

§ 80.1360

§ 80.1360 Who is liable for violations under the gasoline benzene program?

(a) The following persons are liable for violations of prohibited acts:

(1) Any refiner or importer that violates § 80.1358(a) is liable for the violation.

(2) Any person that causes another party to violate § 80.1358(a) is liable for a violation of § 80.1358(b).

(3) Any parent corporation is liable for any violations of this subpart that are committed by any of its wholly-owned subsidiaries.

(4) Each partner to a joint venture, or each owner of a facility owned by two or more owners, is jointly and severally liable for any violation of this subpart that occurs at the joint venture facility or a facility that is owned by the joint owners, or a facility that is committed by the joint venture operation or any of the joint owners of the facility.

(b) Any person who violates § 80.1358 is liable for the violation.

§ 80.1361 What penalties apply under the gasoline benzene program?

(a) Any person liable for a violation under § 80.1360 is subject to civil penalties as specified in sections 205 and 211(d) of the Clean Air Act for every day of each such violation and the amount of economic benefit or savings resulting from each violation.

(b) Any person liable under § 80.1358(a) and (b) for a violation of the applicable benzene standards or causing another person to violate the requirements during any averaging period, is subject to a separate day of violation for each and every day in the averaging period. Any person liable under § 80.1360(b) for a failure to fulfill any requirement of credit generation, transfer, use, banking, or deficit carry-forward correction is subject to a separate violation for each and every day in the averaging period in which invalid credits are generated, banked, transferred or used.

(c) Any person liable under § 80.1360(b) for failure to meet, or causing a failure to meet, a provision of this subpart is liable for a separate day of violation for each and every day such provision remains unfulfilled.

40 CFR Ch. I (7-1-07 Edition)

FOREIGN REFINERS

§ 80.1363 What are the additional requirements under this subpart for gasoline produced at foreign refineries?

(a) Definitions.

(1) A *foreign refinery* is a refinery that is located outside the United States, the Commonwealth of Puerto Rico, the Virgin Islands, Guam, American Samoa, and the Commonwealth of the Northern Mariana Islands (collectively referred to in this section as "the United States").

(2) A *foreign refiner* is a person that meets the definition of refiner under § 80.2(i) for a foreign refinery.

(3) *Benzene-FRGAS* means gasoline produced at a foreign refinery that has been assigned an individual refinery benzene baseline under § 80.1285, has been approved as a small refiner under § 80.1340, or has been granted temporary relief under § 80.1335, and that is imported into the United States.

(4) *Non-Benzene-FRGAS* means

(i) Gasoline meeting any of the conditions specified in paragraph (a)(3) of this section that is not imported into the United States.

(ii) Gasoline meeting any of the conditions specified in paragraph (a)(3) of this section during a year when the foreign refiner has opted to not participate in the Benzene-FRGAS program under paragraph (c)(3) of this section.

(iii) Gasoline produced at a foreign refinery that has not been assigned an individual refinery benzene baseline under § 80.1285, or that has not been approved as a small refiner under § 80.1340, or that has not been granted temporary relief under § 80.1335.

(5) *Certified Benzene-FRGAS* means Benzene-FRGAS the foreign refiner intends to include in the foreign refinery's benzene compliance calculations under § 80.1240 or credit calculations under § 80.1275 and does include in these calculations when reported to EPA.

(6) *Non-Certified Benzene-FRGAS* means Benzene-FRGAS that is not Certified Benzene-FRGAS.

(b) *Baseline for Early Credits*. For any foreign refiner to obtain approval under the benzene foreign refiner program of this subpart for any refinery in order to generate early credits under § 80.1275, it must apply for approval

under the applicable provisions of this subpart.

(1) The refiner shall follow the procedures specified in §§ 80.1280 and 80.1285 to establish a baseline of the volume of gasoline that was produced at the refinery and imported into the United States during the applicable years.

(2) In making determinations for foreign refinery baselines EPA will consider all information supplied by a foreign refiner, and in addition may rely on any and all appropriate assumptions necessary to make such determinations.

(3) Where a foreign refiner submits a petition that is incomplete or inadequate to establish an accurate baseline, and the refiner fails to correct this deficiency after a request for more information, EPA will not assign an individual refinery baseline.

(c) *General requirements for Benzene-FRGAS foreign refiners.* A foreign refiner of a refinery that is approved under the benzene foreign refiner program of this subpart must designate each batch of gasoline produced at the foreign refinery that is exported to the United States as either Certified Benzene-FRGAS or as Non-Certified Benzene-FRGAS, except as provided in paragraph (c)(3) of this section.

(1) In the case of Certified Benzene-FRGAS, the foreign refiner must meet all requirements that apply to refiners under this subpart.

(2) In the case of Non-Certified Benzene-FRGAS, the foreign refiner shall meet all the following requirements:

(i) The designation requirements in this section;

(ii) The recordkeeping requirements in this section and in § 80.1350;

(iii) The reporting requirements in this section and in §§ 80.1352 and 80.1354;

(iv) The product transfer document requirements in this section;

(v) The prohibitions in this section and in § 80.1358; and

(vi) The independent audit requirements in this section and in § 80.1356.

(3)(i) Any foreign refiner that generates early benzene credits under § 80.1275 shall designate all Benzene-FRGAS as Certified Benzene-FRGAS for any year that such credits are generated.

(ii) Any foreign refiner that has been approved to produce gasoline subject to the benzene foreign refiner program for a foreign refinery under this subpart may elect to classify no gasoline imported into the United States as Benzene-FRGAS provided the foreign refiner notifies EPA of the election no later than November 1 preceding the beginning of the next compliance period.

(iii) An election under paragraph (c)(3)(ii) of this section shall be for a 12 month compliance period and apply to all gasoline that is produced by the foreign refinery that is imported into the United States, and shall remain in effect for each succeeding year unless and until the foreign refiner notifies EPA of the termination of the election. The change in election shall take effect at the beginning of the next annual compliance period.

(d) *Designation, product transfer documents, and foreign refiner certification.*

(1) Any foreign refiner of a foreign refinery that has been approved by EPA to produce gasoline subject to the benzene foreign refiner program must designate each batch of Benzene-FRGAS as such at the time the gasoline is produced, unless the refiner has elected to classify no gasoline exported to the United States as Benzene-FRGAS under paragraph (c)(3) of this section.

(2) On each occasion when any person transfers custody or title to any Benzene-FRGAS prior to its being imported into the United States, it must include the following information as part of the product transfer document information:

(i) Designation of the gasoline as Certified Benzene-FRGAS or as Non-Certified Benzene-FRGAS; and

(ii) The name and EPA refinery registration number of the refinery where the Benzene-FRGAS was produced.

(3) On each occasion when Benzene-FRGAS is loaded onto a vessel or other transportation mode for transport to the United States, the foreign refiner shall prepare a certification for each batch of the Benzene-FRGAS that meets the following requirements.

(i) The certification shall include the report of the independent third party under paragraph (f) of this section, and the following additional information:

(A) The name and EPA registration number of the refinery that produced the Benzene-FRGAS;

(B) The identification of the gasoline as Certified Benzene-FRGAS or Non-Certified Benzene-FRGAS;

(C) The volume of Benzene-FRGAS being transported, in gallons;

(D) In the case of Certified Benzene-FRGAS:

(1) The benzene content as determined under paragraph (f) of this section, and the applicable designations stated in paragraph (d)(2)(i) of this section; and

(2) A declaration that the Benzene-FRGAS is being included in the applicable compliance calculations required by EPA under this subpart.

(ii) The certification shall be made part of the product transfer documents for the Benzene-FRGAS.

(e) *Transfers of Benzene-FRGAS to non-United States markets.* The foreign refiner is responsible to ensure that all gasoline classified as Benzene-FRGAS is imported into the United States. A foreign refiner may remove the Benzene-FRGAS classification, and the gasoline need not be imported into the United States, but only if:

(1) The foreign refiner excludes:

(i) The volume of gasoline from the refinery's compliance report under § 80.1354; and

(ii) In the case of Certified Benzene-FRGAS, the volume of the gasoline from the compliance report under § 80.1354.

(2) The foreign refiner obtains sufficient evidence in the form of documentation that the gasoline was not imported into the United States.

(f) *Load port independent sampling, testing and refinery identification.*

(1) On each occasion that Benzene-FRGAS is loaded onto a vessel for transport to the United States a foreign refiner shall have an independent third party:

(i) Inspect the vessel prior to loading and determine the volume of any tank bottoms;

(ii) Determine the volume of Benzene-FRGAS loaded onto the vessel (exclusive of any tank bottoms before loading);

(iii) Obtain the EPA-assigned registration number of the foreign refinery;

(iv) Determine the name and country of registration of the vessel used to transport the Benzene-FRGAS to the United States; and

(v) Determine the date and time the vessel departs the port serving the foreign refinery.

(2) On each occasion that Certified Benzene-FRGAS is loaded onto a vessel for transport to the United States a foreign refiner shall have an independent third party:

(i) Collect a representative sample of the Certified Benzene-FRGAS from each vessel compartment subsequent to loading on the vessel and prior to departure of the vessel from the port serving the foreign refinery;

(ii) Determine the benzene content value for each compartment using the methodology as specified in § 80.46(e) by one of the following:

(A) The third party analyzing each sample; or

(B) The third party observing the foreign refiner analyze the sample;

(iii) Review original documents that reflect movement and storage of the Certified Benzene-FRGAS from the refinery to the load port, and from this review determine:

(A) The refinery at which the Benzene-FRGAS was produced; and

(B) That the Benzene-FRGAS remained segregated from:

(1) Non-Benzene-FRGAS and Non-Certified Benzene-FRGAS; and

(2) Other Certified Benzene-FRGAS produced at a different refinery.

