic crude oil, or where 'major' refiners engaged in the exploration and production of domestic crude are successful in supplying their refineries with domestic crude oil, this incentive will approach the full 63¢ per barrel fee imposed on imported petroleum products.

The analysis indicates that the incentive provided to the domestic petroleum refining industry by the import license-fee program is greater than the costs of pollution control requirements. The

differences in control costs for the small refiner relative to the large refiner will still exist, but with the fee system in operation the small refiner will not be forced into a no-growth situation because of compliance with EPA requirements. Therefore small refineries are not exempt from the standards.

In response to comments received on the proposed opacity standard, additional data were obtained on visible emissions from four well-controlled catalyst regenerators. The data, which are summarized in Volume 3 of the background information document, indicate that 20 percent opacity is too restrictive for a well-controlled plant. As indicated above in the discussion on opacity, it is the Administrator's intent to set opacity standards such that they are not more restrictive than the applicable concentration or mass standard. In the case of catalyst regenerators, it is the judgment of the Administrator that if visible emissions exceed 30 percent opacity except for 3 minutes in any 1 hour, such emissions will also clearly exceed the standard of 1.0 kilogram of particulate matter per 1,000 kilograms of coke burn-off. Therefore, the promulgated standard of 30 percent except for 3 minutes in any 1 hour is judged to be not more restrictive than the mass standard of 1.0 kg/1,000 kg of coke burn-off.

An additional relief from the opacity standard is provided by the regulations promulgated on October 15, 1973 (38 FR 28564), which exempt from opacity standards any emissions generated during startups, shutdowns, or malfunctions. A general discussion of the purpose of opacity standards and the issues involved in setting them is included in Chapter 2 of Volume 3 of the background information document.

Commentators pointed out that the volume of gases discharged to the atmosphere from catalyst regenerators can vary significantly, depending upon the overall system used to control emissions of particulate matter and carbon monoxide. Consequently, the degree of control required to meet the proposed concentration standard (50 mg/Nm<sup>3</sup>) for particulate matter depends upon the overall type of emission control system employed.

The various types of emission control systems utilized by catalyst regenerators and the alternative means of expressing an emission standard for particulate matter other than by an allowable concentration of particulate matter were evaluated. The alternative ways of expressing the standard were (1) specification of control efficiency, (2) limiting emissions based on a process weight restriction, and (3) limiting emissions on the basis of the size or capacity of a unit. Expressing the standard in terms of kilograms of particulate matter per 1,000 kilograms of coke burn-off was determined to be the best alternative.

Several of those who wrote to the Agency indicated that the proposed particulate matter standard for catalyst regenerators (50 mg/Nm<sup>3</sup>) was too restrictive. To fully evaluate these com-

ments, additional data on emissions from well-controlled units were obtained from industry and a control agency. This new information and the detailed rationale for the promulgated standard are presented in Volume 3 of the background information document.

This evaluation led to the conclusion that the allowable particulate matter emissions should be increased to provide for the unavoidable increase in emissions due to the deterioration of the cyclones within a catalyst regenerator. The revision reflects a change in the Agency's judgment on what emission limit is achievable using the best systems of emission reduction; it is not a change in what the Agency considers to be the best systems of emission reduction that have been adequately demonstrated.

#### STORAGE VESSELS FOR PETROLEUM LIQUIDS

The promulgated standard applies to storage vessels with capacities greater than 151,412 liters (40,000 gallons) that contain crude petroleum, condensate, or finished or intermediate products of a petroleum refinery. To reduce emissions of hydrocarbons to the atmosphere, a vapor recovery system or equivalent control is required if the stored liquid has a true vapor pressure, under storage conditions, greater than 570 millimeters of mercury (mm Hg); and a floating roof or equivalent control is required if the stored liquid's pressure is between 78 and 570 mm Hg, inclusive. Records must be kept of liquids stored, by date; of typical vapor pressure; and, in certain cases, of average monthly storage temperatures. The Administrator may require, in specific cases, that the liquid be sampled and true vapor pressure determined, but normally the maintenance of good records that are ready for inspection will be the requirement that owners and operators must meet in order to demonstrate compliance.

The definition of "storage vessel" is changed from the proposed form to specifically exclude high-pressure vessels, subsurface caverns, porous-rock reservoirs, and some underground tanks. As commentators pointed out, these types of storage are optimum for preventing the release of emissions to the atmosphere and need no additional control devices. The proposed definition of "petroleum liquids" was validly criticized as being too inclusive, and it is changed to specify what is included and what is excluded. The definition of vapor recovery system expresses the intent, in part, of such a system as "to prevent \* \* \* emission." Some commentators felt that this could imply a requirement for 100 percent effectiveness. The definition is consistent with the wording found in many State and local regulations for storage of petroleum liquids—regulations that have been sensibly enforced and complied with. EPA recognizes that the effectiveness of such systems varies with climate and types and concentrations of vapors and deliberately avoided requiring a specific level of effectiveness. Control systems which are capable of providing an equivalent amount of con-

trol of hydrocarbon emissions may be used in lieu of the systems specified by the standard. An example of an equivalent control system is one which incinerates with an auxiliary fuel the hydrocarbon emissions from the storage tank before such emissions are released into the atmosphere.

The storage of crude oil and condensate at producing fields is specifically exempted from the standard. The proposed regulation had intended such an exemption by applying the standard only to storage vessels with capacities above 65,000 gallons. Industry representatives indicated that this action would exempt essentially all of the producing field storage, but later data showed that larger tanks are used in these locations. The specific exemption in the promulgated regulation better suits the intention. The standard now applies at capacities greater than 40,000 gallons, the size originally selected as being most consistent with existing State and local regulations before it was increased to exempt producing field storage. Producing field storage is exempt because the low level of emissions, the relatively small size of these tanks, and their commonly remote locations argue against justifying the switch from the bolted-construction, fixed-roof tanks in common use to the welded-construction, floating-roof tanks that would be required for new sources to comply with the standards.

The proposed standard required the use of conservation vents when petroleum liquids were stored at true vapor pressures less than 78 mm Hg. This requirement is deleted because, as commentators validly argued, certain stocks foul these vents, in cold weather the vents must be locked open or removed to prevent freezing, and the beneficial effects of such vents are minimal.

The monitoring and recordkeeping requirements are substantially reduced from those which were proposed. Over half of those who commented on this regulation argued that an unjustifiable burden was placed on owners and operators of remote tank farms, terminals, and marketing operations. EPA agrees. The basis for the proposed standard was the large, modern refinery which could have met the proposed requirements with little difficulty. The reduced requirements aid both enforcement officials and owners/operators by reducing paperwork without sacrificing the objectives of the regulation.

Some specific maintenance requirements were proposed but are deleted. Commentators pointed out that these requirements were not sufficiently explicit. A recent change to the General Provisions, subpart A, (see FEDERAL REGISTER of October 15, 1973, 38 FR 28564) requires that all affected facilities and emission control systems be operated and maintained in a manner consistent with good air pollution control practice for minimizing emissions. This provision will ensure the use of good maintenance practices for storage vessels, which was the intent of the proposed maintenance requirements.

#### SECONDARY LEAD SMELTERS AND REFINERIES

The promulgated standards limit emissions of particulate matter (1) from blast (cupola) and reverberatory furnaces to no more than 50 mg/dscm (0.022 gr/dscf) and to less than 20 percent opacity, and (2) from pot furnaces having charging capacities equal to or greater than 250 kilograms to less than 10 percent opacity.

These standards are the same as those proposed except that the 2-minutes-per-hour exemption is removed from both opacity standards. The general rationale for this change is presented above in the discussion of opacity. Two factors led to this change in the opacity standards: (1) The separately promulgated regulations that provide exemptions from the opacity standards during periods of startup, shutdown, and malfunction (see FEDERAL REGISTER of October 15, 1973, 38 FR 28564), and (2) the comments, reevaluation of data, and collection of new data and information which show that there is no basis for time exemptions in addition to those provided for startups, shutdowns, and malfunctions, and that the opacity standard is not more restrictive than the concentration standard.

Minor changes to the proposed version of the regulation have been made to clarify meanings and to exclude repetitive provisions and definitions which are now included in subpart A, General Provisions, and which are applicable to all new source performance standards.

#### SECONDARY BRASS AND BRONZE INGOT PRODUCTION PLANTS

The promulgated standards limit the emissions of particulate matter (1) from reverberatory furnaces having production capacities equal to or greater than 1,000 kg (2,205 lb) to no more than 50 mg/dscm (0.022 gr/dscf) and to less than 20 percent opacity, (2) from electric furnaces having capacities equal to or greater than 1,000 kg (2,205 lb) to less than 10 percent opacity, and (3) from blast (cupola) furnaces having capacities equal to or greater than 250 kg/hr (550 lb/hr) to less than 10 percent opacity.

These standards are the same as those proposed except that the opacity limit for emissions from the affected reverberatory furnaces is increased from less than 10 percent to less than 20 percent and the 2-minutes-per-hour exemption is removed from all three opacity standards. The general rationale for these changes is presented in the discussion of opacity above. The three factors which led to these changes are (1) the data and comments, summarized in Volume 3 of the background information document, which show, in the judgment of the Administrator, that the opacity standard proposed for reverberatory furnaces was too restrictive and that the promulgated opacity standard is not more restricted than the concentration standard, (2) the separately promulgated regulations which provide exemptions from opacity standards during periods of startup, shutdown, and malfunction (see FEDERAL REGISTER of October 15, 1973, 38 FR 28564), and (3) the comments, re-

evaluation of data, and collection of new data and information which show that there is no basis for additional time exemptions.

Minor changes to the proposed version of the regulation have been made to clarify meanings and to exclude repetitive provisions and definitions which are now included in subpart A, General Provisions, and which are applicable to all new source performance standards.

#### IRON AND STEEL PLANTS

The promulgated standards limit the emissions of particulate matter from basic oxygen process furnaces to no more than 50 mg/dscm (0.022 gr/dscf). This is the same concentration limit as was proposed. The opacity standard and the attendant monitoring requirement are not promulgated at this time. Sections of the regulation are reserved for the inclusion of these portions at a later date. Commentators pointed out the inappropriateness of the proposed opacity standard (10 percent opacity except for 2 minutes each hour) for this cyclic steel-making process. The separate promulgation of regulations which provide exemptions from opacity standards during periods of startup, shutdown, and malfunction (see FEDERAL REGISTER of October 15, 1973, 38 FR 28564) add another dimension to the problem, and new data show variations in opacity for reasons not yet well enough identified.

