Designation: D 5661 – 95 (Reapproved 2001)

Standard Test Method for Relative Solvency of Petroleum Oils by the PKP Method[1]

This standard is issued under the fixed designation D 5661; 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 test method covers a procedure for determining the relative solvency of petroleum oils used in ink formulations by a pentaerythritol ester of resin acids (PKP)[2] titration.

1.2 This test method is applicable to petroleum oils that have an initial boiling point over 90°C and a dry point under 500°C as determined by Method D 86.

1.3 This test method, along with viscosity measurements as determined by Test Method D 445, is used to ensure the compositional consistency of petroleum oils. It can also differentiate between hydrotreated and non-hydrotreated oils that have the same viscosity.

1.4 This test method includes the use of a U.S. Occupational Safety and Health Administration (OSHA)—designated flammable chemical, pentane. Consult the suppliers' material safety data sheet for specific hazard information and guidance relative to use.

1.5 *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.* Specific hazard statements are given in 1.3.

2. Referenced Documents

2.1 *ASTM Standards:*

D 86 Test Method for Distillation of Petroleum Products at Atmospheric Pressure[3]

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (the Calculation of Dynamic Viscosity)[3]

D 611 Test Methods for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents[3]

D 841 Specification for Nitration Grade Toluene[4]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *PKP value of a solvent*—the volume in millilitres, at 25 ± 2°C, of pentane required to produce a defined degree of turbidity of a mixture containing 10 g of the test oil and 5 g of a standard solution of a pentaerythritol ester of resin acids in toluene.

4. Summary of Test Method

4.1 A 40 % solution by weight of PKP in toluene is standardized in two steps by mixing with toluene and titrating with pentane, and also by mixing with a toluene-heptane solution and titrating with pentane until a turbidity end point is reached.

4.2 The standardized PKP solution is mixed with the test oil and titrated with pentane. The test result is expressed as millilitres of pentane.

5. Significance and Use

5.1 PKP values indicate high aromatic or high naphthenic content, or both, which contributes to high relative solvency of the oil.

6. Apparatus

6.1 *Magnetic Stirrer*, with stir bar.

6.2 *Erlenmeyer Flask*, 1000-mL capacity.

6.3 *Glass Beaker*, 250-mL capacity.

6.4 *Burette*, 50-mL capacity.

6.5 *Print Specimen*, such as a 152 by 152 mm sheet of newspaper with 10-point type.

6.6 *Analytical Balance*, 100-g capacity, with reproducibility of 0.01 g.

6.7 *Sieve Screen*, 10 mesh.

7. Reagents

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

[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.37 on Ink Vehicles.

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[2] Pentalyn®K (PKP) is a pentaerythritol ester of dimeric resin acids and is available from Hercules Incorporated, Resins Group, Hercules Plaza, Wilmington, DE 19894.

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

[4] *Annual Book of ASTM Standards*, Vol 06.04.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[6] 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.

7.2 *PKP Solution* (40 %)—Weigh out 200 g of fresh (less than 60 days old) high-dilutability Pentalyn®K flakes,[4] which have been sieved through a No. 10 mesh screen to remove fines. Add this to a 1000-mL Erlenmeyer flask containing 300 g of toluene, and mix. The flakes dissolve slowly; however, placing the flask in an ultrasonic water bath speeds up the process. The solution is allowed to sit for at least three days, with frequent stirring, to allow it to equilibrate.

7.3 *Toluene*, conforming to Specification D 841, for use as a high-solvency standard.

7.4 *Heptane—Toluene Blend*, consisting of 25 ± 0.1 % toluene and 75 ± 0.1 % *n*-heptane on a weight basis, for use as a low-solvency standard. The heptane shall conform to the requirements for knock test grade *n*-heptane prescribed in Table 1 of Test Methods D 611.

7.5 *Pentane*, reagent grade.[6]

8. Standardization

8.1 Weigh out 5.00 ± 0.01 g of the PKP solution into a 250-mL glass beaker. Add 10.00 ± 0.01 g of toluene. Place the beaker on a magnetic stirrer, with the small sheet of newspaper directly under the beaker. Add pentane from the burette while stirring the solution in the beaker. Reduce the increments of pentane gradually as the endpoint nears. The endpoint is reached when the individual letters of the words on the printed page are no longer readable. In other words, the letters blur together. Record the millilitres of pentane required. This pentane volume is value *A* in the equation.

8.2 The volume of pentane used, in millilitres, represents the actual titer for the particular PKP solution at hand. This value should not be over 70 mL nor under 60 mL. If these limits are exceeded, discard this portion of solution, prepare another PKP solution, and re-standardize. Obtain another batch of solid PKP if this portion still exceeds the above limit.

8.3 Weigh out 5.00 ± 0.01 g of the PKP solution into a 250-mL glass beaker. Add 2.50 ± 0.01 g of toluene and 7.50 ± 0.01 g *n*-heptane. Titrate with pentane as just described. Record the millilitres of pentane required. This pentane volume is value *B* in the equation.

9. Procedure

9.1 Weigh out 5.00 ± 0.01 g of PKP solution and 10.00 ± 0.01 g of the ink oil or solvent into a 250-mL glass beaker. Titrate with pentane as just described, and record the volume. This pentane volume is value *C* in the equation.

NOTE 1—When these tests are first run, the titrations may have to be repeated several times for the analyst to get a feel for the test.

NOTE 2—The PKP solution is standardized daily or for each series of tests. It does not have to be standardized for tests run concurrently during one day.

10. Calculation and Report

10.1 Calculate the PKP value, *V*, as follows:

$$V = 43.3\,\frac{(C - B)}{(A - B)} + 25.8 \qquad (1)$$

where:

A = pentane to titrate PKP solution and toluene, mL,
B = pentane to titrate PKP solution and toluene-heptane, mL, and
C = pentane to titrate PKP solution and sample, mL.

11. Precision and Bias

11.1 *Precision*—The precision for this test method was determined by an interlaboratory study in which four petroleum oil samples were analyzed in triplicate by six laboratories ranging in PKP values from 24 to 29. The data were analyzed according to Practice E 691 guidelines.

11.2 *Repeatability*—The repeatability standard deviation is 0.082 (*sr*). At a 95 % confidence level, two results obtained by the same operator should be considered suspect if they differ by a 0.232 PKP value.

11.3 *Reproducibility*—The reproducibility standard deviation is 1.05 (*SR*). At a 95 % confidence level, two results obtained by operators in different laboratories should be considered suspect if they differ by a 2.94 PKP value.

11.4 *Bias*—Bias cannot be determined because there are no reference oils that have known PKP values.

11.5 Supporting documents for the precision statements can be found in RR: D01-1089.[7]

12. Keywords

12.1 ink oils; quality control

[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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[7] Supporting data is available from ASTM Headquarters. Request RR: D01-1089.