(3) The independent third party shall submit a report:

(i) To the foreign refiner containing the information required under paragraphs (f)(1) and (f)(2) of this section, to accompany the product transfer documents for the vessel; and

(ii) To the Administrator containing the information required under paragraphs (f)(1) and (f)(2) of this section, within thirty days following the date of the independent third party's inspection. This report shall include a description of the method used to determine the identity of the refinery at which the gasoline was produced, assurance that the gasoline remained

segregated as specified in paragraph (n)(1) of this section, and a description of the gasoline's movement and storage between production at the source refinery and vessel loading.

(4) The independent third party must:

(i) Be approved in advance by EPA, based on a demonstration of ability to perform the procedures required in this paragraph (f);

(ii) Be independent under the criteria specified in § 80.65(f)(2)(iii); and

(iii) Sign a commitment that contains the provisions specified in paragraph (i) of this section with regard to activities, facilities and documents relevant to compliance with the requirements of this paragraph (f).

(g) *Comparison of load port and port of entry testing.* (1)(i) Any foreign refiner and any United States importer of Certified Benzene-FRGAS shall compare the results from the load port testing under paragraph (f) of this section, with the port of entry testing as reported under paragraph (o) of this section, for the volume of gasoline and the benzene content value; except as specified in paragraph (g)(1)(ii) of this section.

(ii) Where a vessel transporting Certified Benzene-FRGAS off loads this gasoline at more than one United States port of entry, and the conditions of paragraph (g)(2)(i) of this section are met at the first United States port of entry, the requirements of paragraph (g)(2) of this section do not apply at subsequent ports of entry if the United States importer obtains a certification from the vessel owner that meets the requirements of paragraph (s) of this section, that the vessel has not loaded any gasoline or blendstock between the first United States port of entry and the subsequent port of entry.

(2)(i) The requirements of this paragraph (g)(2) apply if—

(A) The temperature-corrected volumes determined at the port of entry and at the load port differ by more than one percent; or

(B) The benzene content value determined at the port of entry is higher than the benzene content value determined at the load port, and the amount of this difference is greater than the reproducibility amount specified for the port of entry test result by the Amer-

ican Society of Testing and Materials (ASTM) for the test method specified at § 80.46(e).

(ii) The United States importer and the foreign refiner shall treat the gasoline as Non-Certified Benzene-FRGAS, and the foreign refiner shall exclude the gasoline volume from its gasoline volumes calculations and benzene standard designations under this subpart.

(h) *Attest requirements.* Refiners, for each annual compliance period, must arrange to have an attest engagement performed of the underlying documentation that forms the basis of any report required under this subpart. The attest engagement must comply with the procedures and requirements that apply to refiners under §§ 80.125 through 80.130, § 80.1356, and other applicable attest engagement provisions, and must be submitted to the Administrator of EPA for the prior annual compliance period within the time period required under § 80.130. The following additional procedures shall be carried out for any foreign refiner of Benzene-FRGAS.

(1) The inventory reconciliation analysis under § 80.128(b) and the tender analysis under § 80.128(c) shall include Non-Benzene-FRGAS.

(2) Obtain separate listings of all tenders of Certified Benzene-FRGAS and of Non-Certified Benzene-FRGAS, and obtain separate listings of Certified Benzene-FRGAS based on whether it is small refiner gasoline, gasoline produced through the use of credits, or other applicable designation under this subpart. Agree the total volume of tenders from the listings to the gasoline inventory reconciliation analysis in § 80.128(b), and to the volumes determined by the third party under paragraph (f)(1) of this section.

(3) For each tender under paragraph (h)(2) of this section, where the gasoline is loaded onto a marine vessel, report as a finding the name and country of registration of each vessel, and the volumes of Benzene-FRGAS loaded onto each vessel.

(4) Select a sample from the list of vessels identified in paragraph (h)(3) of this section used to transport Certified Benzene-FRGAS, in accordance with the guidelines in § 80.127, and for each vessel selected perform the following:

(i) Obtain the report of the independent third party, under paragraph (f) of this section, and of the United States importer under paragraph (o) of this section.

(A) Agree the information in these reports with regard to vessel identification, gasoline volumes and benzene content test results.

(B) Identify, and report as a finding, each occasion the load port and port of entry benzene content and volume results differ by more than the amounts allowed in paragraph (g) of this section, and determine whether the foreign refiner adjusted its refinery calculations as required in paragraph (g) of this section.

(ii) Obtain the documents used by the independent third party to determine transportation and storage of the Certified Benzene-FRGAS from the refinery to the load port, under paragraph (f) of this section. Obtain tank activity records for any storage tank where the Certified Benzene-FRGAS is stored, and pipeline activity records for any pipeline used to transport the Certified Benzene-FRGAS, prior to being loaded onto the vessel. Use these records to determine whether the Certified Benzene-FRGAS was produced at the refinery that is the subject of the attest engagement, and whether the Certified Benzene-FRGAS was mixed with any Non-Certified Benzene-FRGAS, Non-Benzene-FRGAS, or any Certified Benzene-FRGAS produced at a different refinery.

(5) Select a sample from the list of vessels identified in paragraph (h)(3) of this section used to transport Certified and Non-Certified Benzene-FRGAS, in accordance with the guidelines in §80.127, and for each vessel selected perform the following:

(i) Obtain a commercial document of general circulation that lists vessel arrivals and departures, and that includes the port and date of departure of the vessel, and the port of entry and date of arrival of the vessel.

(ii) Agree the vessel's departure and arrival locations and dates from the independent third party and United States importer reports to the information contained in the commercial document.

(6) Obtain separate listings of all tenders of Non-Benzene-FRGAS, and perform the following:

(i) Agree the total volume and benzene content of tenders from the listings to the gasoline inventory reconciliation analysis in §80.128(b).

(ii) Obtain a separate listing of the tenders under this paragraph (h)(6) where the gasoline is loaded onto a marine vessel. Select a sample from this listing in accordance with the guidelines in §80.127, and obtain a commercial document of general circulation that lists vessel arrivals and departures, and that includes the port and date of departure and the ports and dates where the gasoline was off loaded for the selected vessels. Determine and report as a finding the country where the gasoline was off loaded for each vessel selected.

(7) In order to complete the requirements of this paragraph (h) an auditor shall:

(i) Be independent of the foreign refiner;

(ii) Be licensed as a Certified Public Accountant in the United States and a citizen of the United States, or be approved in advance by EPA based on a demonstration of ability to perform the procedures required in §§80.125 through 80.130 and this paragraph (h); and

(iii) Sign a commitment that contains the provisions specified in paragraph (i) of this section with regard to activities and documents relevant to compliance with the requirements of §§80.125 through 80.130 and this paragraph (h).

(i) *Foreign refiner commitments.* Any foreign refiner shall commit to and comply with the provisions contained in this paragraph (i) as a condition to being approved as a foreign refiner under this subpart.

(1) Any United States Environmental Protection Agency inspector or auditor must be given full, complete and immediate access to conduct inspections and audits of the foreign refinery.

(i) Inspections and audits may be either announced in advance by EPA, or unannounced.

(ii) Access will be provided to any location where:

(A) Gasoline is produced;

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(B) Documents related to refinery operations are kept;

(C) Gasoline or blendstock samples are tested or stored; and

(D) Benzene-FRGAS is stored or transported between the foreign refinery and the United States, including storage tanks, vessels and pipelines.

(iii) Inspections and audits may be by EPA employees or contractors to EPA.

(iv) Any documents requested that are related to matters covered by inspections and audits must be provided to an EPA inspector or auditor on request.

(v) Inspections and audits by EPA may include review and copying of any documents related to:

(A) Refinery baseline establishment, if applicable, including the volume and benzene content of gasoline; transfers of title or custody of any gasoline or blendstocks whether Benzene-FRGAS or Non-Benzene-FRGAS, produced at the foreign refinery during the period January 1, 2004 through December 31, 2005, and any work papers related to refinery baseline establishment;

(B) The volume and benzene content of Benzene-FRGAS;

(C) The proper classification of gasoline as being Benzene-FRGAS or as not being Benzene-FRGAS, or as Certified Benzene-FRGAS or as Non-Certified Benzene-FRGAS, and all other relevant designations under this subpart;

(D) Transfers of title or custody to Benzene-FRGAS;

(E) Sampling and testing of Benzene-FRGAS;

(F) Work performed and reports prepared by independent third parties and by independent auditors under the requirements of this section, including work papers; and

(G) Reports prepared for submission to EPA, and any work papers related to such reports.

(vi) Inspections and audits by EPA may include taking samples of gasoline, gasoline additives or blendstock, and interviewing employees.

(vii) Any employee of the foreign refiner must be made available for interview by the EPA inspector or auditor, on request, within a reasonable time period.

(viii) English language translations of any documents must be provided to

an EPA inspector or auditor, on request, within 10 working days.

(ix) English language interpreters must be provided to accompany EPA inspectors and auditors, on request.

