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Designation: IP 446/09

Standard Test Method for Cloud Point of Petroleum Products and Liquid Fuels (Constant Cooling Rate Method)[1]

This standard is issued under the fixed designation D5773; 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.

INTRODUCTION

This test method describes an alternative procedure for the determination of cloud point of petroleum products Test Method D2500/IP 219 using an automatic apparatus. The temperature results from this test method have been found to be equivalent to Test Method D2500/IP 219. When specification requires Test Method D2500/IP 219, do not substitute this test method or any other method without obtaining comparative data and agreement from the specifier.

1. Scope*

1.1 This test method covers the determination of the cloud point of petroleum products and biodiesel fuels that are transparent in layers 40 mm in thickness by an automatic instrument using a constant cooling rate.

1.2 This test method covers the range of temperatures from −60 °C to +49 °C with temperature resolution of 0.1 °C, however, the range of temperatures included in the 1997 interlaboratory cooperative test program only covered the temperature range of –56 °C to +34 °C.

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

1.4 *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.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D2500 Test Method for Cloud Point of Petroleum Products and Liquid Fuels

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

2.2 *Energy Institute Standard:*[3]

IP 219 Test Method for Cloud Point of Petroleum Products

IP 446 Determination of the Cloud Point of Petroleum Products—Automatic Constant Cooling Rate Method

3. Terminology

3.1 *Definitions:*

3.1.1 *biodiesel, n*—a fuel comprised of mono-alkyl esters of long-chain fatty acids derived from vegetable oils or animal fats, designated B100.

3.1.1.1 *Discussion*—Biodiesel is typically produced by a reaction of vegetable oil or animal fat with an alcohol such as methanol or ethanol in the presence of a catalyst to yield mono-esters and glycerin. The fuel typically may contain up to 14 different types of fatty acids that are chemically transformed into fatty acid methyl esters (FAME).

3.1.2 *biodiesel blend, n*—a blend of biodiesel fuel with petroleum-based diesel fuel designated BXX, where XX is the volume percentage of biodiesel.

[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.

[3] Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., http://www.energyinst.org.uk.

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

3.1.3 *cloud point, n—in petroleum products and biodiesel fuels*, the temperature of a liquid specimen when the smallest observable cluster of wax crystals first occurs upon cooling under prescribed conditions.

3.1.3.1 *Discussion*—The cloud point occurs when the temperature of the specimen is low enough to cause wax crystals to precipitate. In a homogeneous liquid, the cloud is always noted first at the location in the specimen where the specimen temperature is the lowest. The cloud point is the temperature at which the crystals first occur, regardless of their location in the specimen, and not after extensive crystallization has taken place. The wax crystals that precipitate at lower temperatures are typically, but not excluded to, straight-chain hydrocarbons and lipids.

3.1.3.2 *Discussion*—The purpose of the cloud point is to measure the wax crystals in the specimen; however, trace amounts of water and inorganic compounds may also be present. The intent of the cloud point measurement is to capture the temperature at which the liquid fuel in the specimen begins to change from a single liquid phase to a two-phase system containing solid and liquid. It is not the intent of this test method to monitor the phase transition of the trace components such as water.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *automatic cloud point, n*—the temperature of a specimen, when the appearance of the cloud is determined under the conditions of this test method.

3.2.1.1 *Discussion*—The cloud point in this test method is determined by an automatic instrument using an optical device for detection of the crystal formation. The apparatus and the conditions are different from those established for Test Method D2500, although according to interlaboratory examination, the results have been determined to be equivalent to Test Method D2500.

3.2.2 *constant cooling rate method, n—in cloud point test methods*, test procedure using prescribed cooling rate, specimen receptacle, and optical system for detection of crystal formation.

3.2.2.1 *Discussion*—The prescribed cooling rate is described in 4.1, the specimen receptacle is described in Annex A1, and the optical system for the detection of crystal formation is described in Annex A1.

