Designation: D 6892 – 03

An American National Standard

Standard Test Method for Pour Point of Petroleum Products (Robotic Tilt Method)[1]

This standard is issued under the fixed designation D 6892; 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 the determination of the pour point of petroleum products by an automatic instrument that tilts the test jar to detect movement of the surface of the test specimen with an optical device, after being removed from a regulated, stepped-bath cooling jacket.

1.2 This test method is designed to cover the range of temperatures from −57 to +51°C; however, the range of temperatures included in the 1998 interlaboratory test program only covered the temperature range from −51 to −11°C.

1.3 Test results from this test method can be determined at either 1 or 3°C testing intervals.

1.4 This test method is not intended for use with crude oils.

NOTE 1—The applicability of this test method on residual fuel samples has not been verified. For further information on the applicability, refer to 13.4.

1.5 The values stated in SI units are regarded as standard.

1.6 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 97 Test Method for Pour Point of Petroleum Products[2]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[3]

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products[3]

2.2 *IP Standard:*

IP 15 Test Method for Pour Point of Petroleum Products[4]

3. Terminology

3.1 *Definitions:*

3.1.1 *pour point, n—in petroleum products,* the lowest temperature at which movement of the test specimen is observed under prescribed conditions of test.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *no-flow point, n—in petroleum products,* the temperature of the test specimen at which a wax crystal structure or viscosity increase, or both, impedes movement of the surface of the test specimen under the conditions of the test.

3.2.1.1 *Discussion*—The no-flow point occurs when, upon cooling, the formation of wax crystal structures or viscosity increase, or both, has progressed to the point where the applied observation device no longer detects movement under the conditions of the test. The preceding observation temperature at which flow of the test specimen is last observed is the pour point.

3.2.2 *tilting, vt*—technique of movement where the test jar in a vertical position is moved towards a horizontal position to induce specimen movement.

3.2.2.1 *Discussion*—When the test jar is tilted and held in a horizontal position for 5 s without detection of movement of the surface of the specimen, this is the no-flow point and the test is complete.

4. Summary of Test Method

4.1 After insertion of the specimen into the automatic pour point apparatus and initiation of the testing program, the specimen is heated and then cooled according to a prescribed profile. The specimen surface is examined periodically for movement using an optical camera system mounted on top of the specimen test jar, while tilting the specimen test jar. The test jar is removed from the jacketed cooling chamber prior to each examination. The lowest temperature, when movement of the surface of the specimen is detected, is recorded as the pour point determined by this Test Method D 6892.

5. Significance and Use

5.1 The pour point of a petroleum product is an index of the lowest temperature of its utility for certain applications. Flow characteristics, such as pour point, can be critical for the correct operation of lubricating systems, fuel systems, and pipeline operations.

5.2 Petroleum blending operations require precise measurement of the pour point.

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

Current edition approved July 10, 2003. Published September 2003.

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

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

[4] Available from Institute of Petroleum (IP), 61 New Cavendish St., London, WIG 7AR, U.K.