(2) An agent for service of process located in the District of Columbia shall be named, and service on this agent constitutes service on the foreign refiner or any employee of the foreign refiner for any action by EPA or otherwise by the United States related to the requirements of this subpart.

(3) The forum for any civil or criminal enforcement action related to the provisions of this section for violations of the Clean Air Act or regulations promulgated thereunder shall be governed by the Clean Air Act, including the EPA administrative forum where allowed under the Clean Air Act.

(4) United States substantive and procedural laws shall apply to any civil or criminal enforcement action against the foreign refiner or any employee of the foreign refiner related to the provisions of this section.

(5) Submitting a petition for participation in the benzene foreign refiner program or producing and exporting gasoline under any such program, and all other actions to comply with the requirements of this subpart relating to participation in any benzene foreign refiner program, or to establish an individual refinery gasoline benzene baseline under this subpart constitute actions or activities covered by and within the meaning of the provisions of 28 U.S.C. 1605(a)(2), but solely with respect to actions instituted against the foreign refiner, its agents and employees in any court or other tribunal in the United States for conduct that violates the requirements applicable to the foreign refiner under this subpart, including conduct that violates the False Statements Accountability Act of 1996 (18 U.S.C. 1001) and section 113(c)(2) of the Clean Air Act (42 U.S.C. 7413).

(6) The foreign refiner, or its agents or employees, will not seek to detain or to impose civil or criminal remedies against EPA inspectors or auditors, whether EPA employees or EPA contractors, for actions performed within the scope of EPA employment related to the provisions of this section.

(7) The commitment required by this paragraph (i) shall be signed by the owner or president of the foreign refiner business.

(8) In any case where Benzene-FRGAS produced at a foreign refinery is stored or transported by another company between the refinery and the vessel that transports the Benzene-FRGAS to the United States, the foreign refiner shall obtain from each such other company a commitment that meets the requirements specified in paragraphs (i)(1) through (7) of this section, and these commitments shall be included in the foreign refiner's petition to participate in any benzene foreign refiner program.

(j) *Sovereign immunity.* By submitting a petition for participation in any benzene foreign refiner program under this subpart (and baseline, if applicable) under this section, or by producing and exporting gasoline to the United States under any such program, the foreign refiner, and its agents and employees, without exception, become subject to the full operation of the administrative and judicial enforcement powers and provisions of the United States without limitation based on sovereign immunity, with respect to actions instituted against the foreign refiner, its agents and employees in any court or other tribunal in the United States for conduct that violates the requirements applicable to the foreign refiner under this subpart, including conduct that violates the False Statements Accountability Act of 1996 (18 U.S.C. 1001) and section 113(c)(2) of the Clean Air Act (42 U.S.C. 7413).

(k) *Bond posting.* Any foreign refiner shall meet the requirements of this paragraph (k) as a condition to approval as benzene foreign refiner under this subpart.

(l) The foreign refiner shall post a bond of the amount calculated using the following equation:

$$\text{Bond} = G \times \$0.01$$

Where:

Bond = amount of the bond in U.S. dollars

G = the largest volume of gasoline produced at the foreign refinery and exported to the United States, in gallons, during a single calendar year among the most recent of the following calendar years, up to a maximum of five calendar years: the calendar

year immediately preceding the date the refinery's baseline petition is submitted, the calendar year the baseline petition is submitted, and each succeeding calendar year.

(2) Bonds shall be posted by:

(i) Paying the amount of the bond to the Treasurer of the United States;

(ii) Obtaining a bond in the proper amount from a third party surety agent that is payable to satisfy United States administrative or judicial judgments against the foreign refiner, provided EPA agrees in advance as to the third party and the nature of the surety agreement; or

(iii) An alternative commitment that results in assets of an appropriate liquidity and value being readily available to the United States, provided EPA agrees in advance as to the alternative commitment.

(3) Bonds posted under this paragraph (k) shall—

(i) Be used to satisfy any judicial judgment that results from an administrative or judicial enforcement action for conduct in violation of this subpart, including where such conduct violates the False Statements Accountability Act of 1996 (18 U.S.C. 1001) and section 113(c)(2) of the Clean Air Act (42 U.S.C. 7413);

(ii) Be provided by a corporate surety that is listed in the United States Department of Treasury Circular 570 "Companies Holding Certificates of Authority as Acceptable Sureties on Federal Bonds"; and

(iii) Include a commitment that the bond will remain in effect for at least five years following the end of latest annual reporting period that the foreign refiner produces gasoline pursuant to the requirements of this subpart.

(4) On any occasion a foreign refiner bond is used to satisfy any judgment, the foreign refiner shall increase the bond to cover the amount used within 90 days of the date the bond is used.

(5) If the bond amount for a foreign refiner increases, the foreign refiner shall increase the bond to cover the shortfall within 90 days of the date the bond amount changes. If the bond amount decreases, the foreign refiner may reduce the amount of the bond beginning 90 days after the date the bond amount changes.

(l) [Reserved]

(m) *English language reports.* Any report or other document submitted to EPA by a foreign refiner shall be in English language, or shall include an English language translation.

(n) *Prohibitions.* (1) No person may combine Certified Benzene-FRGAS with any Non-Certified Benzene-FRGAS or Non-Benzene-FRGAS, and no person may combine Certified Benzene-FRGAS with any Certified Benzene-FRGAS produced at a different refinery, until the importer has met all the requirements of paragraph (o) of this section, except as provided in paragraph (e) of this section.

(2) No foreign refiner or other person may cause another person to commit an action prohibited in paragraph (n)(1) of this section, or that otherwise violates the requirements of this section.

(o) *United States importer requirements.* Any United States importer shall meet the following requirements:

(1) Each batch of imported gasoline shall be classified by the importer as being Benzene-FRGAS or as Non-Benzene-FRGAS, and each batch classified as Benzene-FRGAS shall be further classified as Certified Benzene-FRGAS or as Non-Certified Benzene-FRGAS.

(2) Gasoline shall be classified as Certified Benzene-FRGAS or as Non-Certified Benzene-FRGAS according to the designation by the foreign refiner if this designation is supported by product transfer documents prepared by the foreign refiner as required in paragraph (d) of this section, unless the gasoline is classified as Non-Certified Benzene-FRGAS under paragraph (g) of this section. Additionally, the importer shall comply with all requirements of this subpart applicable to importers.

(3) For each gasoline batch classified as Benzene-FRGAS, any United States importer shall perform the following procedures.

(i) In the case of both Certified and Non-Certified Benzene-FRGAS, have an independent third party:

(A) Determine the volume of gasoline in the vessel;

(B) Use the foreign refiner's Benzene-FRGAS certification to determine the name and EPA-assigned registration number of the foreign refinery that produced the Benzene-FRGAS;

(C) Determine the name and country of registration of the vessel used to transport the Benzene-FRGAS to the United States; and

(D) Determine the date and time the vessel arrives at the United States port of entry.

(ii) In the case of Certified Benzene-FRGAS, have an independent third party:

(A) Collect a representative sample from each vessel compartment subsequent to the vessel's arrival at the United States port of entry and prior to off loading any gasoline from the vessel;

(B) Obtain the compartment samples; and

(C) Determine the benzene content value of each compartment sample using the methodology specified at § 80.46(e) by the third party analyzing the sample or by the third party observing the importer analyze the sample.

(4) Any importer shall submit reports within 30 days following the date any vessel transporting Benzene-FRGAS arrives at the United States port of entry:

(i) To the Administrator containing the information determined under paragraph (o)(3) of this section; and

(ii) To the foreign refiner containing the information determined under paragraph (o)(3)(ii) of this section, and including identification of the port at which the product was offloaded.

(5) Any United States importer shall meet all other requirements of this subpart for any imported gasoline that is not classified as Certified Benzene-FRGAS under paragraph (o)(2) of this section.

(p) *Truck imports of Certified Benzene-FRGAS produced at a foreign refinery.*

(1) Any refiner whose Certified Benzene-FRGAS is transported into the United States by truck may petition EPA to use alternative procedures to meet the following requirements:

(i) Certification under paragraph (d)(5) of this section;

(ii) Load port and port of entry sampling and testing under paragraphs (f) and (g) of this section;

(iii) Attest under paragraph (h) of this section; and

(iv) Importer testing under paragraph (o)(3) of this section.

(2) These alternative procedures must ensure Certified Benzene-FRGAS remains segregated from Non-Certified Benzene-FRGAS and from Non-Benzene-FRGAS until it is imported into the United States. The petition will be evaluated based on whether it adequately addresses the following:

(i) Provisions for monitoring pipeline shipments, if applicable, from the refinery, that ensure segregation of Certified Benzene-FRGAS from that refinery from all other gasoline;

(ii) Contracts with any terminals and/or pipelines that receive and/or transport Certified Benzene-FRGAS, that prohibit the commingling of Certified Benzene-FRGAS with any of the following:

(A) Other Certified Benzene-FRGAS from other refineries.

(B) All Non-Certified Benzene-FRGAS.

(C) All Non-Benzene-FRGAS;

(iii) Procedures for obtaining and reviewing truck loading records and United States import documents for Certified Benzene-FRGAS to ensure that such gasoline is only loaded into trucks making deliveries to the United States;

(iv) Attest procedures to be conducted annually by an independent third party that review loading records and import documents based on volume reconciliation, or other criteria, to confirm that all Certified Benzene-FRGAS remains segregated throughout the distribution system and is only loaded into trucks for import into the United States.

