Designation: D7346 − 15

Standard Test Method for No Flow Point and Pour Point of Petroleum Products and Liquid Fuels[1]

This standard is issued under the fixed designation D7346; 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 no flow point and pour point of petroleum products, liquid fuels, biodiesel, and biodiesel blends using an automatic instrument.

1.2 The measuring range of the apparatus is from –95 °C to 45 °C, however the precision statements were derived only from samples with no flow point temperatures from –77 °C to +2 °C and samples with pour point in the temperature range of –58 °C to +12 °C.

1.3 Pour point results from this test method can be reported at 1 °C or 3 °C intervals.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D97 Test Method for Pour Point of Petroleum Products

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants

D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material

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 the prescribed conditions of the 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 formation or viscosity increase, or both, is sufficient to impede 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, have progressed to the point where the applied observation device no longer detects movement under the conditions of the test.

4. Summary of Test Method

4.1 After inserting the test specimen into the automatic no flow point apparatus and initiating the program, the test specimen is heated, if necessary, to a starting temperature and then cooled by prescribed rates. The test specimen is continuously tested for flow characteristics by continuously monitoring the air pressure variation inside the test specimen vial. When the specimen is still fluid, its movement will partially compensate for the reduction in air pressure in the test chamber above the test specimen surface. At some temperature the pressure measuring system detects a pressure decrease due to incapability of the test specimen to flow caused by a crystal structure formation in the specimen or its viscosity increase, or both. This temperature is recorded as no flow point with a resolution of 0.1 °C. The pour point is recorded by rounding the no flow point temperature to either the next warmer 1 °C interval or 3 °C interval. The test specimen is then reheated to allow for removal from the test chamber.

5. Significance and Use

5.1 The no flow point of a petroleum product is an index of the lowest temperature of its utility for some applications. Flow

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

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

***A Summary of Changes section appears at the end of this standard**

characteristics, such as no flow point, can be critical for the proper operation of lubricating systems, fuel systems, and pipeline operations.

5.2 Petroleum blending operations require precise measurement of the no flow point.

5.3 This test method can determine the temperature of the test specimen with a resolution of 0.1 °C at which either crystals have formed or viscosity has increased sufficiently, or both, to impede flow of the petroleum product.

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

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

5.6 Pour point results from this test method can be reported at either 1 °C or 3 °C intervals.

5.7 This test method yields a pour point in a format similar to Test Method D97/IP15 when the 3 °C interval results are reported.

5.8 This test method has better repeatability and reproducibility relative to Test Method D97/IP15 as measured in the 2011 interlaboratory test program (see 13.1.2).

6. Apparatus (see Annex A1)

6.1 *Automatic No Flow Point Apparatus*[3]—The apparatus consists of a microprocessor–controlled test specimen chamber that is capable of heating and cooling the test specimen at required rate, measuring the pressure inside the test specimen vial, and recording the temperature of the test specimen chamber. A detailed description of the apparatus is provided in Annex A1.

6.2 The apparatus shall be equipped with a thermostatically controlled specimen chamber, digital display, cooling and heating systems, pressure measuring system, and a specimen chamber temperature measuring device.

6.3 The temperature measuring device in the specimen chamber shall be capable of measuring the temperature from –105 °C to 60 °C at a resolution of 0.1 °C.

6.4 *Ultrasonic Bath, Unheated—(optional),* with an operating frequency between 25 kHz to 60 kHz and a typical power output of ≤100 W, of suitable dimensions to hold container(s) placed inside of bath, for use in effectively dissipating and removing air or gas bubbles that can be entrained in viscous sample types prior to analysis. It is permissible to use ultrasonic baths with operating frequencies and power outputs outside this range, however it is the responsibility of the laboratory to conduct a data comparison study to confirm that results determined with and without the use of such ultrasonic baths does not materially impact results.

7. Reagents and Materials

7.1 *Specimen Vial*—Disposable, clear glass cylinder with closed flat bottom, 1 mL capacity.

Dimensions:
Outer diameter: 8.0 mm to 8.3 mm
Wall thickness: 0.75 mm to 0.85 mm
Outer length: 39.25 mm to 40.25 mm

NOTE 1—Standard NWV type vial was found suitable for the application.

7.2 *Specimen Vial Stopper*—Disposable, proprietary designed for use in this apparatus.

7.3 *Micropipette*—Capable of delivering 0.5 mL ± 0.1 mL of sample. Positive displacement type micropipette with capillary piston is preferred for use. Air-displacement type micropipettes are not recommended for viscous samples.