3.2.3 *Peltier device, n*—a solid-state thermoelectric device constructed with dissimilar semiconductor materials and configured in such a way that it will transfer heat to or away from a test specimen dependent on the direction of electric current applied to the device.

3.2.4 *D2500/IP 219 equivalent cloud point, n*—the temperature of a specimen, in integers, calculated by rounding the results of this test method to the next lower integer.

3.2.4.1 *Discussion*—This test method produces results with 0.1 °C resolution. Should the user wish to provide results with a similar format to Test Method D2500, then this calculation can be performed. Some apparatus can perform this calculation automatically.

4. Summary of Test Method

4.1 A prescribed specimen (11.5) is cooled by a Peltier device (A1.1) at a constant rate of 1.5 °C⁄min ± 0.1 °C⁄min while continuously being illuminated by a light source (A1.1.4). The specimen is continuously monitored by an array of optical detectors (A1.1.5, Fig. A1.1) for the first appearance of a cloud of wax crystals. The detectors are sufficient in number to ensure that any solid-phase hydrocarbon crystals that may form are detected. The temperature at which the appearance of a cloud of wax crystals is first detected in the specimen is recorded to 0.1 °C resolution. When the recorded temperature is rounded to the next lower integer temperature, it is designated as the D2500/IP 219 equivalent cloud point per Test Method D5773.

5. Significance and Use

5.1 The cloud point of petroleum products and biodiesel fuels is an index of the lowest temperature of their utility for certain applications. Wax crystals of sufficient quantity can plug filters used in some fuel systems.

5.2 Petroleum blending operations require a precise measurement of the cloud point.

5.3 This test method can determine the temperature of the test specimen at which wax crystals have formed sufficiently to be observed as a cloud with a resolution of 0.1 °C.

5.4 This test method provides results that are equivalent to Test Method D2500.

NOTE 1—This is based on the Test Method D2500 equivalent cloud point in which the 0.1 °C result is rounded to the next lower integer.

5.5 This test method determines the cloud point in a shorter period of time than Test Method D2500.

NOTE 2—In cases of samples with cloud points near ambient temperatures, time savings may not be realized.

5.6 This test method eliminates most of the operator time required of Test Method D2500.

5.7 This test method does not require the use of a mechanical refrigeration apparatus.

NOTE 3—In certain cases of high ambient temperature, a source of cooling water may be required to measure low-temperature cloud points (see 7.1).

6. Apparatus

6.1 *Automatic Apparatus*[4]—The automatic cloud point apparatus described in this test method consists of a test chamber controlled by a microprocessor that is capable of controlling

[4] The sole source of supply of the Phase Technology Cloud Point Analyzer model series 10, 30, 70, 70V, and 70X known to the committee at this time is Phase Technology, 11168 Hammersmith Gate, Richmond, B.C. Canada V7A 5H8. The various model series mentioned above are differentiated by their cooling capacities and user interfaces; however, all of them are capable of covering the entire temperature range specified in the scope. 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.

the heating and cooling of the test specimen, optically observing the first appearance of a cloud of wax crystals and recording the temperature of the specimen described in detail in Annex A1.

6.2 The apparatus shall be equipped with a specimen cup, optical detector array, light source, digital display, Peltier device, and a specimen temperature measuring device.

6.3 The Peltier device shall be capable of heating or cooling the test specimen at a constant rate of 1.5 °C⁄min ± 0.1 °C⁄min.

6.4 The temperature-measuring device in the specimen cup shall be capable of measuring the temperature of the test specimen from −40 °C to +70 °C at a resolution of 0.1 °C.

6.5 The apparatus shall be equipped with fittings to permit the circulation of a liquid cooling medium, if required, to remove heat generated by the Peltier device and other electronic components of the apparatus.

NOTE 4—Some apparatus are designed to use ambient air as a cooling medium. In such cases, a built-in fan is available to provide circulation of air and there is no need for fittings as described for a liquid cooling medium. The function of the cooling medium is to remove heat from the electronic components. The choice of the cooling medium has no impact whatsoever on the test results.