(3) The petition required by this section must be submitted to EPA along with the application for temporary refiner relief individual refinery benzene standard under this subpart.

(q) *Withdrawal or suspension of foreign refiner status.* EPA may withdraw or suspend a foreign refiner's benzene baseline or standard approval for a foreign refinery where—

(1) A foreign refiner fails to meet any requirement of this section;

(2) A foreign government fails to allow EPA inspections as provided in paragraph (i)(1) of this section;

(3) A foreign refiner asserts a claim of, or a right to claim, sovereign immunity in an action to enforce the requirements in this subpart; or

(4) A foreign refiner fails to pay a civil or criminal penalty that is not satisfied using the foreign refiner bond specified in paragraph (k) of this section.

(r) *Early use of a foreign refiner benzene baseline.*

(1) A foreign refiner may begin using an individual refinery benzene baseline under this subpart before EPA has approved the baseline, provided that:

(i) A baseline petition has been submitted as required in paragraph (b) of this section;

(ii) EPA has made a provisional finding that the baseline petition is complete;

(iii) The foreign refiner has made the commitments required in paragraph (i) of this section;

(iv) The persons that will meet the independent third party and independent attest requirements for the foreign refinery have made the commitments required in paragraphs (f)(3)(iii) and (h)(7)(iii) of this section; and

(v) The foreign refiner has met the bond requirements of paragraph (k) of this section.

(2) In any case where a foreign refiner uses an individual refinery baseline before final approval under paragraph (r)(1) of this section, and the foreign refinery baseline values that ultimately are approved by EPA are more stringent than the early baseline values used by the foreign refiner, the foreign refiner shall recalculate its compliance, *ab initio*, using the baseline values approved by the EPA, and the foreign refiner shall be liable for any resulting violation of the requirements of this subpart.

(s) *Additional requirements for petitions, reports and certificates.* Any petition for approval to produce gasoline subject to the benzene foreign refiner program, any alternative procedures under paragraph (p) of this section, any report or other submission required by paragraph (c), (f)(2), or (i) of this section, and any certification under paragraph (d)(3) of this section shall be—

(1) Submitted in accordance with procedures specified by the Administrator, including use of any forms that may be specified by the Administrator.

(2) Be signed by the president or owner of the foreign refiner company, or by that person's immediate designee, and shall contain the following declaration:

I hereby certify: (1) That I have actual authority to sign on behalf of and to bind [insert name of foreign refiner] with regard to all statements contained herein; (2) that I am aware that the information contained herein is being Certified, or submitted to the United States Environmental Protection Agency, under the requirements of 40 CFR part 80, subpart L, and that the information is material for determining compliance under these regulations; and (3) that I have read and understand the information being Certified or submitted, and this information is true, complete and correct to the best of my knowledge and belief after I have taken reasonable and appropriate steps to verify the accuracy thereof. I affirm that I have read and understand the provisions of 40 CFR part 80, subpart L, including 40 CFR 80.1363 apply to [insert name of foreign refiner]. Pursuant to Clean Air Act section 113(c) and 18 U.S.C. 1001, the penalty for furnishing false, incomplete or misleading information in this certification or submission is a fine of up to \$10,000 U.S., and/or imprisonment for up to five years.

APPENDIX A TO PART 80—TEST FOR THE DETERMINATION OF PHOSPHORUS IN GASOLINE

1. Scope.

1.1 This method was developed for the determination of phosphorus generally present as pentavalent phosphate esters or salts, or both, in gasoline. This method is applicable for the determination of phosphorus in the range from 0.0008 to 0.15 g P/U.S. gal, or 0.2 to 49 mg P/liter.

2. Applicable documents.

2.1 ASTM Standards:

D 1100 Specification for Filter Paper for Use in Chemical Analysis.

3. Summary of method.

3.1 Organic matter in the sample is decomposed by ignition in the presence of zinc oxide. The residue is dissolved in sulfuric acid and reacted with ammonium molybdate and hydrazine sulfate. The absorbance of the "Molybdenum Blue" complex is proportional to the phosphorus concentration in the sample and is read at approximately 820 nm in a 5-cm cell.

4. Apparatus.

4.1 Buret, 10-ml capacity, 0.05-ml subdivisions.

4.2 Constant-Temperature Bath, equipped to hold several 100-ml volumetric flasks submerged to the mark. Bath must have a large enough reservoir or heat capacity to keep the temperature at 180 to 190 °F (82.2 to 87.8 °C) during the entire period of sample heating.

NOTE 1: If the temperature of the hot water bath drops below 180 °F (82.2 °C) the color development may not be complete.

4.3 Cooling Bath, equipped to hold several 100-ml volumetric flasks submerged to the mark in ice water.

4.4 Filter Paper, for quantitative analysis, Class G for fine precipitates as defined in Specification D 1100.

4.5 Ignition Dish—Coors porcelain evaporating dish, glazed inside and outside, with pourout (size no. 00A, diameter 75 mm, capacity 70 ml).

4.6 Spectrophotometer, equipped with a tungsten lamp, a red-sensitive phototube capable of operating at 830 nm and with absorption cells that have a 5-cm light path.

4.7 Thermometer, range 50 to 220 °F (10 to 105 °C).

4.8 Volumetric Flask, 100-ml with ground-glass stopper.

4.9 Volumetric Flask, 1000-ml with ground-glass stopper.

4.10 Syringe, Luer-Lok, 10-ml equipped with 5-cm. 22-gage needle.

5. Reagents.

5.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Purity of Water—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

5.3 Ammonium Molybdate Solution—Using graduated cylinders for measurement add slowly (Note 2), with continuous stirring, 225 ml of concentrated sulfuric acid to 500 ml of water contained in a beaker placed in a bath of cold water. Cool to room temperature and add 20 g of ammonium molybdate tetrahydrate ((NH₄)₆ Mo₇ O₂₄·4H₂O). Stir until solution is complete and transfer to a 1000-ml flask. Dilute to the mark with water.

NOTE 2: Wear a face shield, rubber gloves, and a rubber apron when adding concentrated sulfuric acid to water.

5.4 Hydrazine Sulfate Solution—Dissolve 1.5 of hydrazine sulfate ($H_2NNH_2 \cdot H_2SO_4$) in 1 litre of water, measured with a graduated cylinder.

NOTE 3: This solution is not stable. Keep it tightly stoppered and in the dark. Prepare a fresh solution after 3 weeks.

5.5 Molybdate-Hydrazine Reagent—Pipet 25 ml of ammonium molybdate solution into a 100-ml volumetric flask containing approximately 50 ml of water, add by pipet 10 ml of $N_2NNH_2 \cdot H_2SO_4$ solution, and dilute to 100 ml with water.

NOTE 4: This reagent is unstable and should be used within about 4 h. Prepare it immediately before use. Each determination (including the blank) uses 50 ml.

5.6 Phosphorus, Standard Solution (10.0 μg P/ml)—Pipet 10 ml of stock standard phosphorus solution into a 1000-ml volumetric flask and dilute to the mark with water.

5.7 Phosphorus, Stock Standard Solution (1.00 mg P/ml)—Dry approximately 5 g of potassium dihydrogen phosphate (KH_2PO_4) in an oven at 221 to 230 °F (105 to 110 °C) for 3 h. Dissolve 4.393 \pm 0.002 g of the reagent in 150 ml, measured with a graduated cylinder, of $H_2SO_4(1+10)$ contained in a 1000-ml volumetric flask. Dilute with water to the mark.

5.8 Sulfuric Acid (1+10)—Using graduated cylinders for measurement add slowly (Note 2), with continuous stirring, 100-ml of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84) to 1 litre of water contained in a beaker placed in a bath of cold water.

5.9 Zinc Oxide.

NOTE 5: High-bulk density zinc oxide may cause spattering. Density of approximately 0.5 g/cm³ has been found satisfactory.

6. Calibration.

6.1 Transfer by buret, or a volumetric transfer pipet, 0.0, 0.5, 1.0, 1.5, 2.0, 3.0, 3.5, and 4.0 ml of phosphorus standard solution into 100-ml volumetric flasks.

6.2 Pipet 10 ml of $H_2SO_4(1+10)$ into each flask. Mix immediately by swirling.

6.3 Prepare the molybdate-hydrazine solution. Prepare sufficient volume of reagent based on the number of samples being analyzed.

6.4 Pipet 50 ml of the molybdate-hydrazine solution to each volumetric flask. Mix immediately by swirling.

6.5 Dilute to 100 ml with water.

6.6 Mix well and place in the constant-temperature bath so that the contents of the flask are submerged below the level of the bath. Maintain bath temperature at 180 to 190 °F (82.2 to 87.8 °C) for 25 min (Note 1).

6.7 Transfer the flask to the cooling bath and cool the contents rapidly to room temperature. Do not allow the samples to cool more than 5 °F (2.8 °C) below room temperature.

NOTE 6: Place a chemically clean thermometer in one of the flasks to check the temperature.

6.8 After cooling the flasks to room temperature, remove them from the cooling water bath and allow them to stand for 10 min. at room temperature.

6.9 Using the 2.0-ml phosphorus standard in a 5-cm cell, determine the wavelength near 820 nm that gives maximum absorbance. The wavelength giving maximum absorbance should not exceed 830 nm.