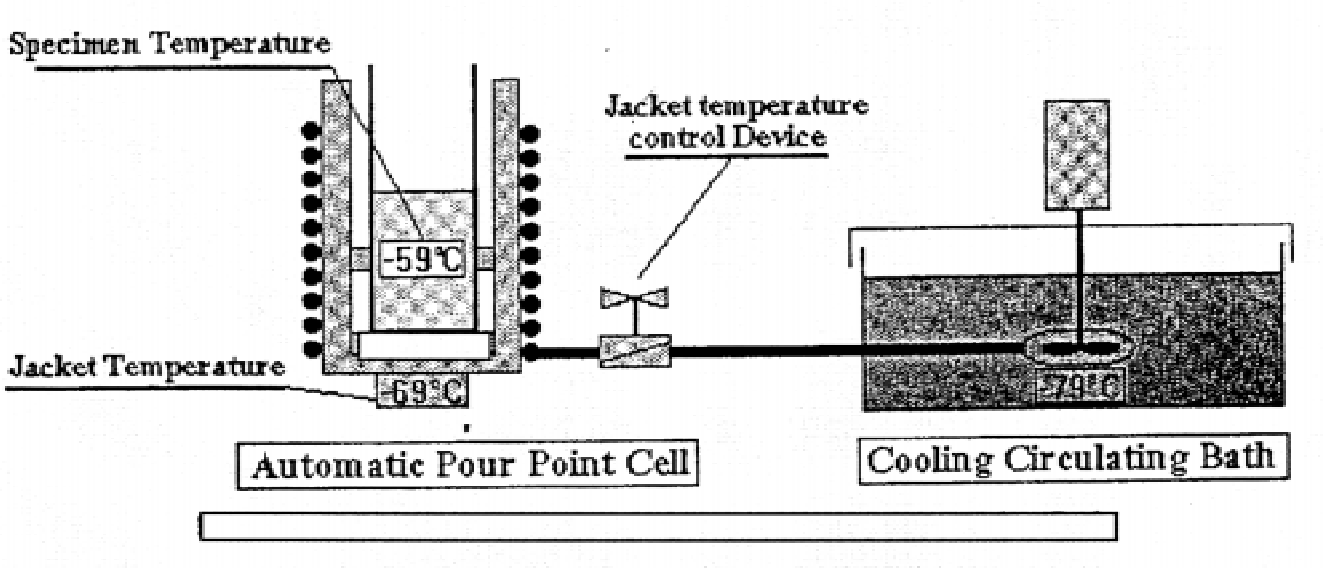

FIG. 1 Schematic of Cooling/Heating Block and Cooling Circulating Bath

5.3 Test results from this test method can be determined at either 1 or 3°C intervals.

5.4 This test method yields a pour point in a format similar to Test Method D 97 or IP 15, when the 3°C interval results are reported.

NOTE 2—Since some users may wish to report their results in a format similar to Test Method D 97 or IP 15 (in 3°C intervals) the precision data were derived for the 3°C intervals. For statements on bias relative to Test Method D 97 or IP 15, see the research report.

5.5 This test method has comparable repeatability and better reproducibility relative to Test Method D 97 or IP 15 as measured in the 1998 interlaboratory program.[5]

6. Apparatus

6.1 *Automatic Apparatus*[6]—The automatic pour point apparatus described in this test method (see Fig. 2) consists of a microprocessor controlled measuring unit that is capable of heating the specimen to programmed temperatures, cooling the specimen according to programmed cooling profiles, mechanically manipulating the test jar according to the programmed test procedure, while optically observing the surface of the specimen for movement, using a camera system mounted on top of the specimen test jar and recording the temperature of the specimen. The apparatus shall be equipped with a user interface, cooling/heating block assembly with cylindrical jacket with an inside diameter of 44.2 to 45.8 mm, and about 115 mm in depth to accept the test jar) robotic mechanisms for lifting, tilting, replacing the test jar, optical camera system, and a temperature measuring device.

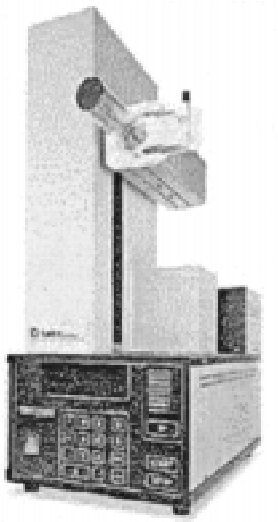

FIG. 2 Picture of Apparatus

6.2 *Test Jar*—Clear, cylindrical glass, flat bottom (darkened), 31.5 ± 0.5 mm inside diameter and 120 ± 2 mm height with a wall thickness of 1.25 ± 0.25 mm. The jar shall be marked with a line to indicate sample filling height corresponding to 45 ± 0.5 mL.

6.3 *Temperature Probe*—Capable of measurement from +70 to −80°C with a resolution of 0.1°C. The temperature probe shall be suspended in the center axis of the test jar and the top of the temperature sensing zone immersed below the surface of the specimen.

6.4 *Circulating Bath*—Refrigeration unit, equipped with a circulating pump, capable of maintaining the liquid cooling medium at a temperature at least 20°C lower than the lowest expected pour point to be measured. The circulating bath is

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1499.