The promulgated regulation represents no substantial change to that proposed. Some wording is changed to clarify meanings and, as discussed under General Provisions above, several provisions and definitions are deleted from this subpart and added to subpart A, which applies to all new source performance standards, to avoid repetition.

#### SEWAGE TREATMENT PLANTS

The promulgated standards for sludge incinerators at municipal sewage treatment plants limit particulate emissions to no more than 0.65 g/kg dry sludge input (1.30 lb/ton dry sludge input) and to less than 20 percent opacity. The proposed standards would have limited emissions to a concentration of 70 mg/Nm<sup>3</sup> (0.031 gr/dscf) and to less than 10 percent opacity except for 2 minutes in any 1 hour. The level of control required by the standard remains the same, but the units are changed from a concentration to a mass basis because the determination of combustion air as opposed to dilution air for these facilities is particularly difficult and could lead to unacceptable degrees of error. The section on test methods is revised in accordance with the change of units for the standard.

A section is added specifying instrumentation and sampling access points needed to determine sludge charging rate. Determination of this rate is necessary as a result of the change of units for the standard. Flow measuring devices with an accuracy of  $\pm 5$  percent must be installed to determine either the mass or volume of the sludge charged to the incinerator, and access to the sludge charged must be provided so a well-

mixed representative grab sample of the sludge can be obtained.

The general rationale for the change in the opacity standard is presented in the discussion of opacity above. The three factors which led to this change are (1) the data, summarized in Volume 3 of the background information document, which, in the judgment of the Administrator, show that the proposed opacity standard was too restrictive and that the promulgated standard is not more restrictive than the mass standard, (2) the separately promulgated regulations which provide exemptions from opacity standards during periods of startup, shutdown, and malfunction (see FEDERAL REGISTER of October 15, 1973, 38 FR 28564), and (3) reevaluation of data and collection of new data and information which show that there is no basis for additional time exemptions.

Minor changes to the proposed version of the regulation have been made to clarify meanings and to exclude repetitive provisions and definitions which are now included in subpart A, General Provisions, and are applicable to all new source performance standards.

TEST METHODS

Test Methods 10 and 11 as proposed contained typographical errors that are now corrected in both text and equations. Some wording is changed to clarify meanings and procedures as well.

In Method 10, which is for determination of CO emissions, the term "grab sampling" is changed to "continuous sampling" to prevent confusion. The Orsat analyzer is deleted from the list of analytical equipment because a less complex method of analysis was judged sufficiently sensitive. For clarification, a sentence is added to the section on reagents requiring calibration gases to be certified by the manufacturer. Temperature of the silica gel is changed from 177°C (350°F) to 175°C (347°F) to be consistent with the emphasis on metric units as the primary units. A technique for determining the CO<sub>2</sub> content of the gas has been added to both the continuous and integrated sampling procedures. This technique may be used rather than the technique described in Method 3. Use of the latter technique was required in the proposed Method 10.

Method 11, which is for determination of H<sub>2</sub>S emissions, is modified to require five midge impingers rather than the proposed four. The fifth impinger contains hydrogen peroxide to remove sulfur dioxide as an interferant. A paragraph is added specifying the hydrogen peroxide solution to be used, and the procedure description is altered to include procedures specific to the fifth impinger. The term "iodine number flask" is changed to "iodine flask" to prevent confusion.

Dated: February 22, 1974.

RUSSELL E. TRAIN,  
Administrator.

Part 60, Chapter I, Title 40, Code of Federal Regulations, is amended by revising subpart A, by adding new subparts I, J, K, L, M, N, and O, and by adding Methods 10 and 11 to the Appendix, as follows:

Subpart A—General Provisions

- Sec. 60.2 Definitions.
- 60.3 Abbreviations.
- 60.4 Address.
- 60.6 Review of plans.
- 60.7 Notification and recordkeeping.
- 60.8 Performance tests.
- 60.12 Circumvention.

Subpart I—Standards of Performance for Asphalt Concrete Plants

- 60.90 Applicability and designation of affected facility.
- 60.91 Definitions.
- 60.92 Standard for particulate matter.
- 60.93 Test methods and procedures.

Subpart J—Standards of Performance for Petroleum Refineries

- 60.100 Applicability and designation of affected facility.
- 60.101 Definitions.
- 60.102 Standard for particulate matter.
- 60.103 Standard for carbon monoxide.
- 60.104 Standard for sulfur dioxide.
- 60.105 Emission monitoring.
- 60.106 Test methods and procedures.

Subpart K—Standards of Performance for Storage Vessels for Petroleum Liquids

- 60.110 Applicability and designation of affected facility.
- 60.111 Definitions.
- 60.112 Standard for hydrocarbons.
- 60.113 Monitoring of operations.

Subpart L—Standards of Performance for Secondary Lead Smelters

- 60.120 Applicability and designation of affected facility.
- 60.121 Definitions.
- 60.122 Standard for particulate matter.
- 60.123 Test methods and procedures.

Subpart M—Standards of Performance for Secondary Brass and Bronze Ingot Production Plants

- 60.130 Applicability and designation of affected facility.
- 60.131 Definitions.
- 60.132 Standard for particulate matter.
- 60.133 Test methods and procedures.

Subpart N—Standards of Performance for Iron and Steel Plants

- 60.140 Applicability and designation of affected facility.
- 60.141 Definitions.
- 60.142 Standard for particulate matter.
- 60.143 [Reserved]
- 60.144 Test methods and procedures.

Subpart O—Standards of Performance for Sewage Treatment Plants

- 60.150 Applicability and designation of affected facility.
- 60.151 Definitions.
- 60.152 Standard for particulate matter.
- 60.153 Monitoring of operations.
- 60.154 Test methods and procedures.

APPENDIX—TEST METHODS

- Method 10—Determination of carbon monoxide emissions from stationary sources.
- Method 11—Determination of hydrogen sulfide emissions from stationary sources.

AUTHORITY: Secs. 111, 114, Pub. L. 91-604 (42 U.S.C. 1857(c) (6) and (9)).

Subpart A—General Provisions

1. Section 60.2 is amended by revising paragraphs (i) and (l) and adding paragraphs (s), (t), (u), (v), and (w) as follows:

§ 60.2 Definitions.

(i) "Commenced" means, with respect to the definition of "new source" in section 111(a) (2) of the Act, that an owner or operator has undertaken a continuous program of construction or modification or that an owner or operator has entered into a contractual obligation to undertake and complete, within a reasonable time, a continuous program of construction or modification.

(l) "Standard conditions" means a temperature of 20°C (68°F) and a pressure of 760 mm of Hg (29.92 in. of Hg).

(s) "Reference method" means any method of sampling and analyzing for an air pollutant as described in the appendix to this part.

(t) "Equivalent method" means any method of sampling and analyzing for an air pollutant which have been demonstrated to the Administrator's satisfaction to have a consistent and quantitatively known relationship to the reference method, under specified conditions.

(u) "Alternative method" means any method of sampling and analyzing for an air pollutant which is not a reference or equivalent method but which has been demonstrated to the Administrator's satisfaction to, in specific cases, produce results adequate for his determination of compliance.

(v) "Particulate matter" means any finely divided solid or liquid material, other than uncombined water, as measured by method 5 of the appendix.

(w) "Run" means the net period of time during which an emission sample is collected. Unless otherwise specified, a run may be either intermittent or continuous within the limits of good engineering practice.

2. Section 60.3 is revised to read as follows:

§ 60.3 Abbreviations.

The abbreviations used in this part have the following meanings:

- A.S.T.M.—American Society for Testing and Materials
- Btu—British thermal unit
- °C—degree Celsius (centigrade)
- cal—calorie
- CdS—cadmium sulfide
- cfm—cubic feet per minute
- CO—carbon monoxide
- CO<sub>2</sub>—carbon dioxide
- dccm—dry cubic meter(s) at standard conditions
- dscf—dry cubic feet at standard conditions
- eq—equivalents
- °F—degree Fahrenheit
- g—gram(s)
- gal—gallon(s)
- g eq—gram equivalents
- gr—grain(s)

hr—hour(s)  
 HCl—hydrochloric acid  
 Hg—mercury  
 H<sub>2</sub>O—water  
 H<sub>2</sub>S—hydrogen sulfide  
 H<sub>2</sub>SO<sub>4</sub>—sulfuric acid  
 in.—inch(es)  
 °K—degree Kelvin  
 k—1,000  
 kg—kilogram(s)  
 l—liter(s)  
 lpm—liter(s) per minute  
 lb—pound(s)  
 m—meter(s)  
 meq—milliequivalent(s)  
 min—minute(s)  
 mg—milligram(s)  
 ml—milliliter(s)  
 mm—millimeter(s)  
 mol. wt.—molecular weight  
 mV—millivolt  
 N<sub>2</sub>—nitrogen  
 nm—nanometer(s)—10<sup>-9</sup> meter  
 NO—nitric oxide  
 NO<sub>2</sub>—nitrogen dioxide  
 NO<sub>x</sub>—nitrogen oxides  
 O<sub>2</sub>—oxygen  
 ppb—parts per billion  
 ppm—parts per million  
 psia—pounds per square inch absolute  
 °R—degree Rankine  
 s—at standard conditions  
 sec—second  
 SO<sub>2</sub>—sulfur dioxide  
 SO<sub>3</sub>—sulfur trioxide  
 µg—microgram(s)—10<sup>-6</sup> gram

3. Section 60.4 is revised to read as follows:

#### § 60.4 Address.

All requests, reports, applications, submittals, and other communications to the Administrator pursuant to this part shall be submitted in duplicate and addressed to the appropriate Regional Office of the Environmental Protection Agency, to the attention of the Director, Enforcement Division. The regional offices are as follows:

Region I (Connecticut, Maine, New Hampshire, Massachusetts, Rhode Island, Vermont), John F. Kennedy Federal Building, Boston, Massachusetts 02203.

Region II (New York, New Jersey, Puerto Rico, Virgin Islands), Federal Office Building, 26 Federal Plaza (Foley Square), New York, N.Y. 10007.

Region III (Delaware, District of Columbia, Pennsylvania, Maryland, Virginia, West Virginia), Curtis Building, Sixth and Walnut Streets, Philadelphia, Pennsylvania 19106.