6.9.1 Using a red-sensitive phototube and 5-cm cells, adjust the spectrophotometer to zero absorbance at the wavelength of maximum absorbance using distilled water in both cells. Use the wavelength of maximum absorbance in the determination of calibration readings and future sample readings.

6.9.2 The use of 1-cm cells for the higher concentrations is permissible.

6.10 Measure the absorbance of each calibration sample including the blank (0.0 ml phosphorus standard) at the wavelength of maximum absorbance with distilled water in the reference cell.

NOTE 7: Great care must be taken to avoid possible contamination. If the absorbance of the blank exceeds 0.04 (for 5-cm cell), check for source of contamination. It is suggested that the results be disregarded and the test be rerun with fresh reagents and clean glassware.

6.11 Correct the absorbance of each standard solution by subtracting the absorbance of the blank (0 ml phosphorus standard).

6.12 Prepare a calibration curve by plotting the corrected absorbance of each standard solution against micrograms of phosphorus. One millilitre of phosphorus standard solution provides 10 μg of phosphorus.

7. Sampling.

7.1 Selection of the size of the sample to be tested depends on the expected concentration of phosphorous in the sample. If a concentration of phosphorus is suspected to be less than 0.0038 g/gal (1.0 mg/litre), it will be necessary to use 10 ml of sample.

NOTE 8: Two grams of zinc oxide cannot absorb this volume of gasoline. Therefore the 10-ml sample is ignited in aliquots of 2 ml in the presence of 2 g of zinc oxide.

7.2 The following table serves as a guide for selecting sample size:

Phosphorus, milligrams per liter	Equivalent, grams per gallon	Sample size, milliliter
2.5 to 40	0.01 to 0.15	1.00
1.3 to 20	0.005 to 0.075	2.00
0.9 to 13	0.0037 to 0.05	3.00
1 or less	0.0038 or less	10.00

8. Procedure.

8.1 Transfer 2 ± 0.2 g of zinc oxide into a conical pile in a clean, dry, unetched ignition dish.

NOTE 9: In order to obtain satisfactory accuracy with the small amounts of phosphorus involved, it is necessary to take extensive precautions in handling. The usual precautions of cleanliness, careful manipulation, and avoidance of contamination should be scrupulously observed; also, all glassware should be cleaned before use, with cleaning acid or by some procedure that does not involve use of commercial detergents. These compounds often contain alkali phosphates which are strongly adsorbed by glass surfaces and are not removed by ordinary rinsing. It is desirable to segregate a special stock of glassware for use only in the determination of phosphorus.

8.2 Make a deep depression in the center of the zinc oxide pile with a stirring rod.

8.3 Pipet the gasoline sample (Note 10) (see 7.2 for suggested sample volume) into the depression in the zinc oxide. Record the temperature of the fuel if the phosphorus content is required at 60 °F (15.6 °C) and make correction as directed in 9.2.

NOTE 10: For the 10-ml sample use multiple additions and a syringe. Hold the tip of the needle at approximately $\frac{2}{3}$ of the depth of the zinc oxide layer and slowly deliver 2 ml of the sample: fast sample delivery may give low results. Give sufficient time for the gasoline to be absorbed by the zinc oxide. Follow step 8.6. Cool the dish to room temperature. Repeat steps 8.3 and 8.6 until all the sample has been burned. Safety—cool the ignition dish before adding the additional aliquots of gasoline to avoid a flash fire.

8.4 Cover the sample with a small amount of fresh zinc oxide from reagent bottle (use the tip of a small spatula to deliver approximately 0.2 g). Tap the sides of the ignition dish to pack the zinc oxide.

8.5 Prepare the blank, using the same amount of zinc oxide in an ignition dish.

8.6 Ignite the gasoline, using the flame from a bunsen burner. Allow the gasoline to burn to extinction (NOTE 10).

8.7 Place the ignition dishes containing the sample and blank in a hot muffle furnace set at a temperature of 1150 to 1300 °F (621 to 704 °C) for 10 min. Remove and cool the ignition dishes. When cool gently tap the sides of the dish to loosen the zinc oxide. Again place the dishes in the muffle furnace for 5 min. Remove and cool the ignition dishes to room temperature. The above treatment is usually sufficient to burn the carbon. If the carbon is not completely burned off place the dish into the oven for further 5-min. periods.

NOTE 11: Step 8.7 may also be accomplished by heating the ignition dish with a Meker burner gradually increasing the intensity of

heat until the carbon from the sides of the dish has been burned, then cool to room temperature.

8.8 Pipet 25 ml of H_2SO_4 (1+10) to each ignition dish. While pipeting, carefully wash all traces of zinc oxide from the sides of the ignition dish.

8.9 Cover the ignition dish with a borosilicate watch glass and warm the ignition dish on a hot plate until the zinc oxide is completely dissolved.

8.10 Transfer the solution through filter paper to a 100-ml volumetric flask. Rinse the watch glass and the dish several times with distilled water (do not exceed 25 ml) and transfer the washings through the filter paper to the volumetric flask.

8.11 Prepare the molybdate-hydrazine solution.

8.12 Add 50 ml of the molybdate-hydrazine solution by pipet to each 100-ml volumetric flask. Mix immediately by swirling.

8.13 Dilute to 100 ml with water and mix well. Remove stoppers from flasks after mixing.

8.14 Place the 100-ml flasks in the constant-temperature bath for 25 min. so that the contents of the flasks are below the liquid level of the bath. The temperature of the bath should be 180 to 190 °F (82.2 to 87.8 °C) (NOTE 1).

8.15 Transfer the 100-ml flasks to the cooling bath and cool the contents rapidly to room temperature (NOTE 6).

8.16 Allow the samples to stand at room temperature before measuring the absorbance.

NOTE 12: The color developed is stable for at least 4 h.

8.17 Set the spectrophotometer to the wavelength of maximum absorbance as determined in 6.9. Adjust the spectrophotometer to zero absorbance, using distilled water in both cells.

8.18 Measure the absorbance of the samples at the wavelength of maximum absorbance with distilled water in the reference cell.

8.19 Subtract the absorbance of the blank from the absorbance of each sample (NOTE 7).

8.20 Determine the micrograms of phosphorus in the sample, using the calibration curve from 6.12 and the corrected absorbance.

9. Calculations.

9.1 Calculate the milligrams of phosphorus per litre of sample as follows:

$$P, \text{ mg/litre} = P/V$$

where:

P = micrograms of phosphorus read from calibration curve, and

V = millilitres of gasoline sample.

To convert to grams of phosphorus per U.S. gallon of sample, multiply mg P/litre by 0.0038.

9.2 If the gasoline sample was taken at a temperature other than 60 °F (15.6 °C) make the following temperature correction:

$$\text{mg P/litre at } 15.6 \text{ }^\circ\text{C} = [\text{mg P/litre at } t] [1+0.001 (t - 15.6)]$$

where:

t = observed temperature of the gasoline, °C.

9.3 Concentrations below 2.5 mg/litre or 0.01 g/gal should be reported to the nearest 0.01 mg/litre or 0.0001 g/U.S. gal.

9.3.1 For higher concentrations, report results to the nearest 1 mg P/litre or 0.005 g P/U.S. gal.

10. Precision.

10.1 The following criteria should be used for judging the acceptability of results (95 percent confidence):

10.2 Repeatability—Duplicate results by the same operator should be considered suspect if they differ by more than the following amounts:

g P/U.S. gal (mg-P/litre)	Repeatability
0.0008 to 0.005 (0.2 to 1.3) ..	0.0002 g P/U.S. gal (0.05 mg P/litre).
0.005 to 0.15 (1.3 to 40)	7% of the mean.

10.3 Reproducibility—The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amounts:

g P/U.S. gal (mg-P/litre)	Reproducibility
0.0008 to 0.005 (0.2 to 1.3) ..	0.0005 g P/U.S. gal (0.13 mg P/litre).
0.005 to 0.15 (1.3 to 40)	13% of the mean.

[39 FR 24891, July 8, 1974; 39 FR 25653, July 12, 1974]

APPENDIX B TO PART 80—TEST METHODS FOR LEAD IN GASOLINE

METHOD 1—STANDARD METHOD TEST FOR LEAD IN GASOLINE BY ATOMIC ABSORPTION SPECTROMETRY

1. Scope.

1.1. This method covers the determination of the total lead content of gasoline. The procedure's calibration range is 0.010 to 0.10 gram of lead/U.S. gal. Samples above this level should be diluted to fall within this range or a higher level calibration standard curve must be prepared. The higher level curve must be shown to be linear and measurement of lead at these levels must be shown to be accurate by the analysis of control samples at a higher level of alkyl lead content. The method compensates for variations in gasoline composition and is independent of lead alkyl type.

2. Summary of method.

2.1 The gasoline sample is diluted with methyl isobutyl ketone and the alkyl lead compounds are stabilized by reaction with iodine and a quarternary ammonium salt. The lead content of the sample is determined by atomic absorption flame spectrometry at 2833 Å, using standards prepared from reagent grade lead chloride. By the use of this treatment, all alkyl lead compounds give identical response.

3. Apparatus.

3.1 Atomic Absorption Spectrometer, capable of scale expansion and nebulizer adjustment, and equipped with a slot burner and premix chamber for use with an air-acetylene flame.