[6] The sole source of supply of the Herzog Model MP 852 or HCP 852 known to the committee at this time is Walter Herzog, Lauda, Germany. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

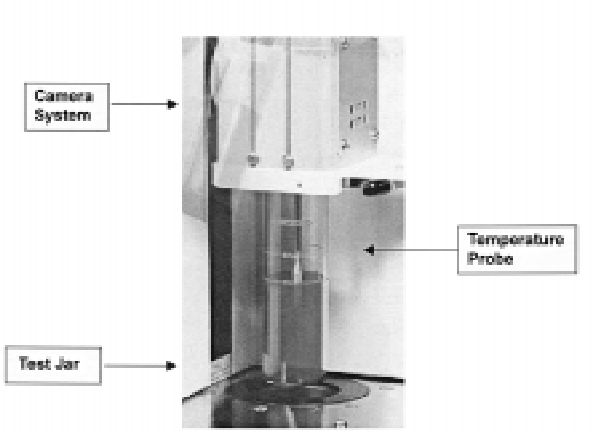

FIG. 3 Assembled Apparatus

connected to the automatic apparatus through suitable means for supplying the liquid cooling medium.

7. Reagents and Materials

7.1 *Bath Cooling Medium*—Suitable for use in the circulating bath (an example is methyl alcohol-anhydrous). (**Warning—**Flammable. Liquid causes eye burns. Vapor harmful. May be fatal or cause blindness if swallowed or inhaled.)

7.2 *Cleaning Solvents*—Suitable for cleaning and drying the test jar and temperature measuring device, such as; petroleum naphtha or acetone. (**Warning—**Flammable. Liquid causes eye burns. Vapor harmful. May be fatal or cause blindness if swallowed or inhaled.)

8. Sampling

8.1 Obtain a sample in accordance with Practice D 4057 or Practice D 4177.

8.2 Samples of very viscous materials may be warmed until they are reasonably fluid before they are tested. However, no sample shall be heated more than is absolutely necessary.

8.3 The sample shall not be heated and transferred into the test jar unless its temperature is 70°C or lower. When the sample is heated above 70°C, allow the sample to cool below 70°C before transferring into the test jar.

9. Preparation of Apparatus

9.1 Prepare the instrument for operation in accordance with the manufacturer's instructions.

9.2 Select the cooling/heating block temperature settings and the cooling/heating block change over temperature settings, in accordance with Table 1.

9.3 Clean and dry the test jar using suitable solvents.

9.4 Prepare the refrigerated circulating bath for operation and allow it to attain a temperature at least 20°C lower than the expected pour point of the sample.

10. Calibration and Verification

10.1 Ensure that all of the manufacturer's instructions for calibrating, checking, and operating the apparatus are followed.

10.2 A sample with a well-documented pour point can be used to verify the performance of the automatic apparatus. Alternatively, a sample which has been extensively tested in a pour point cross- check program can be used. Such verification materials can also be prepared from intra-company cross checks.

11. Procedure

11.1 Fill the test jar up to the marked line with the specimen. When necessary, heat the sample in a water bath or oven until it is just sufficiently fluid to pour into the test jar.

NOTE 3—Residual fuels have been known to be sensitive to thermal history. In the case where a residual fuel sample is tested, refer to Test Method D 97 for sample treatment.

11.2 Insert the test jar into the apparatus and start the test in accordance with the manufacturer's instructions.

11.3 When the expected pour point is known to be above −33°C, preselect a starting temperature which is at least 9°C above the expected pour point, but to at least 45°C.

11.4 When the expected pour point is known to be at or below −33°C, preselect a starting temperature of 45°C.

11.5 When the expected pour point is not known, preselect a starting temperature of 45°C. When the expected pour point is not known and the sample must be heated to allow transfer into the test jar, preselect a starting temperature corresponding to the preheat temperature. (**Warning—**Exercise care when selecting starting temperatures above 45°C. Samples which are fluid at ambient room temperature can also have a low temperature flash point. Use higher start temperatures only on samples known to be solid near ambient room temperature.)

11.6 Preselect the testing interval of 1 or 3°C as determined by your standard laboratory practice. Should the user wish to provide results with a similar format to Test Method D 97 or IP 15, then testing at a 3°C interval shall be selected.

11.7 Once the operation of the apparatus is initiated, the specimen is heated to the temperature preselected by the operator. The cooling/heating block shall be regulated in accordance to the programmed temperature settings obtained from Table 1. The instrument shall automatically change the block temperature in accordance with the specimen temperature (according to Table 1). The time required to move the jacket temperature from one temperature level to the next lower level shall not exceed 180 s.

