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An American National Standard

Standard Test Method for Hydrogen In Petroleum Fractions¹

This standard is issued under the fixed designation D 1018; 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. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

¹ This test method is under the jurisdiction of ASTM Committee D-2 D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.03 on Elemental Analysis.

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1. Scope

1.1 This test method covers the determination of hydrogen in petroleum fractions that can be burned completely without smoking in a wick lamp.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.3 The preferred values stated in SI units are acceptable metric units, to be regarded as the standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1266 Test Method for Sulfur in Petroleum Products (Lamp Method)²

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products³

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products³

D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance⁴

3. Summary of Test Method

3.1 The test specimen is burned from a cotton wick in an atmosphere of purified air. The water formed is collected from the combustion gases by a desiccant and weighed.

4. Significance and Use

4.1 Knowledge of the hydrogen content of petroleum products, particularly fuels, can be helpful in assessing performance characteristics.

4.2 This test method is suitable for most laboratory applications requiring the determination of hydrogen in liquid petroleum lubricants and products.

5. Apparatus

5.1 *Lamp*—A lamp (see Note 1) consisting of a 25-mL Erlenmeyer flask, a burner, and a burner cap conforming to the dimensions shown in Fig. 1. The burner consists of two concentric glass tubes, the external one having an arm, provided with standard-taper glass joints for connection with the flask, chimney, and burner cap. The burner has a small opening near its base to allow equalization of the pressure between the chimney and flask.

NOTE 1—The lamp and burner are constructed in accordance with Appendix A3 of Test Method D 1266.

5.2 *Chimney*—A chimney (see Note 2) conforming to the dimensions shown in Fig. 1.

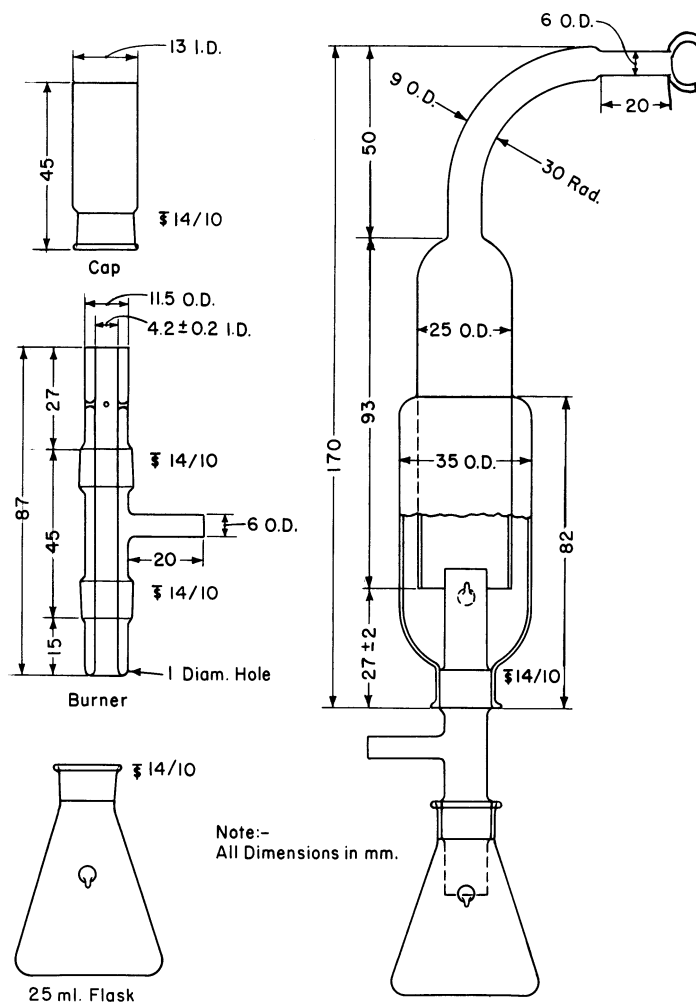
NOTE 2—The chimney is constructed in accordance with Appendix A3 of Test Method D 1266, except that the delivery tube is cut off and a short length of glass tubing is sealed on at right angles to the chimney. The secondary air inlet is not used and is sealed off.

² *Annual Book of ASTM Standards*, Vol 05.01.

³ *Annual Book of ASTM Standards*, Vol 05.02.

⁴ Turner-type absorption bulbs are satisfactory for this method.

⁴ *Annual Book of ASTM Standards*, Vol 05.03.



Note:- All Dimensions in mm.

NOTE 1—In the case of those dimensions for which no specific tolerances are designated above, the permissible variation is ± 10 percent to the nearest 1 mm, provided, however, that in no case shall the deviation be greater than 5 mm.

FIG. 1 Lamp and Chimney

5.3 *Absorbers*—Two absorption bulbs⁵ suitable for collecting and weighing the water formed during combustion of the sample. The bulbs shall be filled in the following manner: a 1 to 2-cm layer of glass wool, a 5-cm layer of 6 to 10-mesh cp anhydrous calcium chloride, a 1-cm layer of glass wool, a 2-cm layer of phosphorus pentoxide, and glass wool to the top of the absorbers. One filling is sufficient for approximately 10 g of water.