8. Sampling

8.1 Obtain a sample in accordance with Practice D4057 or D4177.

8.2 At least 1 mL of sample is required for each test.

8.3 Samples of very viscous materials can be warmed until they are reasonably fluid before they are transferred; however, no sample shall be heated more than is absolutely necessary. The sample shall not be heated and transferred into the test specimen cup unless its temperature is 70 °C or lower.

NOTE 2—In the event the sample has been heated above this temperature, allow the sample to cool until its temperature is below 70 °C before transferring it.

8.4 For some sample types, such as viscous lube oils that are prone to having entrained air or gas bubbles present in the sample, the use of an ultrasonic bath (see 6.4) without the heater turned on (if so equipped), has been found effective in dissipating bubbles typically within 1 min.

9. Preparation of Apparatus

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

10. Calibration and Standardization

10.1 Ensure that all of the manufacturer's instructions for calibration of the mechanical and electronic systems and operation of the apparatus are followed.

10.2 To verify the performance of the apparatus, a sample for which extensive data has been obtained by no flow point test method may be used. Such verification materials can also be prepared from intra-company cross checks.

11. Procedure

11.1 Draw 0.5 mL ± 0.1 mL of sample into a micropipette and transfer the specimen into a new clean, dry specimen vial. When necessary, heat the sample in a water bath or oven until it is just sufficiently fluid to transfer. Samples with an expected no flow point above 25 °C or which appear solid at room

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[3] The sole source of supply of the apparatus known to the committee at this time is ISL model MPP 5Gs Analyzer, available from ISL, B.P. 70285 14653 Verson, France. 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.

temperature can be heated above 45 °C but shall not be heated above 70 °C (see Note 2).

NOTE 3—Some samples like residual fuels, black oils, and cylinder stock have been known to be sensitive to thermal history. In the case where such sample is tested, refer to Test Method D97 for sample treatment prior testing.

11.2 Place a new clean, dry specimen vial stopper on the specimen vial and insert the assembly into the apparatus. Start the operation of the apparatus according to the manufacturer's instructions. When the expected no flow point of the specimen is known, program it in the apparatus as EP (expected point) and start test sequence. From this point up to and including the termination of the test, recording and reporting of the result, the apparatus automatically controls the procedure.

11.3 The apparatus adjusts the specimen chamber to a preselected starting temperature. By default, the preselected starting temperature is 25 °C. Alternatively, the operator can preprogram a defined starting temperature between 25 °C and 60 °C, if desired. When the expected no flow point is known and programmed in the apparatus, the starting temperature shall be at least 30 °C warmer. In the event that the preselected starting temperature is programmed lower than 30 °C above the expected no flow point, the apparatus shall heat the specimen chamber to a starting temperature at least 30 °C above the expected no flow point, but not more than 60 °C.

11.4 When the expected no flow point of the specimen is not known, once the starting temperature is reached (see 11.3) the specimen chamber is cooled at a rate of 1.5 °C/min ± 0.15 °C/min.

11.5 At the same time the cooling begins, the pressure measurement system is engaged to continuously monitor specimen behavior. When a decrease in pressure, as determined by the apparatus, is measured in the specimen vial, which signifies that the test specimen has ceased to flow due to a crystal structure formation in the specimen or its viscosity increase, or both, the temperature of the specimen chamber is recorded as the no flow point and held on a digital display. The test chamber is than reheated and the test sequence is terminated.

11.6 When a no flow point is detected prematurely, as determined by the apparatus, the specimen is automatically reheated to a higher starting temperature, at least 30 °C warmer than the temperature of premature detection, and then cooled as described in 11.4 until the no flow point is detected as described in 11.5.

NOTE 4—Some typical examples of premature no flow point are: when the NFP is detected during the fast cooling phase or when the NFP is detected less than 30 °C from the starting temperature.

11.7 When the expected no flow point of the specimen is known, and programmed into the apparatus, once the starting temperature (see 11.3) is reached, the specimen chamber is cooled at a rate of 10 °C/min ± 1 °C/min until 30 °C warmer than the programmed expected no flow point, then the cooling rate is adjusted to 1.5 °C/min ± 0.15 °C/min and the no flow point temperature is detected as described in 11.5.

11.8 When reporting of the pour point is desired, round the no flow point temperature in 11.5 to the next warmer 1 °C interval or 3 °C interval, as specified, and record. Some apparatus are capable of performing this automatically.

12. Report

12.1 Report the temperature recorded in 11.5 with resolution of 0.1 °C, as the no-flow point in accordance with Test Method D7346.