Region IV (Alabama, Florida, Georgia, Mississippi, Kentucky, North Carolina, South Carolina, Tennessee), Suite 300, 1421 Peachtree Street, Atlanta, Georgia 30309.

Region V (Illinois, Indiana, Minnesota, Michigan, Ohio, Wisconsin), 1 North Wacker Drive, Chicago, Illinois 60606.

Region VI (Arkansas, Louisiana, New Mexico, Oklahoma, Texas), 1600 Patterson Street, Dallas, Texas 75201.

Region VII (Iowa, Kansas, Missouri, Nebraska), 1735 Baltimore Street, Kansas City, Missouri 64108.

Region VIII (Colorado, Montana, North Dakota, South Dakota, Utah, Wyoming), 916 Lincoln Towers, 1860 Lincoln Street, Denver, Colorado 80203.

Region IX (Arizona, California, Hawaii, Nevada, Guam, American Samoa), 100 California Street, San Francisco, California 94111.

Region X (Washington, Oregon, Idaho, Alaska), 1200 Sixth Avenue, Seattle, Washington 98101.

4. In § 60.6, paragraph (b) is revised to read as follows:

#### § 60.6 Review of plans.

(b) (1) A separate request shall be submitted for each construction or modification project.

(2) Each request shall identify the location of such project, and be accompanied by technical information describing the proposed nature, size, design, and method of operation of each affected facility involved in such project, including information on any requirement to be used for measurement or control of emissions.

5. In § 60.7 paragraph (d) is added as follows:

#### § 60.7 Notification and recordkeeping.

(d) Any owner or operator subject to the provisions of this part shall maintain a file of all measurements, including monitoring and performance testing measurements, and all other reports and records required by all applicable subparts. Any such measurements, reports and records shall be retained for at least 2 years following the date of such measurements, reports, and records.

6. Section 60.8 is amended by revising paragraphs (b) and (f) and by deleting in paragraph (d) the number "10" after the word "Administrator" and substituting the number "30." The revised paragraphs (b) and (f) read as follows:

#### § 60.8 Performance tests.

(b) Performance tests shall be conducted and data reduced in accordance with the test methods and procedures contained in each applicable subpart unless the Administrator (1) specifies or approves, in specific cases, the use of a reference method with minor changes in methodology, (2) approves the use of an equivalent method, (3) approves the use of an alternative method the results of which he has determined to be adequate for indicating whether a specific source is in compliance, or (4) waives the requirement for performance tests because the owner or operator of a source has demonstrated by other means to the Administrator's satisfaction that the affected facility is in compliance with the standard. Nothing in this paragraph shall be construed to abrogate the Administrator's authority to require testing under section 114 of the Act.

(f) Each performance test shall consist of three separate runs using the applicable test method. Each run shall be conducted for the time and under the conditions specified in the applicable standard. For the purpose of determining compliance with an applicable standard, the arithmetic means of results of the three runs shall apply. In the event that a sample is accidentally lost or conditions occur in which one of the three runs must be discontinued be-

cause of forced shutdown, failure of an irreplaceable portion of the sample train, extreme meteorological conditions, or other circumstances, beyond the owner or operator's control, compliance may, upon the Administrator's approval, be determined using the arithmetic mean of the results of the two other runs.

7. A new § 60.12 is added to subpart A as follows:

#### § 60.12 Circumvention.

No owner or operator subject to the provisions of this part shall build, erect, install, or use any article, machine, equipment or process, the use of which conceals an emission which would otherwise constitute a violation of an applicable standard. Such concealment includes, but is not limited to, the use of gaseous diluents to achieve compliance with an opacity standard or with a standard which is based on the concentration of a pollutant in the gases discharged to the atmosphere.

8. In Part 60, Subparts I, J, K, L, M, N, and O are added as follows:

#### Subpart I—Standards of Performance for Asphalt Concrete Plants

##### § 60.90 Applicability and designation of affected facility.

The affected facility to which the provisions of this subpart apply is each asphalt concrete plant. For the purpose of this subpart, an asphalt concrete plant is comprised only of any combination of the following: Dryers; systems for screening, handling, storing, and weighing hot aggregate; systems for loading, transferring, and storing mineral filler; systems for mixing asphalt concrete; and the loading, transfer, and storage systems associated with emission control systems.

##### § 60.91 Definitions.

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

(a) "Asphalt concrete plant" means any facility, as described in § 60.90, used to manufacture asphalt concrete by heating and drying aggregate and mixing with asphalt cements.

##### § 60.92 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere from any affected facility any gases which:

(1) Contain particulate matter in excess of 90 mg/dscm (0.04 gr/dscf).

(2) Exhibit 20 percent opacity, or greater. Where the presence of uncombined water is the only reason for failure to meet the requirements of this paragraph, such failure shall not be a violation of this section.

##### § 60.93 Test methods and procedures.

(a) The reference methods appended to this part, except as provided for in § 60.8(b), shall be used to determine

compliance with the standards prescribed in § 60.92 as follows:

(1) Method 5 for the concentration of particulate matter and the associated moisture content,

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for velocity and volumetric flow rate, and

(4) Method 3 for gas analysis.

(b) For Method 5, the sampling time for each run shall be at least 60 minutes and the sampling rate shall be at least 0.9 dscm/hr (0.53 dscf/min) except that shorter sampling times, when necessitated by process variables or other factors, may be approved by the Administrator.

**Subpart J—Standards of Performance for Petroleum Refineries**

**§ 60.100 Applicability and designation of affected facility.**

The provisions of this subpart are applicable to the following affected facilities in petroleum refineries: Fluid catalytic cracking unit catalyst regenerators, fluid catalytic cracking unit incinerator-waste heat boilers, and fuel gas combustion devices.

**§ 60.101 Definitions.**

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

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

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

(c) "Process gas" means any gas generated by a petroleum refinery process unit, except fuel gas and process upset gas as defined in this section.

(d) "Fuel gas" means any gas which is generated by a petroleum refinery process unit and which is combusted, including any gaseous mixture of natural gas and fuel gas which is combusted.

(e) "Process upset gas" means any gas generated by a petroleum refinery process unit as a result of start-up, shut-down, upset or malfunction.

(f) "Refinery process unit" means any segment of the petroleum refinery in which a specific processing operation is conducted.

(g) "Fuel gas combustion device" means any equipment, such as process heaters, boilers and flares used to combust fuel gas, but does not include fluid coking unit and fluid catalytic cracking unit incinerator-waste heat boilers or facilities in which gases are combusted to produce sulfur or sulfuric acid.

(h) "Coke burn-off" means the coke removed from the surface of the fluid catalytic cracking unit catalyst by combustion in the catalyst regenerator. The rate of coke burn-off is calculated by the formula specified in § 60.106.

**§ 60.102 Standard for particulate matter.**

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere from any fluid catalytic cracking unit catalyst regenerator or from any fluid catalytic cracking unit incinerator-waste heat boiler:

(1) Particulate matter in excess of 1.0 kg/1000 kg (1.0 lb/1000 lb) of coke burn-off in the catalyst regenerator.

(2) Gases exhibiting 30 percent opacity or greater, except for 3 minutes in any 1 hour. Where the presence of uncombined water is the only reason for failure to meet the requirements of this subparagraph, such failure shall not be a violation of this section.

(b) In those instances in which auxiliary liquid or solid fossil fuels are burned in the fluid catalytic cracking unit incinerator-waste heat boiler, particulate matter in excess of that permitted by paragraph (a) (1) of this section may be emitted to the atmosphere, except that the incremental rate of particulate emissions shall not exceed 0.18 g/million cal (0.10 lb/million Btu) of heat input attributable to such liquid or solid fuel.

**§ 60.103 Standard for carbon monoxide.**

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere from the fluid catalytic cracking unit catalyst regenerator any gases which contain carbon monoxide in excess of 0.050 percent by volume.

**§ 60.104 Standard for sulfur dioxide.**

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall burn in any fuel gas combustion device any fuel gas which contains H<sub>2</sub>S in excess of 230 mg/dscm (0.10 gr/dscf), except as provided in paragraph (b) of this section. The combustion of process upset gas in a flare, or the combustion in a flare of process gas, or fuel gas which is released to the flare as a result of relief valve leakage, is exempt from this paragraph.

(b) The owner or operator may elect to treat the gases resulting from the combustion of fuel gas in a manner which limits the release of SO<sub>2</sub> to the atmosphere if it is shown to the satisfaction of the Administrator that this prevents SO<sub>2</sub> emissions as effectively as compliance with the requirements of paragraph (a) of this section.

**§ 60.105 Emission monitoring.**

(a) The owner or operator of any petroleum refinery subject to the provisions of this subpart shall install, calibrate, maintain, and operate monitoring instruments as follows:

(1) A photoelectric or other type smoke detector and recorder to continuously monitor and record the opacity of gases discharged into the atmosphere from the fluid catalytic cracking unit catalyst regenerator.

(2) An instrument for continuously monitoring and recording the concentration of CO in gases discharged into the atmosphere from fluid catalytic cracking unit catalyst regenerators, except where the requirements of paragraph (a) (3) of this section are met.

(3) Instruments for continuously monitoring and recording firebox temperature and O<sub>2</sub> concentration in the exhaust gases from any incinerator-waste heat boiler which combusts the exhaust gases from a fluid catalytic cracking unit catalyst regenerator except where the requirements of paragraph (a) (2) of this section are met.

(4) An instrument for continuously monitoring and recording concentrations of H<sub>2</sub>S in fuel gases burned in any fuel gas combustion device, except where the requirements of § 60.104(b) are met. Fuel gas combustion devices having a common source of fuel gas may be monitored at one location if sampling at this location produces results representative of the H<sub>2</sub>S concentration in the fuel gas burned.

(5) An instrument for continuously monitoring and recording concentrations of SO<sub>2</sub> in the gases discharged into the atmosphere from the combustion of fuel gases except where the requirements of § 60.104(a) are met.

(b) Instruments and sampling systems installed and used pursuant to this section shall meet specifications prescribed by the Administrator and each instrument shall be calibrated in accordance with the method prescribed by the manufacturer of such instrument. The instruments shall be subjected to the manufacturer's recommended zero adjustment and calibration procedures at least once per 24-hour operating period unless the manufacturer specifies or recommends calibration at shorter intervals, in which case such specifications or recommendations shall be followed.

