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An American National Standard

Designation: 444/99

Standard Test Method for Cloud Point of Petroleum Products (Optical Detection Stepped Cooling Method)¹

This standard is issued under the fixed designation D 5771; 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 D 2500 using an automatic apparatus. The temperature results from this test method have been found to be equivalent to Test Method D 2500. When specification requires Test Method D 2500, 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 describes 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 an optical device.

1.2 This test method covers the range of temperatures from -60 to $+49^{\circ}\text{C}$ 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 to $+34^{\circ}\text{C}$.

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

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 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~~; April 1, 2004. Published ~~August 2003~~; April 2004. Originally approved in 1995. Last previous edition approved in 2002³ as D 5771-02³.

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

2. Referenced Documents

2.1 ASTM Standards:²

- D 2500 Test Method for Cloud Point of Petroleum Products
- D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products
- D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

2.2 *HP*Energy Institute Standard:³

- IP 219 Test Method for Cloud Point of Petroleum Products³

2.3 Other Standard:⁴

- IEC 751 Industrial Platinum Resistance Thermometer Sensors

3. Terminology

3.1 Definitions:

3.1.1 *biodiesel, n*—fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats, designated B 100.

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 % of biodiesel.

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~~ hydrocarbon crystals first ~~appears~~ occurs upon cooling under prescribed conditions.

3.1.3.1 *Discussion*—The cloud point ~~appears~~ occurs when the temperature of the specimen is low enough to cause ~~wax~~ hydrocarbon crystals to precipitate. In a homogeneous liquids, the cloud is always noted first at the location in the specimen where the specimen temperature is the lowest. The cloud point is typically the temperature at which the lower portion crystals first occur, regardless of the test jar when using the apparatus described their location in Test Method D 2500. the specimen, and not after extensive crystallization has taken place. The hydrocarbon crystals that precipitate at lower temperatures are typically, but not excluded to, straight chain hydrocarbons commonly called “wax crystals.”

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 D 2500, although, according to interlaboratory examination the results have been determined to be equivalent to Test Method D 2500.

3.2.2 *D 2500/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.

3.2.2.1 *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 D 2500, then this calculation can be performed. Some apparatus can perform this calculation automatically.

4. Summary of Test Method

4.1 After insertion of the specimen into the apparatus, and the initiation of the program, the specimen is cooled according to the cooling profile listed in Table 1. The specimen is continuously monitored by an optical system for the formation of a crystalline structure. When the crystallization of the wax in the specimen is detected by the optical system, the temperature is recorded to within 0.1°C resolution. The specimen is then heated to facilitate the start of the next test.

5. Significance and Use

5.1 For petroleum products and biodiesel fuels, the cloud point 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 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.

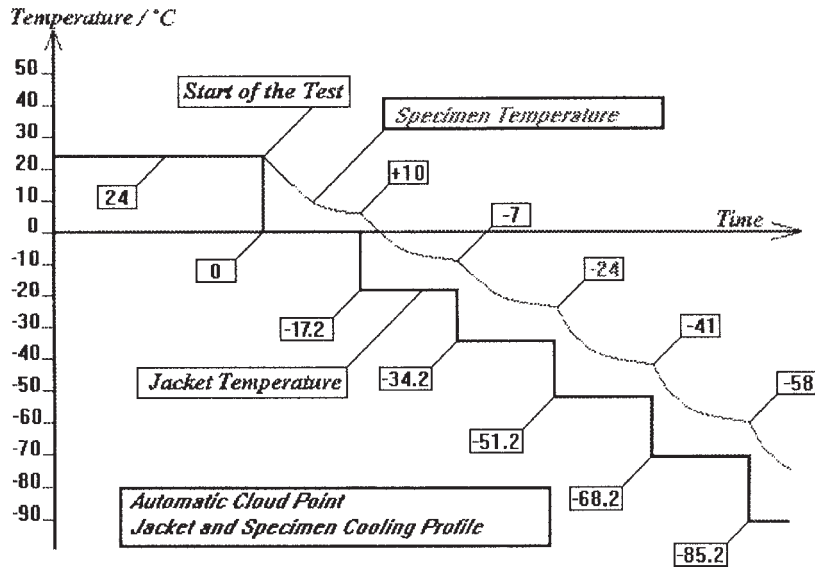
² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards*, Vol 05.04, volume information, refer to the standard's Document Summary page on the ASTM website. *Annual Book of ASTM Standards*, Vol 05.06.

