



Designation: D 526 - 70



Designation: 96/67

American National Standard Z11.48
American National Standards Institute
Method 5501—Federal Test
Method Standard No. 791b
Deutsche Norm Din 51 769 Blatt 1 Und Blatt 3
British Standard 2878

DISCONTINUED

Standard Method of Test for LEAD IN GASOLINE, GRAVIMETRIC METHOD¹

This Standard is issued under the fixed designation D 526; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. This is also a standard of the Institute of Petroleum issued under the fixed designation IP:96. The final number indicates the year of last revision.

This method was adopted as a joint ASTM-IP standard in 1964.

1. Scope

1.1 This method covers the gravimetric determination of the total lead content of gasoline and other volatile distillates blended with lead alkyls (tetraethyllead, tetramethyllead, dimethyldiethyllead, methyltriethyllead, or mixtures thereof) within the concentration range of 0.2 to 5.0 g of lead/U.S. gal, 0.24 to 6.0 g lead/U.K. gal, or 0.05 to 1.3 g lead/liter.

NOTE 1—ASTM Method D 2547, Test for Lead in Gasoline, Volumetric Chromate Method,² is also available for this test.

NOTE 2—The method has not been cooperatively tested below 0.2 g/U.S. gal.

2. Summary of Method

2.1 The lead alkyl is converted to lead chloride and extracted from the gasoline by refluxing with concentrated hydrochloric acid. The acid extract is evaporated to dryness, any organic material present is removed by oxidation with nitric acid, and the lead is determined gravimetrically as lead chromate.

3. Apparatus

3.1 *Extraction Apparatus*, fabricated from borosilicate glass, conforming to the dimensions given in Fig. 1, and consisting of the following component parts:

3.1.1 *Boiling Flask*, 500-ml capacity.

3.1.2 *Hopkins Reflux Condenser*, having a vapor outlet connected by a rubber tube to an outside vent or to a suction hood.

3.1.3 *Thistle Tube*, approximately 70-ml capacity, with a line to indicate approximately the 50-ml level.

3.1.4 *Heating Tube*, containing a chimney

for increasing convection in the liquid.

3.1.5 *Heating Coil*, 250-W, consisting of 9 ft (2.4 m) of No. 30 B & S gage (0.25 mm) or equivalent Nichrome wire.

3.1.6 *Rheostat*, 25- Ω resistance, 2-A minimum capacity, for regulating the heater.

3.2 *Filtering Crucible*, of approximately 25-ml capacity No. 4 porosity sintered glass, or 25-ml porcelain crucibles, having porous bottoms equivalent to Selas No. 3001, or 25-ml Gooch crucibles, capable of retaining a fine precipitate.

4. Reagents and Materials

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.³ 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 in-

¹ This method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants.

Current edition effective Sept. 11, 1970. Originally issued 1939. Replaces D 526-66.

In the IP, this method is under the jurisdiction of the Standardization Committee.

In 1970 the scope of the method was extended down to 0.2 g of lead per U.S. gallon.

² 1974 Annual Book of ASTM Standards, Part 24.

³ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, N.Y., and the "United States Pharmacopeia."

licated, references to water shall be understood to mean lead-free distilled water or water of equal purity.

4.3 *Acetic Acid (1+1)*—Mix 1 volume of glacial acetic acid with 1 volume of water.

4.4 *Ammonium Hydroxide (1+1)*—Mix 1 volume of concentrated ammonium hydroxide (NH_4OH , sp gr 0.9) with 1 volume of water.

4.5 *Asbestos*—Medium - fiber, acid-washed, and ignited for use with Gooch crucibles.

NOTE 3—Test the asbestos used under the conditions of the analysis to determine its weight loss due to solubility or to mechanical disintegration. If necessary, pick over the asbestos by hand to remove coarse material, and then acid-wash prior to use.

4.6 *Heavy Distillate*—A straight-run, lead-free, petroleum distillate, of low bromine number, with approximately 10 percent distilling at 400 F (205 C) and 90 percent at 460 F (240 C).