5.4 *Cotton Wicking*—Clean, unused, uniform, two-strand twisted cotton wicking of good quality, weighing 0.5 to 0.6 g/m per strand.

5.5 *Air Purifying System*—The compressed air (**Warning**—See Note 3)—Compressed gas under higher pressure) for the combustion shall be purified by passage through a scrubber containing sulfuric acid (relative density 1.84), a surge chamber packed with glass wool, and a drying tower filled with 10 to 20-mesh anhydrous calcium sulfate⁶ and phosphorus pentoxide in that order. A length of rubber tubing suitable for connection to the inlet tube of the burner shall be connected to the exit of the tower.

Note 3—**Warning:** Compressed gas under higher pressure.

5.6 *Drying Tube*—A small U-tube containing anhydrous calcium sulfate.⁶

5.7 *Ice-Water Bath*—large enough to hold the two absorbers in tandem, immersed to a depth of approximately 5 cm.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all

⁴“Drierite”[®], available from Fisher Scientific, 711 Forbes Avenue, Pittsburgh, PA 15219, has been found

⁵Turner-type absorption bulbs are satisfactory for this purpose: test method.

⁶“Reagent Chemicals, American Chemical Society Specifications,” American Chemical Society, Washington, DC. 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, NY, and the “United States Pharmacopeia.”

⁶“Drierite,” available from Fisher Scientific, 711 Forbes Avenue, Pittsburgh, PA 15219, has been found satisfactory for this purpose.

reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁷ 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.

6.2 Calcium chloride (CaCl₂)₂.

6.3 Calcium sulfate, anhydrous (CaSO₄)₂⁵.

6.4 Phosphorous pentoxide (P₂O₅)₂.

6.5 Sulfuric acid (H₂SO₄), relative density 1.84.

6.6 Quality Control (QC) Samples, preferably, portions of one or more liquid petroleum materials that are stable and representative of the samples of interest. These QC samples can be used to check the validity of the testing process, as described in Section 10.

7. Sampling

7.1 ~~Take test units~~

7.1 Obtain samples in accordance with the instructions in Practices D 4057 or D 4177. ~~As~~ Ensure that test specimens are representative of the test unit. Vigorous stirring or shaking may be necessary.

8. Procedure

8.1 Connect the charged absorbers to each other and to the chimney delivery tube as shown in the complete assembly of the apparatus in Fig. 2. Attach the U-tube containing anhydrous calcium sulfate⁶ to the exit end of the second absorber to eliminate diffusion of water vapor back into the system. Immerse the lower half of the absorbers into the ice-water bath, connect the air flow system to the chimney, and purge the system with air for 15 min (**Warning**—See Note 3.)—see 5.5.) At the end of the purging period, turn off the air flow, close the absorbers, and remove them from the train. Allow the absorbers to reach room temperature, gently wipe each with a lintfree dry cloth, and place them beside the balance case for 30 min or more. Open the absorbers for a moment to equalize the pressure, close, and weigh to the nearest 1 mg.

8.2 While the absorbers are standing, the lamp may be prepared. Thread the burner with the proper number of wick strands (see Note 4 3) by drawing the strand through the burner tube with a small metal hook. Trim the wick as closely as possible to the top of the burner, and draw the trimmed wick down until it is flush with or slightly below the top of the burner. Pipet approximately 5 mL of test specimen into the burner flask, and insert the prepared burner into the flask. Cap the burner with its glass cap, and close the burner air inlet with a rubber policeman. Allow the sample to rise by capillary action to the top of the wick.

NOTE 43—The burning characteristics of the flame are dependent upon the flow of air to the burner, the volatility of the test specimen, the tightness of the wick in the burner tube, and the position of the wick relative to the top of the burner. The latter two factors can be established before an actual determination is made so that the flame can be controlled by the rate of air flow. A tightly fitting wick is required for highly volatile samples; also, the wick may have to be drawn down several millimetres below the top of the burner tube. Extremely volatile test specimens (**Warning**—Extremely flammable.) may require immersion of the burner flask in an ice-water bath during the burning period. A more loosely fitting wick is required for less volatile test specimens, and in some instances the burner flask may require warming to ~~assure~~ ensure an even flow of test specimen fuel to the burner. In any case, the flame must burn smoothly and symmetrically without jets in the inner cone or smoke on the outer fringes.

8.3 Connect the weighed absorbers to the chimney tube, attach the Drierite tube, and immerse the absorbers in the ice-water bath.