12.2 Report the temperature recorded in 11.8 as the pour point at either the 1 °C interval or 3 °C interval as specified in accordance with Test Method D7346.

13. Precision and Bias

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

13.1.1 *No Flow Point:*[4]

13.1.1.1 *Repeatability*—The difference between two 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 this test method, exceed the following only in one case in twenty.

1.93 °C Range: −77 °C to +2 °C

13.1.1.2 *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 this test method, exceed the following only in one case in twenty.

2.62 °C Range: −77 °C to +2 °C

13.1.2 *Pour Point at 3 °C Intervals:*[5]

13.1.2.1 *Repeatability*—The difference between two 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 this test method, exceed the following only in one case in twenty.

$$0.0384\,(50 - X) \quad \mathit{range}: -58\ \mathit{to} + 12 \tag{1}$$

X = result in °C.

13.1.2.2 *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 this test method, exceed the following only in one case in twenty.

$$0.0535\,(50 - X) \quad \mathit{range}: -58\ \mathit{to} + 12 \tag{2}$$

X = result in °C.

13.1.3 *Pour Point at 1 °C Intervals:*[5]

13.1.3.1 *Repeatability*—The difference between two results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would,

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[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1620.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1744.

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

$$0.0221\,(50 - X) \quad range\!: -58\ to + 12 \tag{3}$$

X = result in °C.

13.1.3.2 *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 this test method, exceed the following only in one case in twenty.

$$0.0411\,(50 - X) \quad range\!: -58\ to + 12 \tag{4}$$

X = result in °C.

13.2 *Bias*—Because there are no liquid hydrocarbon mixtures of known no flow point, bias cannot be established.

13.3 *Relative Bias*[5]—The Degree of Agreement between results by Test Method D7346 at 3 °C and 1 °C intervals and Test Method D97—Results on the same materials produced by Test Method D7346 at 3 °C and 1 °C intervals and Test Method D97 have been assessed in accordance with procedures outlined in Practice D6708. The findings are:

13.3.1 Pour points at 3 °C intervals were compared to the results from Test Method D97. The statistical analysis of the between method bias by Practice D6708 indicates that there is no correctable bias. Some sample specific bias was observed which may be random. The average difference estimated is 1.21 °C, which is not significantly different from zero, and there are some larger difference for some specific samples (see the research report[5] for further information).

13.3.2 Pour point results at 1 °C testing intervals were compared to the results from Test Method D97. The statistical analysis of the between method bias by Practice D6708 indicates that there is some statistical bias between Test Method D7346 1°C interval results and Test Method D97. Some sample specific bias was observed which may be random.

TABLE 1 Typical Calculated Precision (see Eqs 1 to 4)

	3 °C Interval		1 °C Interval	
°C	r	R	r	R
0	1.9	2.7	1.1	2.1
-9	2.3	3.2	1.3	2.4
-18	2.6	3.6	1.5	2.8
-27	3.0	4.1	1.7	3.2
-36	3.3	4.6	1.9	3.5
-45	3.6	5.1	2.1	3.9
-54	4.0	5.6	2.3	4.3

13.3.2.1 The degree of agreement between 1 °C interval results from Test Method D7346 and Test Method D97 can be further improved by applying the bias-correction outlined in Eq 5.

$$Bias - corrected\,X = predicted\,Y = X + 2.12\ \ °C \quad range - 58\ to + 12 \tag{5}$$

where:

X = result obtained by Test Method D7346 1 °C interval, and
Bias – corrected X = predicted Y = result that would have been obtained by Test Method D97 on the same sample.

NOTE 5—When the instrument is capable of applying internal corrections at specific temperature points, refer to the manufacturers instructions. Such corrections may be instrument specific, and should be developed from samples with a known Test Method D97 result, such as those from an interlaboratory study. It is advisable to maintain records of any corrections and is advisable to note in the report if any corrections were applied.

13.4 The precision statements for no flow point were derived according to Practice D6300 from a 2006 interlaboratory cooperative test program.[4] Participants analyzed blind replicates of 38 sample sets comprised of; base oils, formulated engine oils both mineral and synthetic, various lubricating oils, various aviation fuels, diesel fuels, fuel oils, biodiesel, and biodiesel blend over the temperature range of –77 °C to +2 °C. Thirteen laboratories participated with the automatic no flow point apparatus. The precision statistics were compiled and calculated based on the 0.1 °C resolution results. Information on the types of samples and their respective average no flow point is contained in the research report.[4]