4.7 *Hydrochloric Acid (sp gr 1.19)*—Concentrated hydrochloric acid (HCl).

4.8 *Nitric Acid (sp gr 1.42)*—Concentrated nitric acid (HNO_3).

4.9 *Nitric Acid (1+20)*—Mix 1 volume of HNO_3 (sp gr 1.42) with 20 volumes of water.

4.10 *p-Nitrophenol Indicator Solution*—Dissolve 0.5 g of *p*-nitrophenol in 100 ml of water, and filter if necessary to remove insoluble material.

4.11 *Potassium Chlorate-Nitric Acid Solution*—Dissolve 78 g of potassium chlorate (KClO_3) in 550 ml of HNO_3 (sp gr 1.42).

4.12 *Potassium Dichromate Solution (100 g/liter)*—Dissolve 100 g of potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) in water, dilute to 1 liter, and filter.

5. Procedure

5.1 Measure the temperature of the sample to the nearest 1.0 F (0.5 C) (Note 4). Using a pipet, transfer 50 ± 0.05 -ml of the sample of the gasoline to the flask (Fig. 1) through the thistle tube, and add approximately 50 ml of heavy distillate. Add 50 ml of HCl and reflux the mixture for 30 min. Use the full heat of the heater until boiling has begun (usually 0.5 to 1.0 min); then adjust the rheostat to regulate the heat to maintain boiling at a vigorous rate, but not at such a rate to cause bumping in the flask (Note 5) or to cause the condenser to flood (Note 6). After the 30-min reflux pe-

riod, turn off the heat, allow the sample to cool a few minutes, and drain the acid layer into a 400-ml beaker. Then add 50 ml of water and reflux the water and gasoline for 5 min, using the full heat of the heater. Drain the water into the 400-ml beaker, and repeat the water extraction.

NOTE 4—For gasolines having a Reid vapor pressure above 7.0 lb, the sealed sample container shall be cooled to approximately 60 F (15 C) before removing sample for analysis.

NOTE 5—A new or thoroughly cleaned extractor has a tendency to induce super-heating and bumping of the solution may occur. This difficulty is reduced and boiling will continue at an even rate after the apparatus has been used for several analyses.

NOTE 6—During the heating, hydrochloric acid gas is volatilized and constant boiling hydrochloric acid is obtained. Continuous and vigorous boiling is necessary to obtain intimate mixing of the acid and sample to extract completely the decomposed lead alkyls from the gasoline phase.

5.2 Evaporate the aqueous extract to dryness (Note 7). Add 3 ml of HNO_3 (sp gr 1.42) to the residue, cover the beaker with a watch glass, and heat to oxidize any organic material present (Note 8). Repeat the HNO_3 treatment. If a white residue is not obtained after two additions of HNO_3 , oxidize the remaining organic matter with the KClO_3 - HNO_3 mixture as described in Note 8. Then add 4 ml of HNO_3 (1+20) and 25 ml of water; heat until all the lead salt is in solution.

NOTE 7—To reduce the evaporation time it is permissible to employ an air jet under the following conditions: substitute a 500-ml Erlenmeyer flask for the 400-ml beaker; evaporate on a hot plate whose surface temperature is maintained between 230 and 260 C, while impinging upon the surface of the liquid a stream of hot (about 75 C) clean air at a rate of about 10 liters/min. The air stream should be led into the flask by means of a glass tube with an orifice about 5 mm in diameter, placed about 60 mm above the surface of the liquid. The air stream should not be used in the HNO_3 evaporation.

NOTE 8—If the residue flashes on being heated with HNO_3 , the sample should be discarded and the acid extraction repeated on another sample of gasoline. Then evaporate the extract until crystallization commences, but not to complete dryness. Add 10 ml of KClO_3 - HNO_3 mixture, cover the beaker with a watch glass, and evaporate the mixture almost to dryness. Repeat this treatment, if necessary, to obtain a white residue.

5.3 Cool the sample, add 6 drops of *p*-nitrophenol indicator solution, and add NH_4OH until the indicator changes color; then add approximately 4 to 5 ml in excess. Add acetic acid to neutralize the NH_4OH :