⁷There is no extant ASTM Research Report

⁷ Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analar Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset-m, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

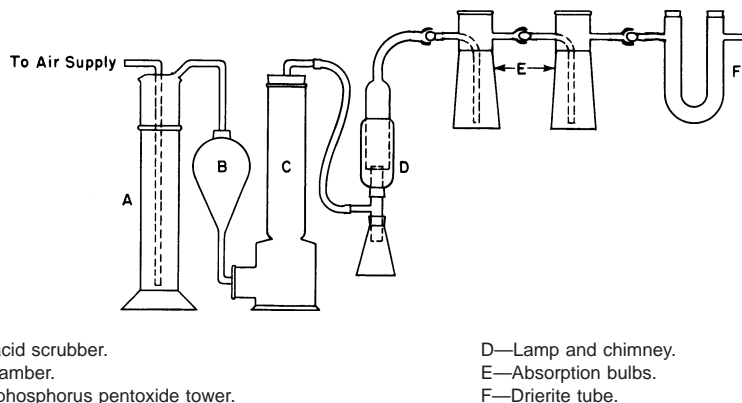


FIG. 2 Apparatus Assembly

8.4 Weigh the assembled lamp quickly to the nearest 1 mg and bring it immediately to the absorption train. Open the absorbers in the train. Remove the cap and rubber policeman from the lamp, and connect the air flow to the burner. Ignite the lamp with a microburner, and rapidly adjust the air flow until a symmetrical nonsmoking flame is obtained. Immediately insert the lamp in the chimney, using rubber bands or small coil springs to hold the lamp in place. Record to the nearest second the time that elapsed between ignition of the burner and insertion of the lamp in the chimney as lost burning time. The flame may require further minor adjustment in rate of air flow after it has been inserted in the chimney.

8.5 After the desired amount (2 to 3 g) of test specimen has been burned, interrupt the air flow, immediately remove the burner from the chimney, cap it, and replace the rubber policeman. Note and record the time for the complete analysis. As quickly as possible, connect the air line to a clean, thoroughly dry, assembled lamp and insert it in the chimney.

8.6 Weigh the lamp containing the test specimen to the nearest 1 mg.

8.7 Purge the system with purified air for a period of 5 min. If any condensed water is visible in the chimney delivery tube, gently heat the tube with a microburner until all moisture has been vaporized and swept into the absorbers. At the end of the purging period, turn off the air flow, close the absorbers, and remove them from the train. Allow the absorbers to reach room temperature, wipe each with a lint-free dry cloth, equilibrate, and weigh to the nearest 1 mg.

9. Calculation

9.1 Correct the mass of water formed for the lost burning time as follows:

$$W = w \times [t/(t - s)] \quad (1)$$

where:

W = corrected grams of water collected,

w = grams of water absorbed,

t = time in seconds for complete analysis, and

s = lost burning time in seconds.

9.2 Calculate the hydrogen content of the sample as follows:

$$\text{Hydrogen, weight \%} = (W \times 11.191)/S \quad (2)$$

where:

W = corrected grams of water collected, and

S = grams of sample burned.

10. Quality Control

10.1 Confirm the performance of the test procedure by analyzing a QC sample (see 6.6).

10.1.1 When QC/Quality Assurance (QA) protocols are already established in the testing facility, these may be used to confirm the reliability of the test result.

10.1.2 When there is no QC/QA protocol established in the testing facility, Appendix X1 can be used as the QC/QA system.

11. Precision and Bias ⁸

11.1 The precision of this test method is not known to have been obtained in accordance with currently accepted guidelines (for example, in Committee D-2 D02 Research Report R:D2-1007, Manual on Determining Precision Data for ASTM Methods on Petroleum Products and Lubricants).³

11.2 The precision of this test method as obtained by statistical examination of interlaboratory test results is as follows:

11.2.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty:

Hydrogen Content, mass %	Repeatability
11 to 16	0.11

11.2.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty:

Hydrogen Content, mass %	Reproducibility
11 to 16	0.18

⁸ There is no extant ASTM Research Report for this test method.

101.3 *Bias*—The bias of this test method cannot be determined since an appropriate standard reference material containing a known level of hydrogen in liquid petroleum hydrocarbon is not available.

112. Keywords

112.1 hydrogen; lamp

APPENDIX

(Nonmandatory Information)

X1. QUALITY CONTROL

X1.1 Confirm the performance of the instrument or the test procedure by analyzing a quality control (QC) sample.

X1.2 Prior to monitoring the measurement process, the user of the test method needs to determine the average value and control limits of the QC sample (see Practice D 6299).^{9,10}

X1.3 Record the QC results, and analyze by control charts or other statistically equivalent techniques to ascertain the statistical control status of the total testing process (see Practice D 6299).^{9,10} Any out-of-control data should trigger investigation for root cause(s).

X1.4 In the absence of explicit requirements given in the test method, the frequency of QC testing is dependent on the criticality of the quality being measured, the demonstrated stability of the testing process, and customer requirements. Generally, a QC sample is analyzed each testing day with routine samples. The QC frequency should be increased if a large number of samples are routinely analyzed. However, when it is demonstrated that the testing is under statistical control, the QC testing frequency may be reduced. The QC sample precision should be checked against the ASTM test method precision to ensure data quality.¹⁰

X1.5 It is recommended that, if possible, the type of QC sample that is regularly tested be representative of the material routinely analyzed. An ample supply of QC sample material should be available for the intended period of use, and must be homogeneous and stable under the anticipated storage conditions.

⁹ ASTM MNL 7: Manual on Presentation of Data Control Chart Analysis, 6th Ed.

¹⁰ "TQA in the Petroleum and Lubricant Testing Laboratories." Available from ASTM Headquarters.

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