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An American National Standard

Standard Practice for Isolation of Representative Saturates Fraction from High-Olefinic Petroleum Naphthas[1]

This standard is issued under the fixed designation D 2003; 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.

1. Scope

1.1 This practice covers the isolation of a representative saturates fraction from depentanized hydrocarbon mixtures that distill below 221°C (430°F). It is applicable for samples containing from 0 to 35 volume % olefinic hydrocarbons. However, for samples that contain less than 1 volume % of olefinic hydrocarbons the procedures given in Test Method D 2002, are recommended.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety problems, 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.* Specific precautionary statements are given in Notes 1, 2, 3, 4, and 5.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1319 Test Method for Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption[2]

D 2002 Test Methods for Isolation of Representative Saturates Fraction from Low-Olefinic Petroleum Naphthas[3]

3. Terminology

3.1 *Description of a Term Specific to This Standard:*

3.1.1 *saturates fraction*—the portion of the sample obtained from the silica gel fractionation procedure described in this practice, and essentially a mixture of paraffinic and naphthenic hydrocarbons.

4. Summary of Practice

4.1 A measured amount of sample is charged to the top of a glass column packed with fine activated silica gel. The adsorbed sample is then desorbed with alcohol, with the saturated hydrocarbons which have the lowest adsorption affinity issuing from the bottom of the silica gel column first. The total percolate collected prior to the elution of the yellow fluorescing olefinic marker is representative of the saturates in the sample.

5. Significance and Use

5.1 The determination of paraffins and naphthenes by the refractivity intercept requires a sample free from olefins and aromatics. This practice is applicable for the isolation of saturates fractions from samples containing up to 35 volume % olefins and 65 volume % total olefins plus aromatics.

6. Apparatus

6.1 *Adsorption Column,* as shown in Fig. 1.

6.2 *Receiver,* graduated, of capacity sufficient to collect the total saturates as one fraction. The inside diameter of the graduated portion should not exceed 10 mm.

6.3 *Vibrator,* for packing silica gel.

6.4 *Hypodermic Syringe,* for charging sample.

6.5 *Ultraviolet Light Source,* with radiation predominantly at 3650 Å.

6.6 *Refrigerant Circulation System,* to circulate liquid cooled to 4 to 16°C (40 to 60°F) through the adsorption column jacket.

7. Reagents

7.1 *Isopropyl Alcohol* (**Warning**—See Note 1).

NOTE 1: **Warning**—Flammable.

7.2 *Pressuring Gas* (**Warning**—See Note 2), delivered to the top of the column at a regulated pressure.

NOTE 2: **Warning**—Compressed gas.

7.3 *Silica Gel,* 149 to 74 μm (100 to 200-mesh).[4]

7.4 *Standard Dyed Gel.*[5]

8. Procedure

8.1 Clean the adsorption column with chromic-sulfuric acid (**Warning**—See Note 3), water, and acetone (**Warning**—See Note 4).

NOTE 3: **Warning**—Causes severe burns. A recognized carcinogen.
NOTE 4: **Warning**—Extremely flammable. Vapors may cause fire.

8.2 Introduce a small metallic or glass pellet, approximately 1.6 mm (1/16 in.) in diameter, to prevent the flow of silica gel from the bottom of the column, and sufficiently nonspherical to permit liquid elution.

8.3 Clamp the column in a vertical position and pack the

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[1] This practice is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.04 on Hydrocarbon Analysis.

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

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

[4] Silica gel, available from W. R. Grace and Co., Davison Chemical Division, Baltimore, MD 21203, by specifying Code 923, has been found satisfactory for this purpose.

[5] The standard dyed gel may be obtained from UOP Process Div., Organics Dept., 25 E. Algonquin Rd., Des Plaines, IL 60017-5017, by requesting "FIA Standard Dyed Gel", UOP Product No. 675.