³ Available from the Energy Institute, 61 New Cavendish St., London, England W1G 7AR, U.K.

⁴ Available from the Institute of Petroleum, 61 New Cavendish St., London, England W1M 8AR; International Electrotechnical Commission, P.O. 131, 3 Rue de Varembe, CH-Geneve 1211-2.0.

TABLE 1 Jacket and Specimen Cooling Temperatures

Specimen Temperature, °C	Jacket Temperature, °C
ST > + 10	0 ± 0.5
+ 10 ≥ ST > - 7	-17.2 ± 0.5
- 7 ≥ ST > - 24	-34.2 ± 0.5
- 24 ≥ ST > - 41	-51.2 ± 0.5
- 41 ≥ ST > - 58	-68.2 ± 0.5
- 58 ≥ ST > - 75	-85.2 ± 0.5



5.4 This test method provides results that, when rounded to the next lower integer, are equivalent to Test Method D 2500. Refer to 12.2.

5.5 This test method is more precise than Test Method D 2500.

NOTE 1—According to interlaboratory examination, the reproducibility of this test method has been found to be more precise than Test Method D 2500.

6. Apparatus

6.1 *Optical Cloud Point Apparatus*⁵—The automated cloud point apparatus (see Fig. 1) described in this test method consists of a microprocessor controller that is capable of controlling one or more independent test cells. The apparatus shall include provisions for independently controlling the temperature of each cell according to the specified cooling profile, continuously monitoring the specimen temperature, detecting the appearance of the cloud point at the bottom of the test jar without removing it from the jacket, and displaying the 0.1 or 1.0°C result (see Fig. 2 and Fig. 3).

6.2 *Temperature Probe, IEC 751 Class A: ΔT = ± (0.15 + 0.002 |T|)*, capable of measurement from -50 to +80°C. The temperature probe shall be in contact with the bottom of the test jar.

6.3 *Test Jar*, clear cylindrical glass, mirrored flat bottom, 34 ± 0.1-mm outside diameter, 31 ± 0.3-mm inside diameter, 120 ± 0.5-mm height, thickness of the bottom 2.0 ± 0.5 mm, marked with a line to indicate the sample height 54 ± 0.5 mm above the inside bottom.

6.4 *Jacket*, brass, cylindrical, flat bottom, 113 ± 0.2 mm in depth, 45 ± 0.1-mm inside diameter. It must be cooled according to the cooling profile specified.

6.5 *Cooling Circulating Bath*, equipped with a circulating pump and capable of maintaining a temperature at least 10°C below the last required jacket temperature level (see Table 1).

6.6 *Cork Disk*, 6 ± 0.2 mm thick, to fit loosely inside the jacket. Felt may be used but special attention must be given to the potential for moisture in the felt disk. The felt must be dried before each test.

6.7 *Cork Ring*, to fit snugly around the outside of the test jar and loosely inside the sample cell. Its purpose is to prevent the test jar from touching the jacket.

7. Reagents and Materials

7.1 *Cleaning Solvents*, suitable for cleaning and drying the test cell, such as petroleum naphtha and hexane.

⁵ The sole source of supply of the ISL Model CPP97-6 and CPP97-2 known to the committee at this time is ISL North America, Stevensville, MD or ISL SA, BP 40, 14790 Verson, France. If you are aware of alternative suppliers, please provide this information to ASTM International-Electrotechnical Commission, P.O. 131, 3 Rue de Varembe, CH Geneva 1211-20, Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee¹, which you may attend.