6.6 The apparatus shall be equipped with fittings to permit the circulation of purge gas to purge the test chamber containing the specimen cup of any atmospheric moisture.

7. Reagents and Materials

7.1 *Cooling Medium*—Air, tap water, or other liquid heat exchange medium sufficient to remove heat generated by the Peltier device and other electronic components from the apparatus. To achieve specimen cooling to −40 °C, supply circulation of liquid cooling medium at +25 °C or lower to the apparatus. For an apparatus which relies on air as cooling medium, the ambient air temperature has to be below +30 °C to achieve specimen cooling to −40 °C.

7.2 *Purge Gas*—A gas such as air, nitrogen, helium, or argon with a dew point below the lowest operating temperature of the analyzer. (**Warning—**Compressed gas under high pressure.) (**Warning—**Inert gas can be an asphyxiant when inhaled.)

7.3 *Precision Volume Dispensing Device,* capable of dispensing 0.15 mL ± 0.01 mL of sample.

7.4 *Cotton Swabs*—Plastic or paper shaft cotton swabs used to clean the sample cup. (**Warning—**The use of swabs with wooden shafts may damage the mirrored surface of the specimen cup.)

8. Sampling

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

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

8.3 The sample shall not be heated above 70 °C. When the sample is heated above 70 °C, allow the sample to cool below 70 °C before filtering or inserting into the apparatus.

8.4 When moisture is present in the sample, remove the moisture by a method such as filtration through dry, lint-free filter paper until the oil is perfectly clear, but make such filtration at a temperature at least 14 °C above the expected cloud point.

NOTE 5—Moisture will be noticed in the sample as a separate phase or as a haze throughout the entire sample. Generally, a slight haze will not interfere with the detection of the wax cloud.

9. Preparation of Apparatus

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

9.2 Make liquid cooling medium connections if required (see Note 4) and ensure that they do not leak.

9.3 Make purge gas connections and ensure that they do not leak.

9.4 Turn on the liquid cooling medium if required (see Note 4).

9.5 Turn on the purge gas.

9.6 Turn on the main power switch of the analyzer. After the automatic self diagnostics startup sequence is completed, the instrument will display a **READY** message.

10. Calibration and Standardization

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 mutually agreed upon cloud point can be used to verify performance of the apparatus.

11. Procedure

11.1 Inspect the specimen cup to ensure it is clean and dry. If not, clean the cup (see 11.3).

11.2 Deliver 0.15 mL ± 0.01 mL of specimen into the specimen cup. Pipette, syringe, or precision positive-displacement devices are suitable for use in delivering the specimen.

11.3 Clean the specimen out of the cup. The cup must be cleaned to the point where no visible droplets of specimen remain in the cup. Non-abrasive absorbent materials such as cotton swabs are suitable for use in cleaning the specimen cup. Cleaning solvents able to clean the specimen and compatible with the components of the apparatus can also be used. Naphtha, hexane, heptane, and toluene are suitable as cleaning solvents.

11.4 Repeat steps 11.2 and 11.3.

11.5 Carefully measure 0.15 mL ± 0.01 mL of specimen into the specimen cup.

11.6 Close and lock the test chamber lid.

11.7 Select the **PRE-HEAT** menu on the apparatus if the expected cloud point is less than 14 °C below the specimen

ambient temperature. The specimen ambient temperature is displayed on the front panel of the apparatus. With this selection, the apparatus will automatically heat the specimen to a starting temperature of 50 °C prior to cooling. If the **PRE-HEAT** menu is not selected, the apparatus will cool the specimen from ambient temperature without any initial heating. When the cloud point is expected to be higher than 35 °C, select a higher starting temperature according to manufacturer's instructions. The highest starting temperature that can be programmed is 70 °C.

11.8 Start the operation of the apparatus in accordance with the manufacturer's instructions.The apparatus will allow the flow of liquid cooling medium, if required, (see Note 4) and the flow of purge gas through the apparatus. (**Warning—**The apparatus will display appropriate warning signals if any of these flows are not properly established. Refer to manufacturer's operating manual for corrective procedures.)