11.8 Beginning at the preselected start testing temperature, the test jar shall be lifted out of the block assembly, tilted toward a horizontal position, until movement of the surface of the specimen is detected by the optical system, and then returned to the block assembly. This complete operation shall take no longer than 3 s when specimen surface movement is observed. This operation shall be repeated at each subsequent lower temperature interval that has been preselected by the operator. The operations shall be repeated until the test jar is tilted horizontally for longer than 3 s and no movement of the surface of the specimen is detected for a maximum of 5 s. If movement is detected between 3 to 5 s, the test jar is returned to the block assembly and the operations continued.

11.9 Record the temperature measured at the last tilting interval as the no-flow point.

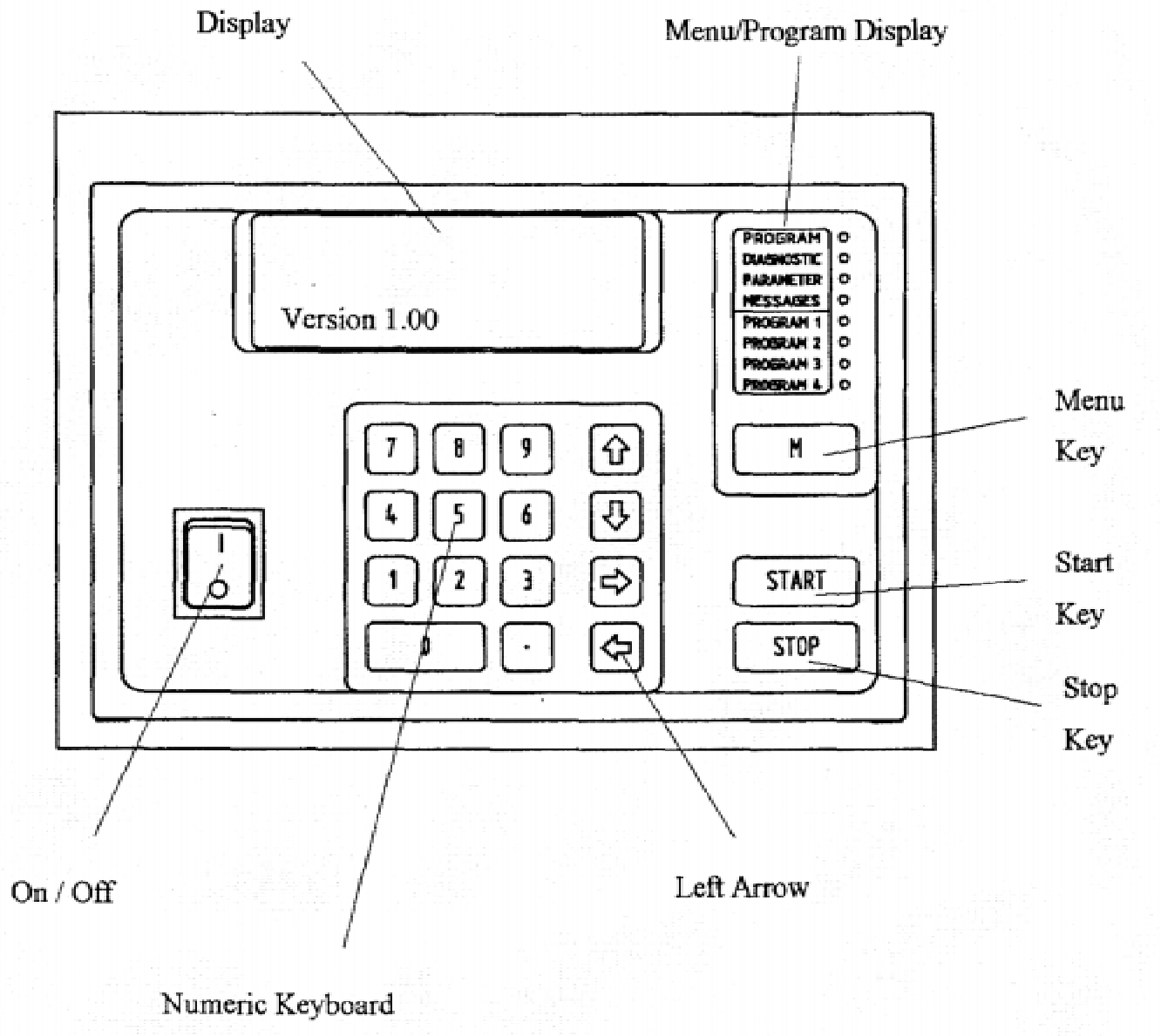

FIG. 4 Interface Panel

TABLE 1 Block and Specimen Temperature

Specimen Temperature °C	Block Temperature °C
ambient to 45	65 ± 2
45 ≥ ST > 27	15 ± 1
27 ≥ ST > 9	0 ± 1
9 ≥ ST > −6	−18 ± 1
−6 ≥ ST > −24	−33 ± 1
−24 ≥ ST > −42	−51 ± 1.5
−42 ≥ ST > −60	−69 ± 1.5
−60 ≥ ST > −78	−87 ± 1.5

NOTE 4—Some apparatus are capable of returning the test jar to the block assembly and the specimen can be reheated to a previously programmed temperature to facilitate disposal and cleaning.

11.10 Remove the test jar from the apparatus and clean the test jar and apparatus with suitable solvents and then dry with clean air according to the manufacturer's instructions.

12. Report

12.1 Increase the temperature recorded in 11.9 by a temperature amount equal to the testing interval used, and report the result including the testing interval as the pour point by this Test Method D 6892.

NOTE 5—Some apparatus are capable of automatically calculating and reporting these temperatures.

13. Precision and Bias

13.1 *Precision*—The precision of this test method as determined by statistical examination of interlaboratory test results is as follows:

13.1.1 *Pour Point at 3°C Testing Intervals*:

13.1.1.1 *Repeatability*—The difference between successive test results, obtained by the same operator using the same apparatus under constant operating conditions on identical test

material, would in the long run, in the normal and correct operation of this test method, exceed the following, only in one case in twenty:

3.2°C

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

3.6°C

13.1.2 *Pour Point at 1°C Testing Intervals*:

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

1.8°C

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

2.3°C

13.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test method, bias has not been determined.

13.3 *Relative Bias*:

13.3.1 Pour points at 3°C testing intervals were compared to the results from Test Method D 97. Relative bias among certain samples was observed. Based on the sample set used in the 1998 interlaboratory test program, the relative bias is not a fixed value but appears to be a linear function of the pour point value. Biases relative to Test Method D 97 or IP 15 may conceivably occur for sample types not included in the 1998 interlaboratory test program.

NOTE 6—For information on the bias relative to Test Method D 97 or IP 15, see the research report on the interlaboratory test program.

13.3.2 Pour point results at 1°C testing intervals were examined for bias relative to the pour point results at 3°C intervals. A bias of 1.1°C on average was observed.

NOTE 7—It shall be noted that when a specimen is tested at 1°C intervals, statistically the results will be 1°C lower than the results produced by 3°C testing intervals. This is due to test increment and reporting differences. Differences greater than 1°C over a number of samples would be from another cause. In the interlaboratory test program, the tests at 1°C intervals yielded pour points lower than those obtained from the tests at 3°C intervals by 1.1°C in average.

13.4 The precision statements were derived from a 1998 interlaboratory test program. Participants analyzed two sets of duplicate distillate diesel fuels, five sets of duplicate base oils, three sets of duplicate multigrade lubricating oils, and one set each of duplicate hydraulic oils and automatic transmission fluid in the temperature range of −51 to −11°C. Seven laboratories participated with the automatic apparatus testing at 1°C and six laboratories participated with the automatic apparatus testing at 3°C intervals, while seven laboratories participated with the manual Test Method D 97 apparatus. Information on the types of samples, and their average pour points, are available in research report RR:D02-1499.[5]

NOTE 8—Large differences in results were observed between methods for one sample in the 1998 interlaboratory test study.[5] The sample was a high-sulfur winter diesel. When cooled during the performance of a test method, this sample formed thin, but very large, crystals, that could be described as large plates. These crystals formed wherever sample-glass contact was made and covered the top surface of the sample as well. The entire sample, except for this all encasing thin skin of crystals, remained liquid with apparent low viscosity. When this occurred and the sample was handled gently, the sample did not pour, but with rougher handling, the crust broke and the sample poured readily. Users of this test method are advised to be alert for differences in results between test methods when this behavior is observed in the sample being tested.

14. Keywords

14.1 automatic pour point; petroleum products; pour point; robotic tilt method