(c) The average coke burn-off rate (thousands of kilogram/hr) and hours of operation for any fluid catalytic cracking unit catalyst regenerator subject to § 60.102 or 60.103 shall be recorded daily.

(d) For any fluid catalytic cracking unit catalyst regenerator which is subject to § 60.102 and which utilizes an incinerator-waste heat boiler to combust the exhaust gases from the catalyst regenerator, the owner or operator shall record daily the rate of combustion of liquid or solid fossil fuels (liters/hr or kilograms/hr) and the hours of operation during which liquid or solid fossil fuels are combusted in the incinerator-waste heat boiler.

(e) For the purpose of reports pursuant to § 60.7(c), periods of excess emissions that shall be reported are defined as follows:

(1) *Opacity.* All hourly periods in which there are four or more 1-minute periods during which the average opacity

of the gases discharged into the atmosphere from any fluid catalytic cracking unit catalyst regenerator subject to § 60.102 exceeds 30 percent.

(2) *Carbon monoxide.* All hourly periods during which the average carbon monoxide concentration in the gases discharged into the atmosphere from any fluid catalytic cracking unit catalyst regenerator subject to § 60.103 exceeds 0.050 percent by volume; or any hourly period in which O<sub>2</sub> concentration and firebox temperature measurements indicate that the average concentration of CO in the gases discharged into the atmosphere exceeds 0.050 percent by volume for sources which combust the exhaust gases from any fluid catalytic cracking unit catalyst regenerator subject to § 60.103 in an incinerator-waste heat boiler and for which the owner or operator elects to monitor in accordance with § 60.105(a) (3).

(3) *Hydrogen sulfide.* All hourly periods during which the average hydrogen sulfide content of any fuel gas combusted in any fuel gas combustion device subject to § 60.104 exceeds 230 mg/dscm (0.10 gr/dscf) except where the requirements of § 60.104(b) are met.

(4) *Sulfur dioxide.* All hourly periods during which the average sulfur dioxide emissions discharged into the atmosphere from any fuel gas combustion device subject to § 60.104 exceed the level specified in § 60.104(b), except where the requirements of § 60.104(a) are met.

#### § 60.106 Test methods and procedures.

(a) For the purpose of determining compliance with § 60.102(a) (1), the following reference methods and calculation procedures shall be used:

(1) For gases released to the atmosphere from the fluid catalytic cracking unit catalyst regenerator:

(i) Method 5 for the concentration of particulate matter and moisture content,

(ii) Method 1 for sample and velocity traverses, and

(iii) Method 2 for velocity and volumetric flow rate.

(2) For Method 5, the sampling time for each run shall be at least 60 minutes and the sampling rate shall be at least 0.015 dscm/min (0.53 dscf/min), except that shorter sampling times may be approved by the Administrator when process variables or other factors preclude sampling for at least 60 minutes.

(3) For exhaust gases from the fluid catalytic cracking unit catalyst regenerator prior to the emission control system: the integrated sample techniques of Method 3 and Method 4 for gas analysis and moisture content, respectively; Method 1 for velocity traverses; and Method 2 for velocity and volumetric flow rate.

(4) Coke burn-off rate shall be determined by the following formula:

$$R_o = 0.2982 Q_{RE} (\%CO_2 + \%CO) + 2.088 Q_{RA} - 0.0094 Q_{RW} \left( \frac{\%CO}{2} + \%CO_2 + \%O_2 \right) \text{ (Metric Units)}$$

or

$$R_o = 0.0188 Q_{RE} (\%CO_2 + \%CO) + 0.1303 Q_{RA} - 0.0062 Q_{RW} \left( \frac{\%CO}{2} + \%CO_2 + \%O_2 \right) \text{ (English Units)}$$

where:

R<sub>o</sub> = coke burn-off rate, kg/hr (English units: lb/hr).

0.2982 = metric units material balance factor divided by 100, kg-min/hr-m<sup>3</sup>.

0.0188 = English units material balance factor divided by 100, lb-min/hr-ft<sup>3</sup>.

Q<sub>RE</sub> = fluid catalytic cracking unit catalyst regenerator exhaust gas flow rate before entering the emission control system, as determined by method 2, dscm/min (English units: dscf/min).

%CO<sub>2</sub> = percent carbon dioxide by volume, dry basis, as determined by Method 3.

%CO = percent carbon monoxide by volume, dry basis, as determined by Method 3.

2.088 = metric units material balance factor divided by 100, kg-min/hr-m<sup>3</sup>.

0.1303 = English units material balance factor divided by 100, lb-min/hr-ft<sup>3</sup>.

Q<sub>RA</sub> = air rate to fluid catalytic cracking unit catalyst regenerator, as determined from fluid catalytic cracking unit control room instrumentation, dscm/min (English units: dscf/min).

0.0094 = metric units material balance factor divided by 100, kg-min/hr-m<sup>3</sup>.

0.0062 = English units material balance factor divided by 100, lb-min/hr-ft<sup>3</sup>.

(5) Particulate emissions shall be determined by the following equation:

$$R_E = (60 \times 10^{-6}) Q_{RV} C_p \text{ (Metric Units)}$$

or

$$R_E = (8.67 \times 10^{-3}) Q_{RV} C_p \text{ (English Units)}$$

where:

R<sub>E</sub> = particulate emission rate, kg/hr (English units: lb/hr).

60 × 10<sup>-6</sup> = metric units conversion factor, min-kg/hr-mg.

8.67 × 10<sup>-3</sup> = English units conversion factor, min-lb/hr-gr.

Q<sub>RV</sub> = volumetric flow rate of gases discharged into the atmosphere from the fluid catalytic cracking unit catalyst regenerator following the emission control system, as determined by Method 2, dscm/min (English units: dscf/min).

C<sub>p</sub> = particulate emission concentration discharged into the atmosphere, as determined by Method 5, mg/dscm (English units: gr/dscf).

(6) For each run, emissions expressed in kg/1000 kg (English units: lb/1000 lb) of coke burn-off in the catalyst regenerator shall be determined by the following equation:

$$R_o = 1000 \frac{R_E}{R_o} \text{ (Metric or English Units)}$$

where:

R<sub>o</sub> = particulate emission rate, kg/1000 kg (English units: lb/1000 lb) of coke burn-off in the fluid catalytic cracking unit catalyst regenerator.

1000 = conversion factor, kg to 1000 kg (English units: lb to 1000 lb).

R<sub>E</sub> = particulate emission rate, kg/hr (English units: lb/hr).

R<sub>o</sub> = coke burn-off rate, kg/hr (English units: lb/hr).

(7) In those instances in which auxiliary liquid or solid fossil fuels are burned in an incinerator-waste heat boiler, the rate of particulate matter emissions permitted under § 60.102(b) must be determined. Auxiliary fuel heat input, expressed in millions of cal/hr (English units: Millions of Btu/hr) shall be calculated for each run by fuel flow rate measurement and analysis of the liquid or solid auxiliary fossil fuels. For each run, the rate of particulate emissions permitted under § 60.102(b) shall be calculated from the following equation:

$$R_o = 1.0 + \frac{0.18 H}{R_o} \text{ (Metric Units)}$$

or

$$R_o = 1.0 + \frac{0.10 H}{R_o} \text{ (English Units)}$$

where:

R<sub>o</sub> = allowable particulate emission rate, kg/1000 kg (English units: lb/1000 lb) of coke burn-off in the fluid catalytic cracking unit catalyst regenerator.

1.0 = emission standard, 1.0 kg/1000 kg (English units: 1.0 lb/1000 lb) of coke burn-off in the fluid catalytic cracking unit catalyst regenerator.

0.18 = metric units maximum allowable incremental rate of particulate emissions, g/million cal.

0.10 = English units maximum allowable incremental rate of particulate emissions, lb/million Btu.

H = heat input from solid or liquid fossil fuel, million cal/hr (English units: million Btu/hr).

R<sub>o</sub> = coke burn-off rate, kg/hr (English units: lb/hr).

(b) For the purpose of determining compliance with § 60.103, the integrated sample technique of Method 10 shall be used. The sample shall be extracted at a rate proportional to the gas velocity at a sampling point near the centroid of the duct. The sampling time shall not be less than 60 minutes.

(c) For the purpose of determining compliance with § 60.104(a), Method 11 shall be used. When refinery fuel gas lines are operating at pressures substantially above atmospheric, the gases sam-

pled must be introduced into the sampling train at approximately atmospheric pressure. This may be accomplished with a flow control valve. If the line pressure is high enough to operate the sampling train without a vacuum pump, the pump may be eliminated from the sampling train. The sample shall be drawn from a point near the centroid of the fuel gas line. The minimum sampling time shall be 10 minutes and the minimum sampling volume 0.01 dscm (0.35 dscf) for each sample. The arithmetic average of

two samples shall constitute one run. Samples shall be taken at approximately 1-hour intervals. For most fuel gases, sample times exceeding 20 minutes may result in depletion of the collecting solution, although fuel gases containing low concentrations of hydrogen sulfide may necessitate sampling for longer periods of time.

(d) Method 6 shall be used for determining concentration of SO<sub>2</sub> in determining compliance with § 60.104(b), except that H<sub>2</sub>S concentration of the fuel gas may be determined instead. Method 1 shall be used for velocity traverses and Method 2 for determining velocity and volumetric flow rate. The sampling site for determining SO<sub>2</sub> concentration by Method 6 shall be the same as for determining volumetric flow rate by Method 2. The sampling point in the duct for determining SO<sub>2</sub> concentration by Method 6 shall be at the centroid of the cross section if the cross sectional area is less than 5 m<sup>2</sup> (54 ft<sup>2</sup>) or at a point no closer to the walls than 1 m (39 inches) if the cross sectional area is 5 m<sup>2</sup> or more and the centroid is more than one meter from the wall. The sample shall be extracted at a rate proportional to the gas velocity at the sampling point. The minimum sampling time shall be 10 minutes and the minimum sampling volume 0.01 dscm (0.35 dscf) for each sample. The arithmetic average of two samples shall constitute one run. Samples shall be taken at approximately 1-hour intervals.