13.5 The precision statements for pour point at 1 °C interval or 3 °C interval were derived from a 2011 interlaboratory cooperative test program.[5] Participants analyzed blind replicates of 20 sample sets comprised of; base oils both mineral and synthetic, formulated engine oils both mineral and synthetic, various lubricating oils, aviation turbine fuel, diesel fuels, biodiesel, and biodiesel blend over the temperature range of –58 °C to +12 °C. The samples were preheated to +45 °C to maintain consistent treatment relative to Test Method D97. Eleven laboratories participated with the automatic Test Method D7346 no flow point apparatus and ten laboratories participated with Test Method D97 apparatus. The precision statistics were compiled and calculated based on the 0.1 °C resolution results rounded to either the next warmer 1 °C interval or 3 °C interval. Information on the types of samples and their respective average no flow point is contained in the research report.[5] See Table 1.

14. Keywords

14.1 no flow point; petroleum products; pour point

ANNEX

(Mandatory Information)

A1. AUTOMATIC NO FLOW POINT APPARATUS

A1.1 *General*—The microprocessor controlled test apparatus, described in A1.2, and illustrated in Figs. A1.1 and A1.2.

A1.2 The apparatus shall be equipped with a thermostatically controlled specimen chamber, digital display, cooling and heating systems, pressure measuring system, and a specimen chamber temperature measuring device, arranged in a configuration as shown in Fig. A1.2.

A1.2.1 *Specimen Chamber*—Comprised of an aluminum block bored with a hole of 10.0 mm ± 0.2 mm on a length of 35 mm ± 1 mm. See Fig. A1.1.

A1.2.2 *Temperature Sensor*—Capable of reading to 0.1 °C over the range –105 °C to 60 °C with a maximum error of 0.1 °C. The sensor shall be embedded into the wall of the specimen chamber and located in a position to predict accurately the specimen temperature.

A1.2.3 *Cooling System*—An integral cooling system, capable of controlling the specimen chamber temperature within the range of anticipated test temperatures with a 0.1 °C accuracy.

A1.2.4 *Heating System*—Coupled to the specimen chamber capable of controlling the specimen chamber temperature within the range of anticipated test temperatures with a 0.1 °C accuracy.

A1.2.5 *Pressure Measuring System*—An electronic transducer connected to the inner tube of specimen vial stopper and capable of measuring the variation of air pressure above the test specimen surface up to 2 kPa with resolution of 1 Pa and accuracy of 5 Pa.

A1.2.6 *Apparatus Exterior Interface*—The exact layout may vary. A typical apparatus is shown as an example (see Fig. A1.3).