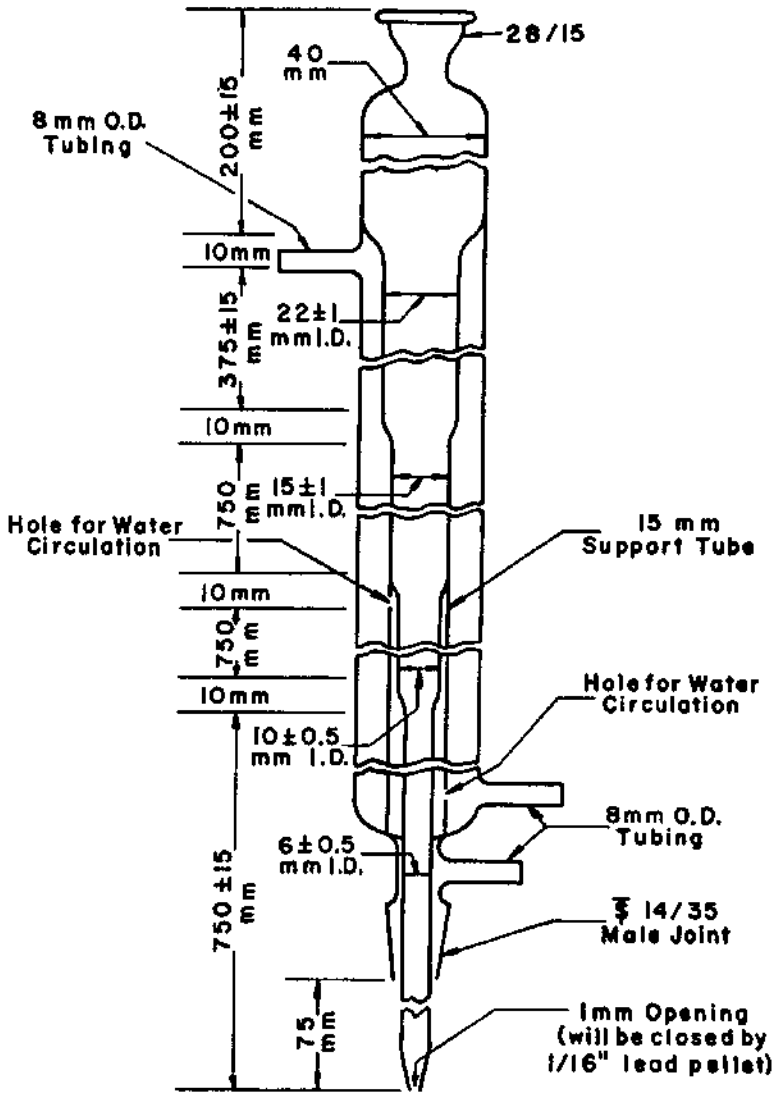

FIG. 1 High Efficiency Silica Gel Column

column by adding silica gel slowly through a funnel while applying the vibrator (or tapping with a padded rod). When the silica gel surface is within approximately 50 mm of the top of the 6-mm section, add approximately 0.5 mL of standard dyed gel. Continue adding silica gel and applying vibration, or tapping, until the surface reaches the top of the 22-mm section and does not drop noticeably with continued vibrating or tapping.

8.4 The quantity of sample (**Warning**—See Note 5) that may be charged is dependent upon the volume of aromatics plus olefins in the sample, the column efficiency, and the gel capacity of the column. The volume of aromatics plus olefins in the charge should not exceed 20 mL which corresponds to 1 mL/18 mL of silica gel. The recovery of a maximum quantity of the saturates charged is required to ensure representation. Thus, the charge should be large enough that the effect of the loss of saturates due to holdup at the column tip is minimized. It is recommended that an FIA analysis, see Test Method D 1319, be made prior to this test. The FIA results should be used to determine the volume of charge necessary to comply with the above conditions and to assure that sufficient saturates will be obtained for the desired physical property determinations.

NOTE 5: **Warning**—Flammable.

8.5 Chill the sample (**Warning**—See Note 5) and a hypodermic syringe of sufficient size to 2 to 4°C (34 to 40°F). Draw the sample into the syringe and adjust the level to the volume desired. Discharge the total sample with the tip of syringe needle inserted 20 to 30 mm below the surface of the silica gel. Add sufficient silica gel to raise the level 10 mm. Record the volume charged to the nearest 0.1 mL.

NOTE 1—Accurate values for the volume charged and the volume of saturates recovered are not required as they are used only to determine that a satisfactorily high percentage of the available saturates are recovered. Accordingly, to avoid unnecessary handling of the sample both measurements are made volumetrically on chilled samples.

8.6 Fill the remaining portion of the column with isopropyl alcohol (**Warning**—See Note 5) and apply nitrogen pressure (**Warning**—See Note 6) to move the liquid down the column. Usually 69 kPa gage (10 psig) is sufficient pressure to obtain elution of 10-mL saturates from a 30-mL