**Subpart K—Standards of Performance for Storage Vessels for Petroleum Liquids**

**§ 60.110 Applicability and designation of affected facility.**

(a) Except as provided in § 60.110(b), the affected facility to which this subpart applies is each storage vessel for petroleum liquids which has a storage capacity greater than 151,412 liters (40,000 gallons).

(b) This subpart does not apply to storage vessels for the crude petroleum or condensate stored, processed, and/or treated at a drilling and production facility prior to custody transfer.

**§ 60.111 Definitions.**

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

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

(1) Pressure vessels which are designed to operate in excess of 15 pounds per square inch gauge without emissions to the atmosphere except under emergency conditions,

(2) Subsurface caverns or porous rock reservoirs, or

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

(b) "Petroleum liquids" means crude petroleum, condensate, and any finished

or intermediate products manufactured in a petroleum refinery but does not mean Number 2 through Number 6 fuel oils as specified in ASTM-D-396-69, gas turbine fuel oils Numbers 2-GT through 4-GT as specified in ASTM-D-2880-71, or diesel fuel oils Numbers 2-D and 4-D as specified in ASTM-D-975-68.

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

(d) "Crude petroleum" means a naturally occurring mixture which consists of hydrocarbons and/or sulfur, nitrogen and/or oxygen derivatives of hydrocarbons and which is a liquid at standard conditions.

(e) "Hydrocarbon" means any organic compound consisting predominantly of

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

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

(h) "Drilling and production facility" means all drilling and servicing equipment, wells, flow lines, separators, equipment, gathering lines, and auxiliary non-transportation-related equipment used in the production of crude petroleum but does not include natural gasoline plants.

(i) "True vapor pressure" means the equilibrium partial pressure exerted by a petroleum liquid as determined in accordance with methods described in American Petroleum Institute Bulletin 2517, Evaporation Loss from Floating Roof Tanks, 1962.

(j) "Floating roof" means a storage vessel cover consisting of a double deck, pontoon single deck, internal floating cover or covered floating roof, which rests upon and is supported by the petroleum liquid being contained, and is equipped with a closure seal or seals to close the space between the roof edge and tank wall.

(k) "Vapor recovery system" means a vapor gathering system capable of collecting all hydrocarbon vapors and gases discharged from the storage vessel and a vapor disposal system capable of processing such hydrocarbon vapors and gases so as to prevent their emission to the atmosphere.

(l) "Reid vapor pressure" is the absolute vapor pressure of volatile crude oil and volatile non-viscous petroleum liquids, except liquified petroleum gases, as determined by ASTM-D-323-58 (re-approved 1968).

**§ 61.112 Standard for hydrocarbons.**

(a) The owner or operator of any storage vessel to which this subpart applies shall store petroleum liquids as follows:

(1) If the true vapor pressure of the petroleum liquid, as stored, is equal to or greater than 78 mm Hg (1.5 psia) but not greater than 570 mm Hg (11.1 psia), the storage vessel shall be equipped with a floating roof, a vapor recovery system, or their equivalents.

(2) If the true vapor pressure of the petroleum liquid as stored is greater than 570 mm Hg (11.1 psia), the storage vessel shall be equipped with a vapor recovery system or its equivalent.

**§ 60.113 Monitoring of operations.**

(a) The owner or operator of any storage vessel to which this subpart applies shall for each such storage vessel maintain a file of each type of petroleum liquid stored, of the typical Reid vapor pressure of each type of petroleum liquid stored, and of the dates of storage. Dates on which the storage vessel is empty shall be shown.

(b) The owner or operator of any storage vessel to which this subpart applies shall for each such storage vessel determine and record the average monthly storage temperature and true vapor pressure of the petroleum liquid stored at such temperature if:

(1) The petroleum liquid has a true vapor pressure, as stored, greater than 26 mm Hg (0.5 psia) but less than 78 mm Hg (1.5 psia) and is stored in a storage vessel other than one equipped with a floating roof, a vapor recovery system or their equivalents; or

(2) The petroleum liquid has a true vapor pressure, as stored, greater than 470 mm Hg (9.1 psia) and is stored in a storage vessel other than one equipped with a vapor recovery system or its equivalent.

(c) The average monthly storage temperature is an arithmetic average calculated for each calendar month, or portion thereof if storage is for less than a month, from bulk liquid storage temperatures determined at least once every 7 days.

(d) The true vapor pressure shall be determined by the procedures in API Bulletin 2517. This procedure is dependent upon determination of the storage temperature and the Reid vapor pressure, which requires sampling of the petroleum liquids in the storage vessels. Unless the Administrator requires in specific cases that the stored petroleum liquid be sampled, the true vapor pressure may be determined by using the average monthly storage temperature and the typical Reid vapor pressure. For those liquids for which certified specifications limiting the Reid vapor pressure exist, that Reid vapor pressure may be used. For other liquids, supporting analytical data must be made available on request to the Administrator when typical Reid vapor pressure is used.

**Subpart L—Standards of Performance for Secondary Lead Smelters**

**§ 60.120 Applicability and designation of affected facility.**

The provisions of this subpart are applicable to the following affected facil-

ties in secondary lead smelters: Pot furnaces of more than 250 kg (550 lb) charging capacity, blast (cupola) furnaces, and reverberatory furnaces.

#### § 60.121 Definitions.

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

(a) "Reverberatory furnace" includes the following types of reverberatory furnaces: stationary, rotating, rocking, and tilting.

(b) "Secondary lead smelter" means any facility producing lead from a lead-bearing scrap material by smelting to the metallic form.

(c) "Lead" means elemental lead or alloys in which the predominant component is lead.

#### § 60.122 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere from a blast (cupola) or reverberatory furnace any gases which:

(1) Contain particulate matter in excess of 50 mg/dscm (0.022 gr/dscf).

(2) Exhibit 20 percent opacity or greater.

(b) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere from any pot furnace any gases which exhibit 10 percent opacity or greater.

(c) Where the presence of uncombined water is the only reason for failure to meet the requirements of paragraphs (a) (2) or (b) of this section, such failure shall not be a violation of this section.

#### § 60.123 Test methods and procedures.

(a) The reference methods appended to this part, except as provided for in § 60.8 (b), shall be used to determine compliance with the standards prescribed in § 60.122 as follows:

(1) Method 5 for the concentration of particulate matter and the associated moisture content,

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for velocity and volumetric flow rate, and

(4) Method 3 for gas analysis.

(b) For method 5, the sampling time for each run shall be at least 60 minutes and the sampling rate shall be at least 0.9 dscm/hr (0.53 dscf/min) except that shorter sampling times, when necessitated by process variables or other factors, may be approved by the Administrator. Particulate sampling shall be conducted during representative periods of furnace operation, including charging and tapping.

### Subpart M—Standards of Performance for Secondary Brass and Bronze Ingot Production Plants

#### § 60.130 Applicability and designation of affected facility.

The provisions of this subpart are applicable to the following affected facilities in secondary brass or bronze ingot production plants: Reverberatory and electric furnaces of 1,000 kg (2,205 lb) or greater production capacity and blast (cupola) furnaces of 250 kg/hr (550 lb/hr) or greater production capacity.

#### § 60.131 Definitions.

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

(a) "Brass or bronze" means any metal alloy containing copper as its predominant constituent, and lesser amounts of zinc, tin, lead, or other metals.

(b) "Reverberatory furnace" includes the following types of reverberatory furnaces: Stationary, rotating, rocking, and tilting.

(c) "Electric furnace" means any furnace which uses electricity to produce over 50 percent of the heat required in the production of refined brass or bronze.

(d) "Blast furnace" means any furnace used to recover metal from slag.

#### § 60.132 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere from a reverberatory furnace any gases which:

(1) Contain particulate matter in excess of 50 mg/dscm (0.022 gr/dscf).

(2) Exhibit 20 percent opacity or greater.

(b) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere from any blast (cupola) or electric furnace any gases which exhibit 10 percent opacity or greater.

(c) Where the presence of uncombined water is the only reason for failure to meet the requirements of paragraphs (a) (2) or (b) of this section, such failure shall not be a violation of this section.

#### § 60.133 Test methods and procedures.

(a) The reference methods appended to this part, except as provided for in § 60.8(b), shall be used to determine compliance with the standards prescribed in § 60.132 as follows:

(1) Method 5 for the concentration of particulate matter and the associated moisture content.

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for velocity and volumetric flow rate, and

(4) Method 3 for gas analysis.

(b) For Method 5, the sampling time for each run shall be at least 120 minutes and the sampling rate shall be at least 0.9 dscm/hr (0.53 dscf/min) except that shorter sampling times, when necessitated by process variables or other factors, may be approved by the Administrator. Particulate matter sampling shall be conducted during representative periods of charging and refining, but not during pouring of the heat.

### Subpart N—Standards of Performance for Iron and Steel Plants

#### § 60.140 Applicability and designation of affected facility.

The affected facility to which the provisions of this subpart apply is each basic oxygen process furnace.

#### § 60.141 Definitions.

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

(a) "Basic oxygen process furnace" (BOPF) means any furnace producing steel by charging scrap steel, hot metal, and flux materials into a vessel and introducing a high volume of an oxygen-rich gas.

(b) "Steel production cycle" means the operations required to produce each batch of steel and includes the following major functions: Scrap charging, preheating (when used), hot metal charging, primary oxygen blowing, additional oxygen blowing (when used), and tapping.

#### § 60.142 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere from any affected facility any gases which:

(1) Contain particulate matter in excess of 50 mg/dscm (0.022 gr/dscf).

(2) [Reserved.]

#### § 60.143 [Reserved]

#### § 60.144 Test methods and procedures.

(a) The reference methods appended to this part, except as provided for in § 60.8(b), shall be used to determine compliance with the standards prescribed in § 60.142 as follows:

(1) Method 5 for concentration of particulate matter and associated moisture content,

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for volumetric flow rate, and

(4) Method 3 for gas analysis.

(b) For Method 5, the sampling for each run shall continue for an integral number of cycles with total duration of at least 60 minutes. The sampling rate shall be at least 0.9 dscm/hr (0.53 dscf/min) except that shorter sampling times,

when necessitated by process variables or other factors, may be approved by the Administrator. A cycle shall start at the beginning of either the scrap preheat or the oxygen blow and shall terminate immediately prior to tapping.

**Subpart O—Standards of Performance for Sewage Treatment Plants**

**§ 60.150 Applicability and designation of affected facility.**

The affected facility to which the provisions of this subpart apply is each incinerator which burns the sludge produced by municipal sewage treatment facilities.