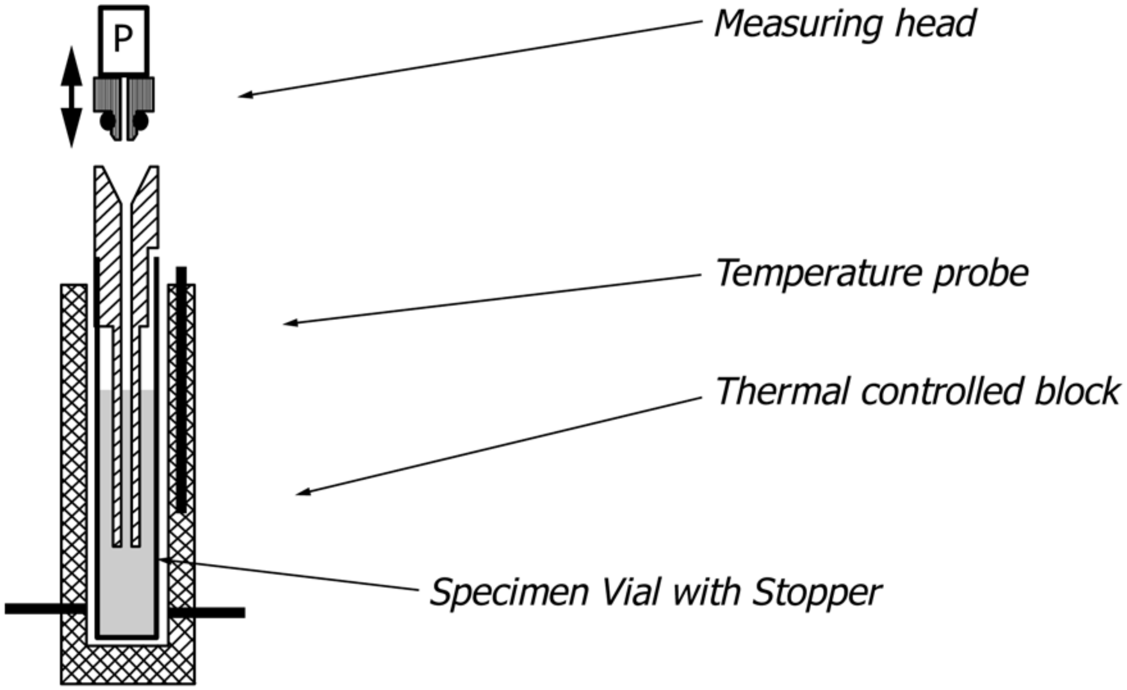

FIG. A1.1 Specimen Chamber

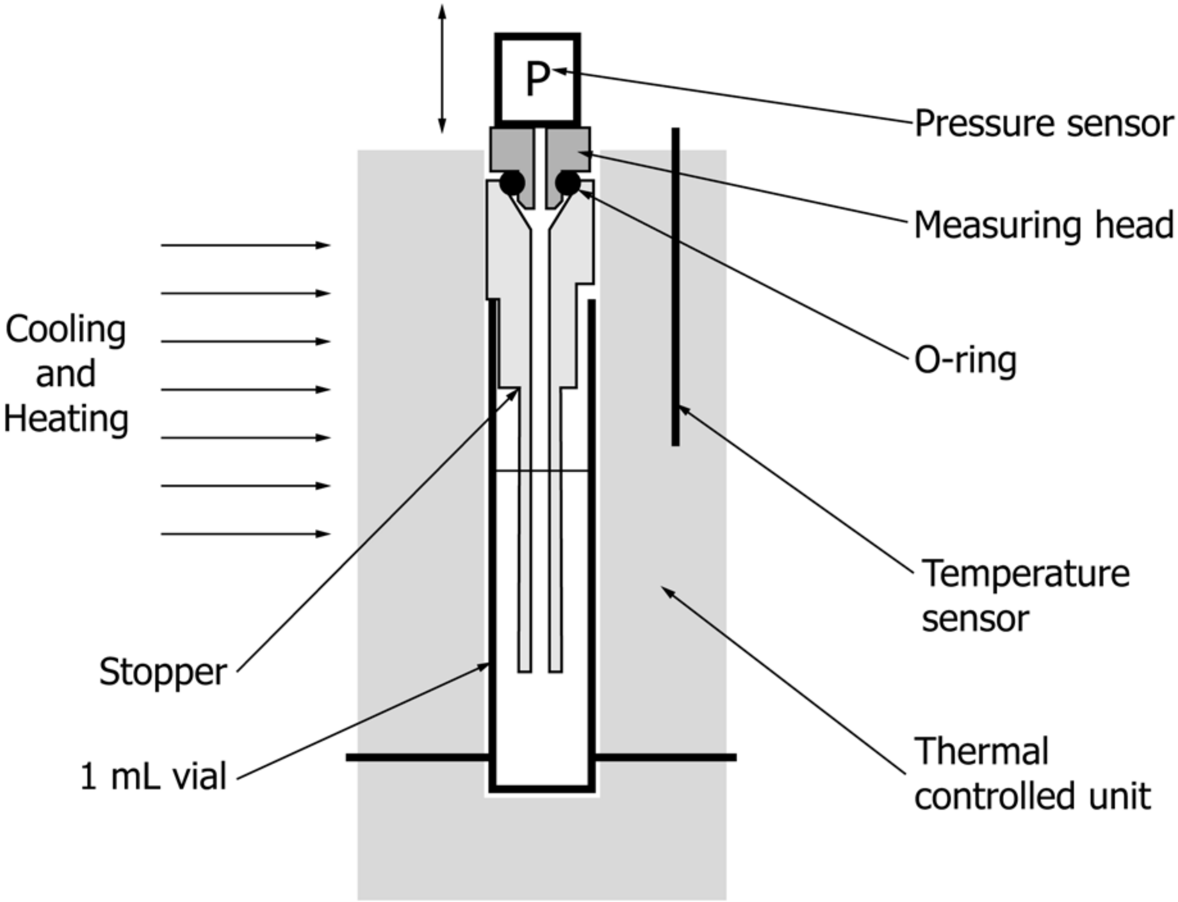

FIG. A1.2 Principle of Apparatus

FIG. A1.3 Automatic No Flow Point Apparatus

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D7346 – 14) that may impact the use of this standard. (Approved July 1, 2015.)

(1) Updated title of test method and subsection 1.1

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D7346 – 12) that may impact the use of this standard. (Approved May 1, 2014.)

(1) Added subsections 6.4 and 8.4.

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