3.2 Volumetric Flasks, 50-ml, 100-ml, 250-ml, and one litre sizes.

3.3 Pipets, 2-ml, 5-ml, 10-ml, 20-ml, and 50-ml sizes.

3.4 Micropipet, 100-µl, Eppendorf type or equivalent.

4. Reagents.

4.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

4.2 Purity of Water—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

4.3 Aliquat 336 (tricapryl methyl ammonium chloride).

4.4 Aliquat 336/MIBK Solution (10 percent v/v)—Dissolve and dilute 100 ml (88.0 g) of Aliquat 336 with MIBK to one liter.

4.5 Aliquat 336/MIBK Solution (1 percent v/v)—Dissolve and dilute 10 ml (8.8 g) of Aliquat 336 with MIBK to one liter.

4.6 Iodine Solution—Dissolve and dilute 3.0 g iodine crystals with Toluene to 100 ml.

4.7 Lead Chloride.

4.8 Lead-Sterile Gasoline—Gasoline containing less than 0.005 g Pb/gal.

4.9 Lead, Standard Solution (5.0 g Pb/gal)—Dissolve 0.4433 g of lead chloride (PbCl₂) previously dried at 105 °C for 3 h in about 200 ml of 10 percent Aliquat 336/MIBK solution in a 250-ml volumetric flask. Dilute to the mark with the 10 percent Aliquat solution, mix, and store in a brown bottle having a polyethylene-lined cap. This solution contains 1,321 µg Pb/ml, which is equivalent to 5.0 g Pb/gal.

4.10 Lead, Standard Solution (1.0 g Pb/gal)—By means of a pipet, accurately transfer 50.0 ml of the 5.0 g Pb/gal solution to a 250-ml volumetric flask, dilute to volume with 1 percent Aliquat/MIBK solution. Store in a brown bottle having a polyethylene-lined cap.

4.11 Lead, Standard Solutions (0.02, 0.05, and 0.10 g Pb/gal)—Transfer accurately by means of pipets 2.0, 5.0, and 10.0 ml of the 1.0-g Pb/gal solution to 100-ml volumetric flasks; add 5.0 ml of 1 percent Aliquat 336 solution to each flask; dilute to the mark with MIBK. Mix well and store in bottles having polyethylene-lined caps.

4.12 Methyl Isobutyl Ketone (MIBK). (4-methyl-2-pentanone).

5. Calibration.

5.1 Preparation of Working Standards—Prepare three working standards and a blank using the 0.02, 0.05, and 0.10-g Pb/gal standard lead solutions described in 4.11.

5.1.1 To each of four 50-ml volumetric flasks containing 30 ml of MIBK, add 5.0 ml of low lead standard solution and 5.0 ml of lead-free gasoline. In the case of the blank, add only 5.0 ml of lead-free gasoline.

5.1.2 Add immediately 0.1 ml of iodine/toluene solution by means of the 100- μ l Eppendorf pipet. Mix well.¹

5.1.3 Add 5 ml of 1 percent Aliquat 336 solution and mix.

5.1.4 Dilute to volume with MIBK and mix well.

5.2 Preparation of Instrument—Optimize the atomic absorption equipment for lead at 2833 Å. Using the reagent blank, adjust the gas mixture and the sample aspiration rate to obtain an oxidizing flame.

5.2.1 Aspirate the 0.1-g Pb/gal working standard and adjust the burner position to give maximum response. Some instruments require the use of scale expansion to produce a reading of 0.150 to 0.170 for this standard.

5.2.2 Aspirate the reagent blank to zero the instrument and check the absorbances of the three working standards for linearity.

6. Procedure.

6.1 To a 50 ml volumetric flask containing 30 ml MIBK, add 5.0 ml of gasoline sample and mix.²

6.1.1 Add 0.10 ml (100 μ l) of iodine/toluene solution and allow the mixture to react about 1 minute.³

6.1.2 Add 5.0 ml of 1 percent Aliquot 336/MIBK solution and mix.

6.1.3 Dilute to volume with MIBK and mix.

¹EPA practice will be to mix well by shaking vigorously for approximately one minute.

²The gasoline should be allowed to come to room temperature (25 °C).

³See footnote 1 of section 5.1.2.

6.2 Aspirate the samples and working standards and record the absorbance values with frequent checks of the zero.

6.3 Any sample resulting in a peak greater than 0.05 g Pb/gal will be run in duplicate. Samples registering greater than 0.10 g Pb/gal should be diluted with iso-octane or unleaded fuel to fall within the calibration range or a higher level calibration standard curve must be prepared. The higher level curve must be shown to be linear and measurement of lead at these levels must be shown to be accurate by the analysis of control samples at a higher level of alkyl lead content.

7. Calculations.

7.1 Plot the absorbance values versus concentration represented by the working standards and read the concentrations of the samples from the graph.

8. Precision.

8.1 The following criteria should be used for judging the acceptability of results (95 percent confidence):

8.1.1 Repeatability—Duplicate results by the same operator should be considered suspect if they differ by more than 0.005 g/gal.

8.1.2 Reproducibility—The results submitted by each of two laboratories should not be considered suspect unless the two results differ by more than 0.01 g/gal.

METHOD 2—AUTOMATED METHOD TEST FOR LEAD IN GASOLINE BY ATOMIC ABSORPTION SPECTROMETRY

1. Scope and application.

1.1 This method covers the determination of the total lead content of gasoline. The procedure's calibration range is 0.010 to 0.10 gram of lead/U.S. gal. Samples above this level should be diluted to fall within this range or a higher level calibration standard curve must be prepared. The higher level curve must be shown to be linear and measurement of lead at these levels must be shown to be accurate by the analysis of control samples at a higher level of alkyl lead content. The method compensates for variations in gasoline composition and is independent of lead alkyl type.

1.2 This method may be used as an alternative to the Standard Method set forth above.

1.3 Where trade names or specific products are noted in the method, equivalent apparatus and chemical reagents may be used. Mention of trade names or specific products is for the assistance of the user and does not constitute endorsement by the U.S. Environmental Protection Agency.

2. Summary of method.

2.1 The gasoline sample is diluted with methyl isobutyl ketone (MIBK) and the alkyl lead compounds are stabilized by reacting with iodine and a quarternary ammonium salt. An automated system is used to perform the diluting and the chemical reactions and feed the products to the atomic absorption spectrometer with an air-acetylene flame.

2.2 The dilution of the gasoline with MIBK compensates for severe non-atomic absorption, scatter from unburned carbon containing species and matrix effects caused in part by the burning characteristics of gasoline.

2.3 The *in-situ* reaction of alkyl lead in gasoline with iodine eliminates the problem of variations in response due to different alkyl types by leveling the response of all alkyl lead compounds.

2.4 The addition of the quarternary ammonium salt improves response and increases the stability of the alkyl iodide complex.

3. Sample handling and preservation.

3.1 Samples should be collected and stored in containers which will protect them from changes in the lead content of the gasoline such as from loss of volatile fractions of the gasoline by evaporation or leaching of the lead into the container or cap.

3.2 If samples have been refrigerated they should be brought to room temperature prior to analysis.

4. Apparatus.

4.1 AutoAnalyzer system consisting of:

4.1.1 Sampler 20/hr cam, 30/hr cam.

4.1.2 Proportioning pump.

4.1.3 Lead in gas manifold.

4.1.4 Disposable test tubes.

4.1.5 Two 2-liter and one 0.5 liter Erlenmeyer solvent displacement flasks. Alternatively, high pressure liquid chromatography (HPLC) or syringe pumps may be used.

4.2 Atomic Absorption Spectroscopy (AAS) Detector System consisting of:

4.2.1 Atomic absorption spectrometer.

4.2.2 10" strip chart recorder.

4.2.3 Lead hollow cathode lamp or electrodeless discharge lamp (EDL).

5. Reagents.

5.1 Aliquat 336/MIBK solution (10% v/v): Dissolve and dilute 100 ml (88.0 g) of Aliquat 336 (Aldrich Chemical Co., Milwaukee, Wisconsin) with MIBK (Burdick & Jackson Lab., Inc., Muskegon, Michigan) to one liter.

5.2 Aliquat 336/iso-octane solution (1% v/v): Dissolve and dilute 10 ml (8.8 g) of Aliquat 336 (reagent 5.1) with iso-octane to one liter.

5.3 Iodine solution (3% w/v): Dissolve and dilute 3.0 g iodine crystals (American Chem-

ical Society) with toluene (Burdick & Jackson Lab., Inc., Muskegon, Michigan) to 100 ml.

5.4 Iodine working solution (0.24% w/v): Dilute 8 ml of reagent 5.3 to 100 ml with toluene.

5.5 Methyl isobutyl ketone (MIBK) (4-methyl-2-pentanone).

5.6 Certified unleaded gasoline (Phillips Chemical Co., Borger, Texas) or iso-octane (Burdick & Jackson Lab, Inc., Muskegon, Michigan).

6. Calibration standards.

6.1 Stock 5.0 g Pb/gal Standard:

Dissolve 0.4433 gram of lead chloride (PbCl₂) previously dried at 105 °C for 3 hours in 200 ml of 10% v/v Aliquat 336/MIBK solution (reagent 5.1) in a 250 ml volumetric flask. Dilute to volume with reagent 5.1 and store in an amber bottle.