**§ 60.151 Definitions.**

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

**§ 60.152 Standard for particulate matter.**

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator of any sewage sludge incinerator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere of:

(1) Particulate matter at a rate in excess of 0.65 g/kg dry sludge input (1.30 lb/ton dry sludge input).

(2) Any gases which exhibit 20 percent opacity or greater. Where the presence of uncombined water is the only reason for failure to meet the requirements of this paragraph, such failure shall not be a violation of this section.

**§ 60.153 Monitoring of operations.**

(a) The owner or operator of any sludge incinerator subject to the provisions of this subpart shall:

(1) Install, calibrate, maintain, and operate a flow measuring device which can be used to determine either the mass or volume of sludge charged to the incinerator. The flow measuring device shall have an accuracy of ±5 percent over its operating range.

(2) Provide access to the sludge charged so that a well-mixed representative grab sample of the sludge can be obtained.

**§ 60.154 Test Methods and Procedures.**

(a) The reference methods appended to this part, except as provided for in § 60.8(b), shall be used to determine compliance with the standards prescribed in § 60.152 as follows:

(1) Method 5 for concentration of particulate matter and associated moisture content,

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for volumetric flow rate, and

(4) Method 3 for gas analysis.

(b) For Method 5, the sampling time for each run shall be at least 60 minutes and the sampling rate shall be at least 0.015 dscm/min (0.53 dscf/min), except that shorter sampling times, when necessitated by process variables or other factors, may be approved by the Administrator.

(c) Dry sludge charging rate shall be determined as follows:

(1) Determine the mass ( $S_M$ ) or volume ( $S_V$ ) of sludge charged to the incinerator during each run using a flow measuring device meeting the requirements of § 60.153(a) (1). If total input during a run is measured by a flow measuring device, such readings shall be used. Otherwise, record the flow measuring device readings at 5-minute intervals during a run. Determine the quantity charged during each interval by averaging the flow rates at the beginning and end of the interval and then multiplying the average for each interval by the time for each interval. Then add the quantity for each interval to determine the total quantity charged during the entire run, ( $S_M$ ) or ( $S_V$ ).

(2) Collect samples of the sludge charged to the incinerator in non-porous collecting jars at the beginning of each run and at approximately 1-hour intervals thereafter until the test ends, and

determine for each sample the dry sludge content (total solids residue) in accordance with "224 G. Method for Solid and Semisolid Samples," *Standard Methods for the Examination of Water and Wastewater*, Thirteenth Edition, American Public Health Association, Inc., New York, N.Y., 1971, pp. 539-41, except that:

(i) Evaporating dishes shall be ignited to at least 103°C rather than the 550°C specified in step 3(a) (1).

(ii) Determination of volatile residue, step 3(b) may be deleted.

(iii) The quantity of dry sludge per unit sludge charged shall be determined in terms of either  $R_{DV}$  (metric units: mg dry sludge/liter sludge charged or English units: lb/ft<sup>3</sup>) or  $R_{DM}$  (metric units: mg dry sludge/mg sludge charged or English units: lb/lb).

(3) Determine the quantity of dry sludge per unit sludge charged in terms of either  $R_{DV}$  or  $R_{DM}$ .

(i) If the volume of sludge charged is used:

$$S_D = (60 \times 10^{-3}) \frac{R_{DV} S_V}{T} \text{ (Metric Units)}$$

or

$$S_D = (8.021) \frac{R_{DV} S_V}{T} \text{ (English Units)}$$

where:

- $S_D$  = average dry sludge charging rate during the run, kg/hr (English units: lb/hr);
- $R_{DV}$  = average quantity of dry sludge per unit volume of sludge charged to the incinerator, mg/l (English units: lb/ft<sup>3</sup>);
- $S_V$  = sludge charged to the incinerator during the run, m<sup>3</sup> (English units: gal);
- $T$  = duration of run, min (English units: min);
- $60 \times 10^{-3}$  = metric units conversion factor, 1 kg-min/m<sup>3</sup>-mg-lr.
- $8.021$  = English units conversion factor, ft<sup>3</sup>-min/gal-hr.

(ii) If the mass of sludge charged is used:

$$S_D = (60) \frac{R_{DM} S_M}{T} \text{ (Metric or English Units)}$$

where:

- $S_D$  = average dry sludge charging rate during the run, kg/hr (English units: lb/hr);
- $R_{DM}$  = average ratio of quantity of dry sludge to quantity of sludge charged to the incinerator, mg/mg (English units: lb/lb);
- $S_M$  = sludge charged during the run, kg (English units: lb);
- $T$  = duration of run, min (Metric or English units);
- $60$  = conversion factor, min/hr (Metric or English units).

(d) Particulate emission rate shall be determined by:

$$e_w = e_s Q_s \text{ (Metric or English Units)}$$

where:

- $e_w$  = particulate matter mass emissions, mg/hr (English units: lb/hr);
- $e_s$  = particulate matter concentration, mg/m<sup>3</sup> (English units: lb/1000 ft<sup>3</sup>);
- $Q_s$  = volumetric stack gas flow rate, dscm/hr (English units: dscf/hr).  $Q_s$  and  $e_s$  shall be determined using Methods 2 and 5, respectively.

(e) Compliance with § 60.152(a) shall be determined as follows:

$$C_{21} = (10^{-3}) \frac{C_{21}^*}{S_D} \text{ (Metric Units)}$$

or

$$C_{21} = (2000) \frac{C_{21}^*}{S_D} \text{ (English Units)}$$

where:

- $C_{21}$  = particulate emission discharge, g/kg dry sludge (English units: lb/ton dry sludge);
- $10^{-3}$  = Metric conversion factor, g/mg;
- $2000$  = English conversion factor, lb/ton.

9. Methods 10 and 11 are added to the appendix as follows:

**METHOD 10—DETERMINATION OF CARBON MONOXIDE EMISSIONS FROM STATIONARY SOURCES**

**1. Principle and Applicability.**

1.1 *Principle.* An integrated or continuous gas sample is extracted from a sampling point and analyzed for carbon monoxide (CO) content using a Luft-type nondispersive infrared analyzer (NDIR) or equivalent.

1.2 *Applicability.* This method is applicable for the determination of carbon monoxide emissions from stationary sources only when specified by the test procedures for determining compliance with new source

performance standards. The test procedure will indicate whether a continuous or an integrated sample is to be used.

**2. Range and sensitivity.**

2.1 *Range.* 0 to 1,000 ppm.

2.2 *Sensitivity.* Minimum detectable concentration is 20 ppm for a 0 to 1,000 ppm span.

3. *Interferences.* Any substance having a strong absorption of infrared energy will interfere to some extent. For example, discrimination ratios for water (H<sub>2</sub>O) and carbon dioxide (CO<sub>2</sub>) are 3.5 percent H<sub>2</sub>O per 7 ppm CO and 10 percent CO<sub>2</sub> per 10 ppm CO, respectively, for devices measuring in the 1,500 to 3,000 ppm range. For devices meas-

uring in the 0 to 100 ppm range, interference ratios can be as high as 3.5 percent H<sub>2</sub>O per 25 ppm CO and 10 percent CO<sub>2</sub> per 50 ppm CO. The use of silica gel and ascarite traps will alleviate the major interference problems. The measured gas volume must be corrected if these traps are used.

4. Precision and accuracy.

4.1 Precision. The precision of most NDIR analyzers is approximately ±2 percent of span.

4.2 Accuracy. The accuracy of most NDIR analyzers is approximately ±5 percent of span after calibration.

5. Apparatus.

5.1 Continuous sample (Figure 10-1).

5.1.1 Probe. Stainless steel or sheathed Pyrex<sup>1</sup> glass, equipped with a filter to remove particulate matter.

5.1.2 Air-cooled condenser or equivalent. To remove any excess moisture.

5.2 Integrated sample (Figure 10-2).

5.2.1 Probe. Stainless steel or sheathed Pyrex glass, equipped with a filter to remove particulate matter.

5.2.2 Air-cooled condenser or equivalent. To remove any excess moisture.

5.2.3 Valve. Needle valve, or equivalent, to adjust flow rate.

5.2.4 Pump. Leak-free diaphragm type, or equivalent, to transport gas.

5.2.5 Rate meter. Rotameter, or equivalent, to measure a flow range from 0 to 1.0 liter per min. (0.035 cfm).

5.2.6 Flexible bag. Tedlar, or equivalent, with a capacity of 60 to 90 liters (2 to 3 ft<sup>3</sup>). Leak-test the bag in the laboratory before using by evacuating bag with a pump followed by a dry gas meter. When evacuation is complete, there should be no flow through the meter.

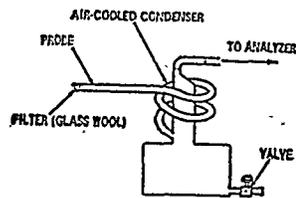


Figure 10-1. Continuous sampling train.

5.3.1 Carbon monoxide analyzer. Nondispersive infrared spectrometer, or equivalent. This instrument should be demonstrated, preferably by the manufacturer, to meet or exceed manufacturer's specifications and those described in this method.

5.3.2 Drying tube. To contain approximately 200 g of silica gel.

5.3.3 Calibration gas. Refer to paragraph 6.1.

5.3.4 Filter. As recommended by NDIR manufacturer.

5.3.5 CO<sub>2</sub> removal tube. To contain approximately 500 g of ascarite.

5.3.6 Ice water bath. For ascarite and silica gel tubes.

5.3.7 Valve. Needle valve, or equivalent, to adjust flow rate.

5.3.8 Rate meter. Rotameter or equivalent to measure gas flow rate of 0 to 1.0 liter per min. (0.035 cfm) through NDIR.

5.3.9 Recorder (optional). To provide permanent record of NDIR readings.

6. Reagents.

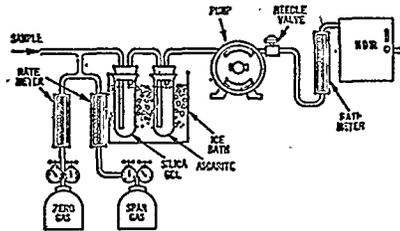


Figure 10-3. Analytical equipment.