11.9 The specimen is heated if specified as described in 11.7. It is then cooled by the Peltier device while the optical detectors continuously monitor the specimen for the appearance of a cloud of wax crystals. The measurement is automatically terminated once the cloud point is detected.

11.10 When the measurement is complete, the automatic cloud point value per Test Method D5773 will be displayed on the front panel of the apparatus.

11.11 Open the test chamber lid to access the specimen cup and clean the specimen out of the cup (see 11.3).

12. Report

12.1 Report the temperature recorded in 11.10 as the automatic cloud point Test Method D5773.

12.2 When specified, round the temperature recorded in 11.10 to the next lower integer and report as the Test Method D2500 equivalent cloud point per Test Method D5773.

13. Precision and Bias

13.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory test results[5,6] is as follows:

13.1.1 *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 1.3 °C only in one case in twenty.

13.1.2 *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 2.5 °C only in one case in twenty.

13.1.3 The precision statements were derived from a 1997 interlaboratory cooperative test program.[5] Participants analyzed eleven sample sets as blind duplicates, comprised of various distillate fuels and lubricating oils, and with a cloud point range of +34 °C to −56 °C. Ten laboratories participated with the automatic apparatus and eight laboratories participated with the manual Test Method D2500/IP 219 test method. The precision statistics were compiled and calculated based on the 0.1 °C resolution offered by this automatic apparatus. Information on the type of samples and their average cloud points are in the research report.

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*—The results of the interlaboratory program were examined for bias relative to Test Method D2500/IP 219. A statistically insignificant bias of −0.03 °C was observed.

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

13.4.1 *Repeatability for Blends of Biodiesel in Diesel*—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 $0.017 \times (27 - X)$ °C only in 1 case in 20, where X is the average of results being compared.

13.4.2 *Reproducibility for Blends of Biodiesel in Diesel*—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 the normal and correct operation of this test method, exceed $0.063 \times (27 - X)$ °C only in 1 case in 20, where X is the average of results being compared.

NOTE 6—The precision for blends of biodiesel in diesel samples comprised cloud points from about –27.2 °C to 13.8 °C. Table 1 illustrates repeatability and reproducibility with a range of cloud point. The degrees of freedom associated with the reproducibility estimate from this round robin study is 23. Since the minimum requirement of 30 (in accordance with Practice D6300) is not met, users are cautioned that the actual repeatability/reproducibility may be significantly different than these estimates.

[5] Supporting data (the results of the 1990 interlaboratory cooperative test program) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1373. Contact ASTM Customer Service at service@astm.org.

[6] Supporting data (the results of the 1997 interlaboratory cooperative test program) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1510. Contact ASTM Customer Service at service@astm.org.

TABLE 1 Precision for Biodiesel and Biodiesel Blends (–28.0 °C to 12.0 °C)

Cloud Point (°C)	–28.0	–24.0	–20.0	–16.0	–12.0	–8.0	–4.0	0.0	4.0	8.0	12.0
Repeatability	0.9	0.9	0.8	0.7	0.7	0.6	0.5	0.5	0.4	0.3	0.3
Reproducibility	3.5	3.2	3.0	2.7	2.5	2.2	2.0	1.7	1.4	1.2	0.9

13.4.3 The precision statements were derived from a 2006 interlaboratory cooperative test program.[7] Seven participants analyzed sample sets comprised of different biodiesel blends from various feedstocks, and included seasonal diesel samples and biodiesel blends B5, B10, and B20, as well as B100 including soy methyl ester (SME), yellow grease methyl ester (YGME), and tallow methyl ester (TME) with temperature range from –27.2 °C to 13.8 °C. Seven laboratories participated with the D5773/IP 446 test method. Information on the type of samples and their average cloud points are in the research report.

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

13.6 *Relative Bias*—The statistical analysis for the interlaboratory test program for bias relative to Test Method D2500/IP 219 has been determined for blends of biodiesel in diesel samples.