6.2 Intermediate 1.0 g Pb/gal Standard:

Pipet 50 ml of the 5.0 g Pb/gal standard into a 250 ml volumetric flask and dilute to volume with a 1% v/v Aliquat 336/iso-octane solution (reagent 5.2). Store in an amber bottle.

6.3 Working 0.02, 0.05, 0.10 g Pb/gal Standards:

Pipet 2.0, 5.0, and 10.0 ml of the 1.0 g Pb/gal solution to 100 ml volumetric flasks. Add 5 ml of a 1% Aliquat 336/iso-octane solution to each flask. Dilute to volume with iso-octane. These solutions contain 0.02, 0.05, and 0.10 g Pb/gal in a 0.05% Aliquat 336/iso-octane solution.

7. AAS Instrumental conditions.

7.1 Lead hollow cathode lamp.

7.2 Wavelength: 283.3 nm.

7.3 Slit: 4 (0.7mm).

7.4 Range: UV.

7.5 Fuel: Acetylene (approx. 20 ml/min at 8 psi).

7.6 Oxidant: Air (approx. 65 ml/min at 31 psi).

7.7 Nebulizer: 5.2 ml/min.

7.8 Chart speed: 10 in/hr.

8. Procedures.

8.1 AAS start-up.

8.1.1 Assure that instrumental conditions have been optimized and aligned according to Section 7 and the instrument has had substantial time for warm-up.

8.2 Auto Analyzer start-up [see figure 1].

8.2.1 Check all pump tubing and replace as necessary. Iodine tubing should be changed daily. All pump tubing should be replaced after one week of use. Place the platen on the pump.

8.2.2 Withdraw any water from the sample wash cup and fill with certified unleaded gasoline (reagent 5.6).

8.2.3 Fill the 2-liter MIBK dilution displacement Erlenmeyer flask (reagent 5.5)

and the 0.5 liter Aliquat 336/MIBK 1% v/v (reagent 5.2) displacement flask and place the rubber stopper glass tubing assemblies in their respective flasks.

8.2.4 Fill a 2-liter Erlenmeyer flask with distilled water. The water will be used to displace the solvents. Therefore, place the appropriate lines in this flask. This procedure is not relevant if syringe pumps are used.

8.2.5 Fill the final debubbler reverse displacement 2-liter Erlenmeyer flask with distilled water and place the rubber stopper glass tubing assembly in the flask.

8.2.6 Place the appropriate lines for the iodine reagent (reagent 5.4) and the wash solution (reagent 5.6) in their respective bottles.

8.2.7 Start the pump and connect the aspiration line from the manifold to the AAS.

8.2.8 Some initial checks to assure that the reagents are being added are:

a. A good uniform bubble pattern.

b. Yellow color evident due to iodine in the system.

c. No surging in any tubing.

8.3 Calibration.

8.3.1 Turn the chart drive on and obtain a steady baseline.

8.3.2 Load standards and samples into sample tray.

8.3.3 Start the sampler and run the standards (Note: first check the sample probe positioning with an empty test tube).

8.3.4 Check the linearity of calibration standards response and slope by running a least squares fit. Check these results against previously obtained results. They should agree within 10%.

8.3.5 If the above is in control then start the sample analysis.

8.4 Sample Analysis.

8.4.1 To minimize gasoline vapor in the laboratory, load the sample tray about 5-10 test tubes ahead of the sampler.

8.4.2 Record the sample number on the strip chart corresponding to the appropriate peak.

8.4.3 Every ten samples run the high calibration standard and a previously analyzed sample (duplicate). Also let the sampler skip to check the baseline.

8.4.4 After an acceptable peak (within the calibration range) is obtained, pour the excess sample from the test tube into the waste gasoline can.

8.4.5 Any sample resulting in a peak greater than 0.05 g Pb/gal will be run in duplicate. Samples registering greater than 0.10 g Pb/gal should be diluted with iso-octane or unleaded fuel to fall within the calibration range or a higher level calibration standard curve must be prepared. The higher level curve must be shown to be linear and measurement of lead at these levels must be shown to be accurate by the analysis of con-

trol samples at a higher level of alkyl lead content.

8.5 Shut Down.

8.5.1 Replace the solvent displacement flask with flasks filled with distilled water. Also place all other lines in a beaker of distilled water. Rinse the system with distilled water for 15 minutes.

8.5.2 Withdraw the gasoline from the wash cup and fill with water.

8.5.3 Dispose of all solvent waste in waste glass bottles.

8.5.4 Turn the AAS off after extinguishing the flame. Also turn the recorder and pump off. Remove the platen and release the pump tubing.

8.5.5 Shut the acetylene off at the tank and bleed the line.

9. Quality control.

9.1 Precision.

9.1.1 All duplicate results should be considered suspect if they differ by more than 0.005 g Pb/gal.

9.2 Accuracy.

9.2.1 All quality control standard checks should agree within 10% of the nominal value of the standard.

9.2.2 All spikes should agree within 10% of the known addition.

10. Past quality control data.

10.1 Precision.

10.1.1 Duplicate analysis for 156 samples in a single laboratory has resulted in an average difference of 0.00011 g Pb/gal with a standard deviation of 0.0023.

10.1.2 Replicate analysis in a single laboratory (greater than 5 determinations) of samples at concentrations of 0.010, 0.048, and 0.085 g Pb/gal resulted in relative standard deviations of 4.2%, 3.5%, and 3.3% respectively.

10.2 Accuracy.

10.2.1 The analysis of National Bureau of Standards (NBS) lead in reference fuel of known concentrations in a single laboratory has resulted in found values deviating from the true value for 11 determinations of 0.0322 g Pb/gal by an average of 0.56% with a standard deviation of 6.8%, for 15 determinations of 0.0519 g Pb/gal by an average of -1.1% with a standard deviation of 5.8%, and for 7 determinations of 0.0725 g Pb/gal by an average of 3.5% with a standard deviation of 4.8%.

10.2.2 Twenty-three analyses of blind reference samples in a single laboratory (U.S. EPA, RTP, N.C.) have resulted in found values differing from the true value by an average of -0.0009 g Pb/gal with a standard deviation of 0.004.

10.2.3 In a single laboratory, the average percent recovery of 108 spikes made to samples was 101% with a standard deviation of 5.6%.

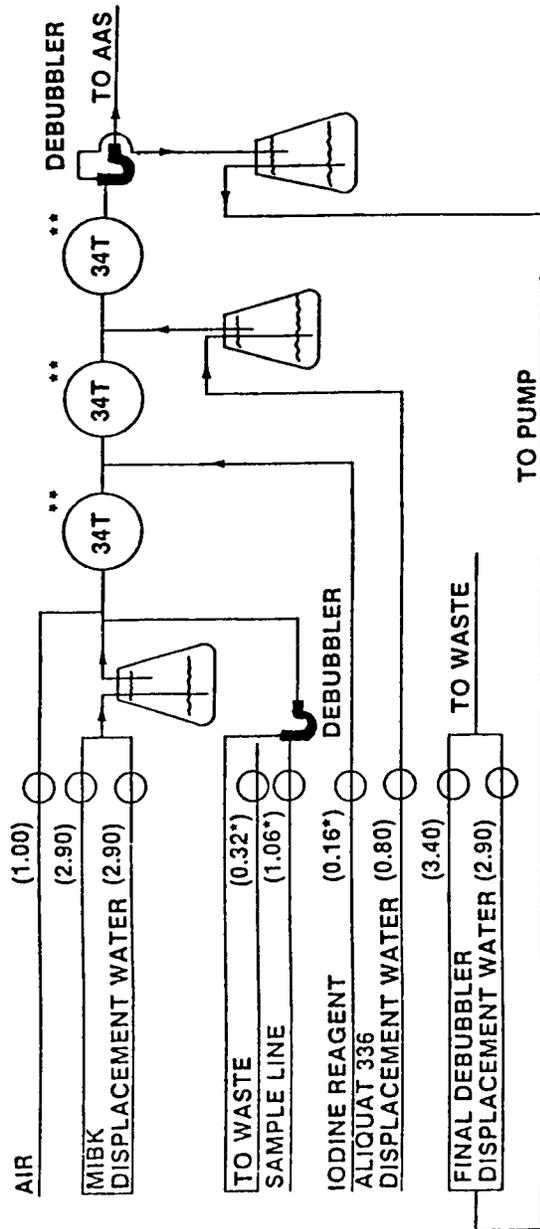


Figure 1

Flow Diagram for the Automated System. (*: Solvaflex pump tubing, **: Technicon part no. 157-0225) Numbers in parentheses refer to the flow rate (ml/min.).

METHOD 3—TEST FOR LEAD IN GASOLINE BY X-RAY SPECTROMETRY

1. Scope and application.

1.1 This method covers the determination of the total lead content of gasoline. The procedure's calibration range is 0.010 to 5.0 grams of lead/U.S. gallon. Samples above this level should be diluted to fall within the range of 0.05 to 5.0 grams of lead/U.S. gallon. The method compensates for variations in gasoline composition and is independent of lead alkyl type.

1.2 This method may be used as an alternative to Method 1—Standard Method Test for Lead in Gasoline by Atomic Absorption Spectrometry, or to Method 2—Automated Method Test for Lead in Gasoline by Atomic Absorption Spectrometry.