6.1 Calibration gases. Known concentration of CO in nitrogen (N<sub>2</sub>) for instrument span, prepurified grade of N<sub>2</sub> for zero; and two additional concentrations corresponding approximately to 60 percent and 30 percent span. The span concentration shall not exceed 1.5 times the applicable source performance standard. The calibration gases shall be certified by the manufacturer to be within ±2 percent of the specified concentration.

6.2 Silica gel. Indicating type, 6 to 16 mesh, dried at 175° C (347° F) for 2 hours.

6.3 Ascarite. Commercially available.

7. Procedure.

7.1 Sampling.

7.1.1 Continuous sampling. Set up the equipment as shown in Figure 10-1 making sure all connections are leak free. Place the probe in the stack at a sampling point and purge the sampling line. Connect the analyzer and begin drawing sample into the analyzer. Allow 5 minutes for the system to stabilize, then record the analyzer reading as required by the test procedure. (See ¶ 7.3 and 8). CO<sub>2</sub> content of the gas may be determined by using the Method 3 integrated sample procedure (36 FR 24886), or by weighing the ascarite CO<sub>2</sub> removal tube and computing CO<sub>2</sub> concentration from the gas volume sampled and the weight gain of the tube.

7.1.2 Integrated sampling. Evacuate the flexible bag. Set up the equipment as shown in Figure 10-2 with the bag disconnected. Place the probe in the stack and purge the sampling line. Connect the bag, making sure that all connections are leak free. Sample at a rate proportional to the stack velocity. CO<sub>2</sub> content of the gas may be determined by using the Method 3 integrated sample procedures (36 FR 24886), or by weighing the ascarite CO<sub>2</sub> removal tube and computing CO<sub>2</sub> concentration from the gas volume sampled and the weight gain of the tube.

7.2 CO Analysis. Assemble the apparatus as shown in Figure 10-3, calibrate the instrument, and perform other required operations as described in paragraph 8. Purge analyzer with N<sub>2</sub> prior to introduction of each sample. Direct the sample stream through the instrument for the test period, recording the readings. Check the zero and span again after the test to assure that any drift or malfunction is detected. Record the sample data on Table 10-1.

8. Calibration. Assemble the apparatus according to Figure 10-3. Generally an instrument requires a warm-up period before stability is obtained. Follow the manufacturer's instructions for specific procedure. Allow a minimum time of one hour for warm-up. During this time check the sample conditioning apparatus, i.e., filter, condenser, drying tube, and CO<sub>2</sub> removal tube, to ensure that each component is in good operating condition. Zero and calibrate the instrument according to the manufacturer's procedures using, respectively, nitrogen and the calibration gases.

TABLE 10-1.—Field data

Location _____		Comments: _____ _____ _____
Test _____		
Date _____		
Operator _____		
Clock time	Rotameter setting, liters per minute (cubic feet per minute)	

9. Calculation—Concentration of carbon monoxide. Calculate the concentration of carbon monoxide in the stack using equation 10-1.

$$C_{CO_{stack}} = C_{CO_{NDIR}}(1 - F_{CO_2}) \quad \text{equation 10-1}$$

where:

C<sub>CO<sub>stack</sub></sub> = concentration of CO in stack, ppm by volume (dry basis).

C<sub>CO<sub>NDIR</sub></sub> = concentration of CO measured by NDIR analyzer, ppm by volume (dry basis).

F<sub>CO<sub>2</sub></sub> = volume fraction of CO<sub>2</sub> in sample, i.e., percent CO<sub>2</sub> from Orsat analysis divided by 100.

5.2.7 Pitot tube. Type S, or equivalent, attached to the probe so that the sampling rate can be regulated proportional to the stack gas velocity when velocity is varying with the time or a sample traverse is conducted.

5.3 Analysis (Figure 10-3).

<sup>1</sup> Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

10. Bibliography.

- 10.1 McElroy, Frank, The Intertech NDIR-CO Analyzer, Presented at 11th Methods Conference on Air Pollution, University of California, Berkeley, Calif., April 1, 1970.
- 10.2 Jacobs, M. B., et al., Continuous Determination of Carbon Monoxide and Hydrocarbons in Air by a Modified Infrared Analyzer, J. Air Pollution Control Association, 9(2):110-114, August 1959.
- 10.3 MSA LIRA Infrared Gas and Liquid

- Analyzer Instruction Book, Mine Safety Appliances Co., Technical Products Division, Pittsburgh, Pa.
- 10.4 Models 215A, 315A, and 415A Infrared Analyzers, Beckman Instruments, Inc., Beckman Instructions 1635-B, Fullerton, Calif., October 1967.
- 10.5 Continuous CO Monitoring System, Model A5611, Intertech Corp., Princeton, N.J.
- 10.6 UNOR Infrared Gas Analyzers, Bendix Corp., Roncoverte, West Virginia.

- 2.3.2 Burette—One 50 ml.
- 2.3.2 Flask—125-ml conical.
- 3. Reagents.
- 3.1 Sampling.

3.1.1 Absorbing solution—Cadmium hydroxide (Cd(OH)<sub>2</sub>)—Mix 4.3 g cadmium sulfate hydrate (3 CdSO<sub>4</sub>·8H<sub>2</sub>O) and 0.3 g of sodium hydroxide (NaOH) in 1 liter of distilled water (H<sub>2</sub>O). Mix well.

Note: The cadmium hydroxide formed in this mixture will precipitate as a white suspension. Therefore, this solution must be thoroughly mixed before using to ensure an even distribution of the cadmium hydroxide.

3.1.2 Hydrogen peroxide, 3 percent—Dilute 30 percent hydrogen peroxide to 3 percent as needed. Prepare fresh daily;

3.2 Sample recovery.

3.2.1 Hydrochloric acid solution (HCl), 10 percent by weight—Mix 230 ml of concentrated HCl (specific gravity 1.19) and 770 ml of distilled H<sub>2</sub>O.

3.2.2 Iodine solution, 0.1 N—Dissolve 24 g potassium iodide (KI) in 30 ml of distilled H<sub>2</sub>O in a 1-liter graduated cylinder. Weigh 12.7 g of resublimed iodine (I<sub>2</sub>) into a weighing bottle and add to the potassium iodide solution. Shake the mixture until the iodine is completely dissolved. Slowly dilute the solution to 1 liter with distilled H<sub>2</sub>O, with swirling. Filter the solution, if cloudy, and store in a brown glass-stoppered bottle.

3.2.3 Standard iodine solution, 0.01 N—Dilute 100 ml of the 0.1 N iodine solution in a volumetric flask to 1 liter with distilled water.

Standardize daily as follows: Pipette 25 ml of the 0.01 N iodine solution into a 125-ml conical flask. Titrate with standard 0.01 N thiosulfate solution (see paragraph 3.3.2) until the solution is a light yellow. Add a few drops of the starch solution and continue titrating until the blue color just disappears. From the results of this titration, calculate the exact normality of the iodine solution (see paragraph 5.1).

3.2.4 Distilled, deionized water.

3.3 Analysis.

3.3.1 Sodium thiosulfate solution, standard 0.1 N—For each liter of solution, dissolve 24.8 g of sodium thiosulfate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>·5H<sub>2</sub>O) in distilled water and add 0.01 g of anhydrous sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) and 0.4 ml of chloroform (CHCl<sub>3</sub>) to stabilize. Mix thoroughly by shaking or by aerating with nitrogen for approximately 15 minutes, and store in a glass-stoppered glass bottle.

Standardize frequently as follows: Weigh into a 500-ml volumetric flask about 2 g of potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>) weighed to the nearest milligram and dilute to the 500-ml mark with distilled H<sub>2</sub>O. Use dichromate which has been crystallized from distilled water and oven-dried at 162°C to 199°C (360°F to 390°F). Dissolve approximately 3 g of potassium iodide (KI) in 50 ml of distilled water in a glass-stoppered, 500-ml conical flask, then add 5 ml of 20-percent hydrochloric acid solution. Pipette 50 ml of the dichromate solution into this mixture. Gently swirl the solution once and allow it to stand in the dark for 5 minutes. Dilute the solution with 100 to 200 ml of distilled water, washing down the sides of the flask with part of the water. Swirl the solution slowly and titrate with the thiosulfate solution until the solution is light yellow. Add 4 ml of starch solution and continue with a slow titration with the thiosulfate until the bright blue color has disappeared and only the pale green color of the chromic ion remains. From this titration, calculate the exact normality of the sodium thiosulfate solution (see paragraph 5.2).

3.3.2 Sodium thiosulfate solution, standard 0.01 N—Pipette 100 ml of the standard 0.1 N thiosulfate solution into a volumetric flask and dilute to one liter with distilled water.

ADDENDA

A. Performance Specifications for NDIR-Carbon Monoxide Analyzers.

Range (minimum)-----	0-1000ppm.
Output (minimum)-----	0-10mV.
Minimum detectable sensitivity-----	20 ppm.
Rise time, 90 percent (maximum)-----	30 seconds.
Fall time, 90 percent (maximum)-----	30 seconds.
Zero drift (maximum)-----	10% in 8 hours.
Span drift (maximum)-----	10% in 8 hours.
Precision (minimum)-----	± 2% of full scale.
Noise (maximum)-----	± 1% of full scale.
Linearity (maximum deviation)-----	2% of full scale.
Interference rejection ratio-----	CO <sub>2</sub> —1000 to 1, H <sub>2</sub> O—500 to 1.

B. Definitions of Performance Specifications.

Range—The minimum and maximum measurement limits.

Output—Electrical signal which is proportional to the measurement; intended for connection to readout or data processing devices. Usually expressed as millivolts or milliamperes full scale at a given impedance.

Full scale—The maximum measuring limit for a given range.

Minimum detectable sensitivity—The smallest amount of input concentration that can be detected as the concentration approaches zero.

Accuracy—The degree of agreement between a measured value and the true value; usually expressed as ± percent of full scale.

Time to 90 percent response—The time interval from a step change in the input concentration at the instrument inlet to a reading of 90 percent of the ultimate recorded concentration.

Rise Time (90 percent)—The interval between initial response time and time to 90 percent response after a step increase in the inlet concentration.

Fall Time (90 percent)—The interval between initial response time and time to 90 percent response after a step decrease in the inlet concentration.

Zero Drift—The change in instrument output over a stated time period, usually 24 hours, of unadjusted continuous operation when the input concentration is zero; usually expressed as percent full scale.