[7] Supporting data (the results of the 2006 interlaboratory cooperative test program) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1627. Supporting data (the results of the precision determinations for D5773 from the interlaboratory cooperative test program) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1823. Contact ASTM Customer Service at service@astm.org.

14. Keywords

14.1 automatic cloud point; cloud point; constant cooling rate; Peltier; petroleum products; thermoelectric; wax crystals

ANNEX

(Mandatory Information)

A1. DETAILED DESCRIPTION OF APPARATUS

A1.1 *Test Chamber*, comprised of optical detectors, lens, light source, specimen cup, temperature sensor, Peltier device, and heat sink arranged in a configuration as shown in Fig. A1.1. The lid of the test chamber can be opened to allow cleaning of specimen cup and introduction of new specimen. Once closed and locked, the chamber becomes airtight. An O-ring is used to seal the mating surfaces between the lid and the rest of the chamber. The air trapped in the closed chamber is purged by dry gas. The dry gas inlet and outlet are shown in Fig. A1.1. The test chamber wall is made of black colored metal and plastic components to minimize light reflection.

A1.1.1 *Specimen Cup,* comprised of black plastic wall and a highly polished metal bottom. The polished surface of the bottom serves as a reflective surface for light. The transfer of heat to and away from the specimen through the metal bottom is controlled by the Peltier device.

A1.1.2 *Temperature Sensor,* reading to 0.1 °C, permanently embedded into the bottom of the specimen cup and positioned less than 0.1 mm below the top surface of the cup bottom. This temperature sensor, which is made of a single strand platinum, provides accurate measurement of the specimen temperature.

A1.1.3 *Peltier Device,* capable of controlling the specimen temperature over a wide range. The range varies depending on the model series. During specimen cooling, heat is transferred from the top of the device to the bottom. Since the top is in thermal contact with the bottom of the specimen cup, the specimen will be chilled. The bottom of the Peltier device is in thermal contact with the heat sink where heat is dissipated to the cooling medium. During specimen warming, the reverse process will take place.

A1.1.4 *Light Source,* to provide a beam of light with a wavelength of 660 nm ± 10 nm. The light source is positioned such that it provides an incident beam (Fig. A1.1) impinging onto the specimen at an acute angle. The light is reflected from the polished bottom of the specimen cup. When the specimen is a homogeneous liquid, the reflected beam impinges onto the chamber lid, which is black in color. The reflected light is then absorbed by the black surface. When wax crystals appear in the specimen, the reflected beam is scattered by the solid-liquid phase boundaries. A significant amount of scattered light impinges onto the lens (Fig. A1.2).

A1.1.5 *Optical Detectors,* positioned above the lens to monitor the clarity of the specimen. The distance between the optical detectors and the lens is adjusted such that the image of the specimen is projected onto the light-sensitive surface of the optical detectors. Sufficient optical detectors are used to cover the image area.

A1.2 *Apparatus Exterior Interface*, composed of several displays and buttons as shown in Fig. A1.3 (the exact layout of the displays and buttons may vary depending on the model series).

A1.2.1 *Message Display,* provides information on the status of the apparatus. It displays a **READY** message when the apparatus is idle and no fault is found. At the end of a test, the result is displayed. It displays a diagnostic message if a fault is detected in any of the major components of the apparatus. Detailed explanation of the diagnostic messages is available in the manufacturer's service manual.

A1.2.2 *Specimen Temperature Display,* gives an update of the specimen temperature, recorded to 0.1 °C, every 2 s.

A1.2.3 *Light Signal Display,* gives an update of the scattered light level received by the optical detectors every 2 s. This information is used by service personnel for troubleshooting purposes.

A1.2.4 *Menu Buttons,* allow the operator to specify the pre-heat requirement for the specimen.

A1.2.5 *Run Button,* allows the operator to start the measurement sequence once the specimen is put inside the test chamber.