1.3 Where trade names or specific products are noted in the method, equivalent apparatus and chemical reagents may be used. Mention of trade names or specific products is for the assistance of the user and does not constitute endorsement by the U.S. Environmental Protection Agency.

2. Summary of method.

2.1 A portion of the gasoline sample is placed in an appropriate holder and loaded into an X-ray spectrometer. The ratio of the net X-ray intensity of the lead L alpha radiation to the net intensity of the incoherently scattered tungsten L alpha radiation is measured. The lead content is determined by reference to a linear calibration equation which relates the lead content to the measured ratio.

2.2 The incoherently scattered tungsten radiation is used to compensate for variations in gasoline samples.

3. Sample handling and preservation.

3.1 Samples should be collected and stored in containers which will protect them from changes in the lead content of the gasoline, such as loss of volatile fractions of the gasoline by evaporation or leaching of the lead into the container or cap.

3.2 If samples have been refrigerated they should be brought to room temperature prior to analysis.

3.3 Gasoline is extremely flammable and should be handled cautiously and with adequate ventilation. The vapors are harmful if inhaled and prolonged breathing of vapors should be avoided. Skin contact should be minimized. See precautionary statements in Annex Al.3.

4. Apparatus.

4.1 X-ray Spectrometer, capable of exciting and measuring the fluorescence lines mentioned in 2.1 and of being operated under the following instrumental conditions or others giving equivalent results: a tungsten

target tube operated at 50 kV, a lithium fluoride analyzing crystal, an air or helium optical path and a proportional or scintillation detector.

4.2 Some manufacturers of X-ray Spectrometer units no longer allow use of air as the beam path medium because the X-ray beam produces ozone, which may degrade seals and electronics. In addition, use of the equipment with liquid gasoline in close proximity to the hot X-ray tube could pose flammability problems with any machine in case of a rupture of the sample container. Therefore, use of the helium alternative is recommended.

5. Reagents.

5.1 Isooctane. Isooctane is flammable and the vapors may be harmful. See precautions in Annex Al.1.

5.2 Lead standard solution, in isooctane, toluene or a mixture of these two solvents, containing approximately 5 gm Pb/U.S. gallon may be prepared from a lead-in-oil concentrate such as those prepared by Conostan (Conoco, Inc., Ponca City, Oklahoma). Isooctane and toluene are flammable and the vapors may be harmful. See precautionary statements in Annex Al.1 and Al.2.

6. Calibration.

6.1 Make exact dilutions with isooctane of the lead standard solution to give solutions with concentrations of 0.01, 0.05, 0.10, 0.50, 1.0, 3.0 and 5.0 g Pb/U.S. gallon. If a more limited range is desired as required for linearity, such range shall be covered by at least five standard solutions approximately equally spaced and this range shall not be exceeded by any of the samples. Place each of the standard solutions in a sample cell using techniques consistent with good operating practice for the spectrometer employed. Insert the sample in the spectrometer and allow the spectrometer atmosphere to reach equilibrium (if appropriate). Measure the intensity of the lead L alpha peak at 1.175 angstroms, the Compton scatter peak of the tungsten L alpha line at 1.500 angstroms and the background at 1.211 angstroms. Each measured intensity should exceed 200,000 counts or the time of measurement should be at least 30 seconds. The relative standard deviation of each measurement, based on counting statistics, should be one percent or less. The Compton scatter peak given above is for 90° instrument geometry and should be changed for other geometries. The Compton scatter peak (in angstroms) is found at the wavelength of the tungsten L alpha line plus 0.024 (1-cos phi), where phi is the angle between the incident radiation and the take-off collimator.

6.2 For Each of the standards, as well as for an isooctane blank, determine the net lead intensity by subtracting the corrected

background from the gross intensity. Determine the corrected background by multiplying the intensity of the background at 1.211 angstroms by the following ratio obtained on an isoctane blank:

$$\frac{\text{Background at 1.175 angstroms}}{\text{Background at 1.211 angstroms}}$$

$$R = \frac{\text{Lead L alpha-background}}{\text{Incoherent tungsten L alpha-corrected background}}$$

6.4 Obtain a linear calibration curve by performing a least squares fit of the corrected lead intensity ratios to the standard concentrations.

7. Procedure.

7.1 Prepare a calibration curve as described in 6. Since the scattered tungsten radiation serves as an internal standard, the calibration curve should serve for at least several days. Each day the suitability of the calibration curve should be checked by analyzing several National Bureau of Standards (NBS) lead-in-reference-fuel standards or other suitable standards.

7.2 Determine the corrected lead intensity ratio for a sample in the same manner as was done for the standards. The samples should be brought to room temperature before analysis.

7.3 Determine the lead concentration of the sample from the calibration curve. If the sample concentration is greater than 5.0 g Pb/U.S. gallon or the range calibrated for in 6.1, the sample should be diluted so that the result is within the calibration span of the instrument.

7.4 Quality control standards, such as NBS standard reference materials, should be analyzed at least once every testing session.

7.5 For each group of ten samples, a spiked sample should be prepared by adding a known amount of lead to a sample. This known addition should be at least 0.05 g Pb/U.S. gallon, at least 50% of the measured lead content of the unspiked sample, and not more than 200% of the measured lead content of the unspiked sample (unless the minimum addition of 0.05 g Pb/U.S. gallon exceeds 200%). Both the spiked and unspiked samples should be analyzed.

8. Quality control.

8.1 The difference between duplicates should not exceed 0.005 g Pb/U.S. gallon or a relative difference of 6%.

6.3 Determine the corrected lead intensity ratio, which is the net lead intensity corrected for matrix effects by division by the net incoherently scattered tungsten radiation. The net scattered intensity is calculated by subtracting the background intensity at 1.211 angstroms from the gross intensity of the incoherently scattered tungsten L alpha peak. The equation for the corrected lead intensity ratio follows:

8.2 All quality control standard check samples should agree within 10% of the nominal value of the standard.

8.3 All spiked samples should have a percent recovery of 100% ±10%. The percent recovery, P, is calculated as follows:

$$P = 100 \times (A - B) / K$$

where

A = the analytical result from the spiked sample, B = the analytical result from the unspiked sample, and K = the known addition.

8.4 The difference between independent analyses of the same sample in different laboratories should not exceed 0.01 g Pb/U.S. gallon or a relative difference of 12%.

9. Past quality control data.

9.1 Duplicate analysis for 26 samples in the range of 0.01 to 0.10 g Pb/U.S. gallon resulted in an average relative difference of 5.2% with a standard deviation of 5.4%. Duplicate analysis of 14 samples in the range 0.1 to 0.5 g Pb/U.S. gallon resulted in an average relative difference of 2.3% with a standard deviation of 2.0. Duplicate analysis of 47 samples in the range of 0.5 to 5 g Pb/U.S. gallon resulted in an average relative difference of 2.1% with a standard deviation of 1.8%.

9.2 The average percent recovery for 23 spikes made to samples in the 0.0 to 0.1 g Pb/U.S. gallon range was 103% with a standard deviation of 3.2%. For 42 spikes made to samples in the 0.1 to 5.0 g Pb/U.S. gallon range, the average percent recovery was 102% with a standard deviation of 4.2%.

9.3 The analysis of National Bureau of Standards lead-in-reference-fuel standards of known concentrations in a single laboratory has resulted in found values deviating from the true value for 14 determinations of 0.0490 g Pb/U.S. gallon by an average of 2.8% with a standard deviation of 6.4%, for 11 determinations of 0.065 g Pb/U.S. gallon by an average of 4.4% with a standard deviation of 2.9%, and for 15 determinations of 1.994 g Pb/U.S. gallon by an average of 0.3% with a standard deviation of 1.3%.

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9.4 Eighteen analyses of reference samples (U.S. EPA, Research Triangle Park, NC) have resulted in found values differing from the true value by an average of 0.0004 g Pb/U.S. gallon with a standard deviation of 0.004 g Pb/U.S. gallon.

ANNEX

A1. Precautionary Statements

A1.1 Isooctane

Danger—Extremely flammable. Vapors harmful if inhaled.
Vapor may cause flash fire.
Keep away from heat, sparks, and open flame.
Vapors are heavier than air and may gather in low places, resulting in explosion hazard.
Keep container closed.
Use adequate ventilation.
Avoid buildup of vapors.
Avoid prolonged breathing of vapor or spray mist.
Avoid prolonged or repeated skin contact.

A1.2 Toluene

Warning—Flammable. Vapor harmful.
Keep away from heat, sparks, and open flame.

Keep container closed.
Use with adequate ventilation.
Avoid breathing of vapor or spray mist.
Avoid prolonged or repeated contact with skin.

A1.3 Gasoline

Danger—Extremely flammable. Vapors harmful if inhaled.
Vapor may cause flash fire.
Keep away from heat, sparks, and open flame.
Vapors are heavier than air and may gather in low places, resulting in explosion hazard.
Keep container closed.
Use adequate ventilation.
Avoid buildup of vapors.
Avoid prolonged breathing of vapor or spray mist.
Avoid prolonged or repeated skin contact.

[39 FR 24891, July 8, 1974; 39 FR 25653, July 12, 1974; 39 FR 26287, July 18, 1974, as amended at 47 FR 765, Jan. 7, 1982; 52 FR 259, Jan. 5, 1987; 56 FR 13768, Apr. 4, 1991]

APPENDIXES C-G TO PART 80
[RESERVED]