Span Drift—The change in instrument output over a stated time period, usually 24 hours, of unadjusted continuous operation when the input concentration is a stated upscale value; usually expressed as percent full scale.

Precision—The degree of agreement between repeated measurements of the same concentration, expressed as the average deviation of the single results from the mean.

Noise—Spontaneous deviations from a mean output not caused by input concentration changes.

Linearity—The maximum deviation between an actual instrument reading and the reading predicted by a straight line drawn between upper and lower calibration points.

METHOD 11—DETERMINATION OF HYDROGEN SULFIDE EMISSIONS FROM STATIONARY SOURCES

1. Principle and applicability.

1.1 Principle. Hydrogen sulfide (H<sub>2</sub>S) is collected from the source in a series of midget

impingers and reacted with alkaline cadmium hydroxide [Cd(OH)<sub>2</sub>] to form cadmium sulfide (CdS). The precipitated CdS is then dissolved in hydrochloric acid and absorbed in a known volume of iodine solution. The iodine consumed is a measure of the H<sub>2</sub>S content of the gas. An impinger containing hydrogen peroxide is included to remove SO<sub>2</sub> as an interfering species.

1.2 Applicability. This method is applicable for the determination of hydrogen sulfide emissions from stationary sources only when specified by the test procedures for determining compliance with the new source performance standards.

2. Apparatus.

2.1 Sampling train.

2.1.1 Sampling line—6- to 7-mm (¼-inch) Teflon tubing to connect sampling train to sampling valve, with provisions for heating to prevent condensation. A pressure reducing valve prior to the Teflon sampling line may be required depending on sampling stream pressure.

2.1.2 Impingers—Five midget impingers, each with 30-ml capacity, or equivalent.

2.1.3 Ice bath container—To maintain absorbing solution at a constant temperature.

2.1.4 Silica gel drying tube—To protect pump and dry gas meter.

2.1.5 Needle valve, or equivalent—Stainless steel or other corrosion resistant material, to adjust gas flow rate.

2.1.6 Pump—Leak free, diaphragm type, or equivalent, to transport gas. (Not required if sampling stream under positive pressure.)

2.1.7 Dry gas meter—Sufficiently accurate to measure sample volume to within 1 percent.

2.1.8 Rate meter—Rotameter, or equivalent, to measure a flow rate of 0 to 3 liters per minute (0.1 ft<sup>3</sup>/min).

2.1.9 Graduated cylinder—25 ml.

2.1.10 Barometer—To measure atmospheric pressure within ±2.5 mm (0.1 in.) Hg.

2.2 Sample Recovery.

2.2.1 Sample container—500-ml glass-stoppered iodine flask.

2.2.2 Pipette—50-ml volumetric type.

2.2.3 Beakers—250 ml.

2.2.4 Wash bottle—Glass.

2.3 Analysis.

2.3.1 Flask—500-ml glass-stoppered iodine flask.

<sup>1</sup> Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

3.3.3 *Starch indicator solution*—Suspend 10 g of soluble starch in 100 ml of distilled water and add 15 g of potassium hydroxide pellets. Stir until dissolved, dilute with 900 ml of distilled water, and let stand 1 hour. Neutralize the alkali with concentrated hydrochloric acid, using an indicator paper similar to Alkacid test ribbon, then add 2 ml of glacial acetic acid as a preservative.

Test for decomposition by titrating 4 ml of starch solution in 200 ml of distilled water with 0.01 N iodine solution. If more than 4 drops of the 0.01 N iodine solution are required to obtain the blue color, make up a fresh starch solution.

4. Procedure.

4.1 Sampling.

4.1.1 Assemble the sampling train as shown in Figure 11-1, connecting the five midgelet impingers in series. Place 15 ml of 3 percent hydrogen peroxide in the first impinger. Place 15 ml of the absorbing solution in each of the next three impingers, leaving the fifth dry. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger at about 20°C (70°F), or less.

4.1.2 Purge the connecting line between the sampling valve and the first impinger. Connect the sample line to the train. Record the initial reading on the dry gas meter as shown in Table 11-1.

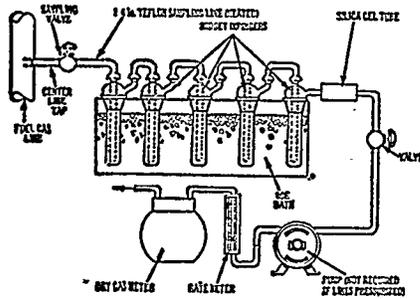


Figure 11-1. H<sub>2</sub>S sampling train.

TABLE 11-1.—Field data

Location	Comments:
Test	
Date	
Operator	
Barometric pressure	

Clock time	Gas volume through meter (V <sub>m</sub> ), liters (cubic feet)	Rotameter setting, Lpm (cubic feet per minute)	Meter temperature, °C (°F)

4.1.3 Open the flow control valve and adjust the sampling rate to 1.13 liters per minute (0.04 cfm). Read the meter temperature and record on Table 11-1.

4.1.4 Continue sampling a minimum of 10 minutes. If the yellow color of cadmium sulfide is visible in the third impinger, analysis should confirm that the applicable standard has been exceeded. At the end of the sample time, close the flow control valve and read the final meter volume and temperature.

4.1.5 Disconnect the impinger train from the sampling line. Purge the train with clean ambient air for 15 minutes to ensure that all H<sub>2</sub>S is removed from the hydrogen peroxide. Cap the open ends and move to the sample clean-up area.

4.2 Sample recovery.

4.2.1 Pipette 50 ml of 0.01 N iodine solution

into a 250-ml beaker. Add 50 ml of 10 percent HCl to the solution. Mix well.

4.2.2 Discard the contents of the hydrogen peroxide impinger. Carefully transfer the contents of the remaining four impingers to a 500-ml iodine flask.

4.2.3 Rinse the four absorbing impingers and connecting glassware with three portions of the acidified iodine solution. Use the entire 100 ml of acidified iodine for this purpose. Immediately after pouring the acidified iodine into an impinger, stopper it and shake for a few moments before transferring the rinse to the iodine flask. Do not transfer any rinse portion from one impinger to another; transfer it directly to the iodine flask. Once acidified iodine solution has been poured into any glassware containing cadmium sulfide sample, the container must be tightly stoppered at all times except when adding more solution, and this must be done as quickly and carefully as possible. After adding any acidified iodine solution to the iodine flask, allow a few minutes for absorption of the H<sub>2</sub>S into the iodine before adding any further rinses.

5. Calculations.

5.1 Normality of the standard iodine solution.

$$N_I = \frac{N_T V_T}{V_I} \tag{equation 11-1}$$

where:

- N<sub>I</sub> = normality of iodine, g-eq/liter.
- V<sub>I</sub> = volume of iodine used, ml.
- N<sub>T</sub> = normality of sodium thiosulfate, g-eq/liter.
- V<sub>T</sub> = volume of sodium thiosulfate used, ml.

5.2 Normality of the standard thiosulfate solution.

$$N_T = 2.04 \frac{W}{V_T} \tag{equation 11-2}$$

where:

- W = weight of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> used, g.
- V<sub>T</sub> = volume of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> used, ml.
- N<sub>T</sub> = normality of standard thiosulfate solution, g-eq/liter.
- 2.04 = conversion factor

$$= \frac{(6 \text{ eq } I_2/\text{mole } K_2Cr_2O_7) (1,000 \text{ ml/l})}{(294.2 \text{ g } K_2Cr_2O_7/\text{mole}) (10 \text{ aliquot factor})}$$

5.3 Dry gas volume. Correct the sample volume measured by the dry gas meter to standard conditions [21°C(70°F) and 760 mm (29.92 inches) Hg] by using equation 11-3.

$$V_{mstd} = V_m \left( \frac{T_{std}}{T_m} \right) \left( \frac{P_{bar}}{P_{std}} \right) \tag{equation 11-3}$$

where:

- V<sub>mstd</sub> = volume at standard conditions of gas sample through the dry gas meter, standard liters (scf).
- V<sub>m</sub> = volume of gas sample through the dry gas meter (meter conditions), liters (cu. ft.).
- T<sub>std</sub> = absolute temperature at standard conditions, 294°K (530°R).
- T<sub>m</sub> = average dry gas meter temperature, °K (°R).
- P<sub>bar</sub> = barometric pressure at the orifice meter, mm Hg (in. Hg).
- P<sub>std</sub> = absolute pressure at standard conditions, 760 mm Hg (29.92 in. Hg).

5.4 Concentration of H<sub>2</sub>S.—Calculate the concentration of H<sub>2</sub>S in the gas stream at standard conditions using equation 11-4:

$$C_{H_2S} = \frac{K[(V_I N_I - V_T N_T)_{sample} - (V_I N_I - V_T N_T)_{blank}]}{V_{mstd}}$$

where (metric units):

- C<sub>H<sub>2</sub>S</sub> = concentration of H<sub>2</sub>S at standard conditions, mg/dscm
- K = conversion factor = 17.0 × 10<sup>3</sup>

$$= \frac{(34.07 \text{ g/mole } H_2S)(1,000 \text{ l/m}^3)(1,000 \text{ mg/g})}{(1,000 \text{ ml/l})(2H_2S \text{ eq/mole})}$$

- V<sub>I</sub> = volume of standard iodine solution, ml.
- N<sub>I</sub> = normality of standard iodine solution, g-eq/liter.
- V<sub>T</sub> = volume of standard sodium thiosulfate solution, ml.
- N<sub>T</sub> = normality of standard sodium thiosulfate solution, g-eq/liter.
- V<sub>mstd</sub> = dry gas volume at standard conditions, liters.

where (English units):

$$K=0.263=\frac{17.0(15.43 \text{ gr/g})}{(1,000 \text{ l/m}^3)}$$

$V_{std}$  = scf.

$C_{H_2S}$  = gr/dscf.

6. References.

6.1 Determination of Hydrogen Sulfide, Ammoniacal Cadmium Chloride Method, API Method 772-54. In: Manual on Disposal of Refinery Wastes, Vol. V: Sampling and Analysis of Waste Gases and Particulate Matter, American Petroleum Institute, Washington, D.C., 1954.

6.2 Tentative Method for Determination of Hydrogen Sulfide and Mercaptan Sulfur in Natural Gas, Natural Gas Processors Association, Tulsa, Oklahoma, NGPA Publication No. 2265-65, 1965.

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