A1.2.6 *Reset Button,* allows the operator to stop the measurement sequence. Upon pressing this button, the apparatus will immediately stop the measurement sequence and warm the specimen to about 20 °C.

NOTE A1.1—A full description, installation, setup instructions, and maintenance instructions are contained within the manufacturer's manual supplied with each instrument.

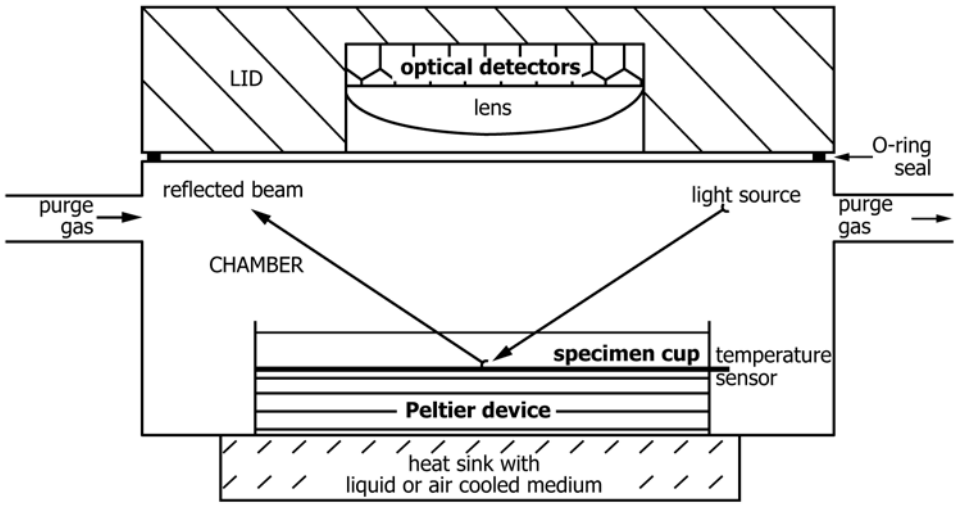

FIG. A1.1 Schematic of Test Chamber

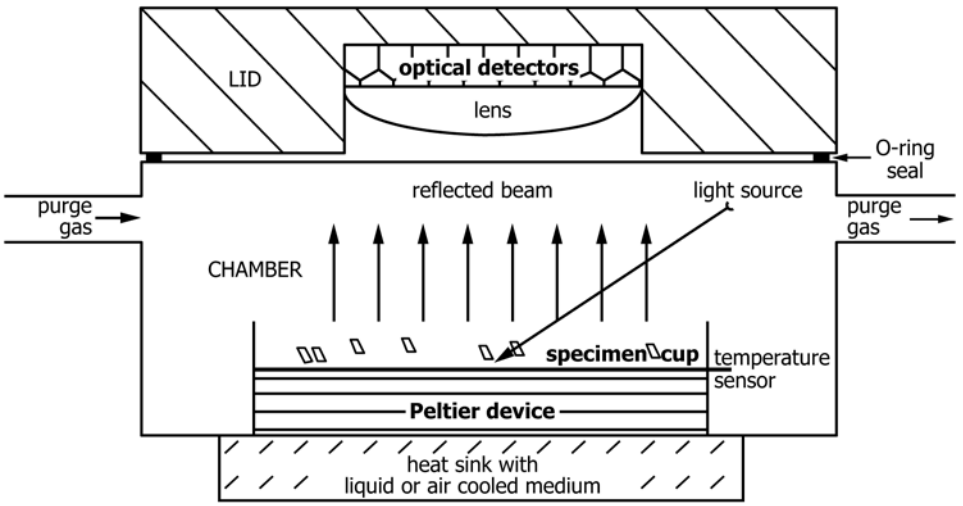

FIG. A1.2 Detection of Crystal Formation

FIG. A1.3 Apparatus Exterior

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D5773 – 15a) that may impact the use of this standard. (Approved May 1, 2017.)

(1) Revised title.
(2) Revised 3.1.3.
(3) Revised 3.1.3.1.