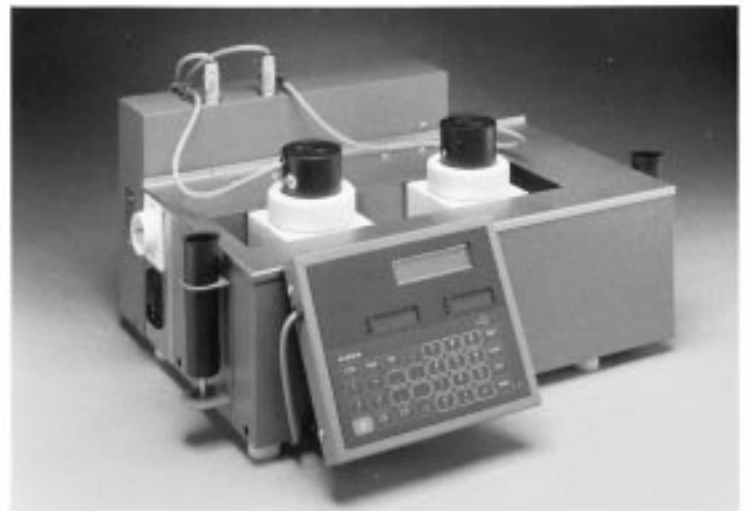
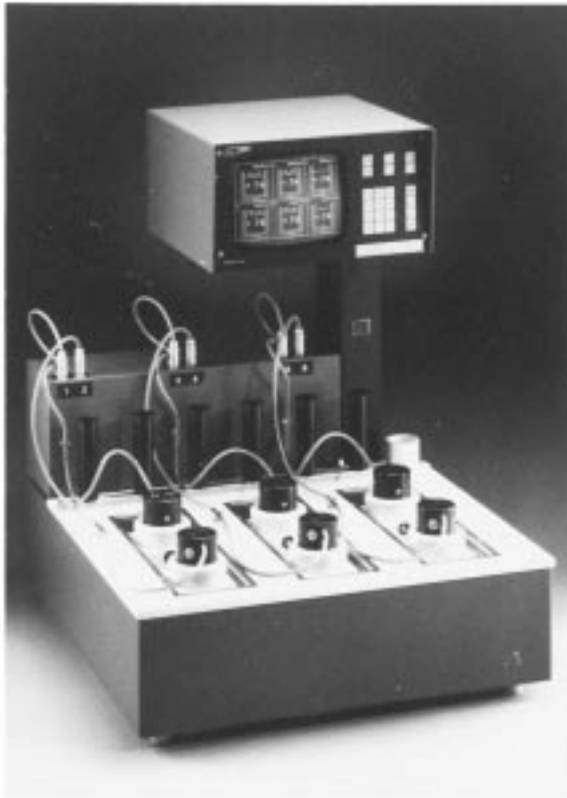


FIG. 1 Automated Cloud Point Apparatus

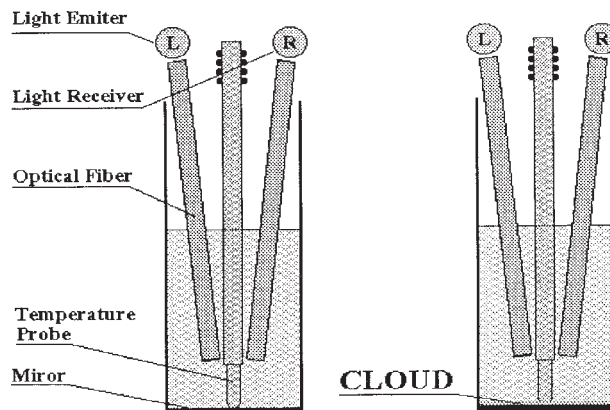


FIG. 2 Test Jar and Detection System

7.2 *Methyl Alcohol, Anhydrous*, for use as cooling medium in circulating bath.

7.3 *Sodium Sulfate*—A reagent grade of anhydrous sodium sulfate should be used when required. (**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 Practices D 4057 or by D 4177.

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

8.3 The sample shall not be heated above 70°C. If 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 2—A wax cloud or haze is always noted at the bottom of the test jar where the temperature is lowest. A slight haze throughout the entire specimen,

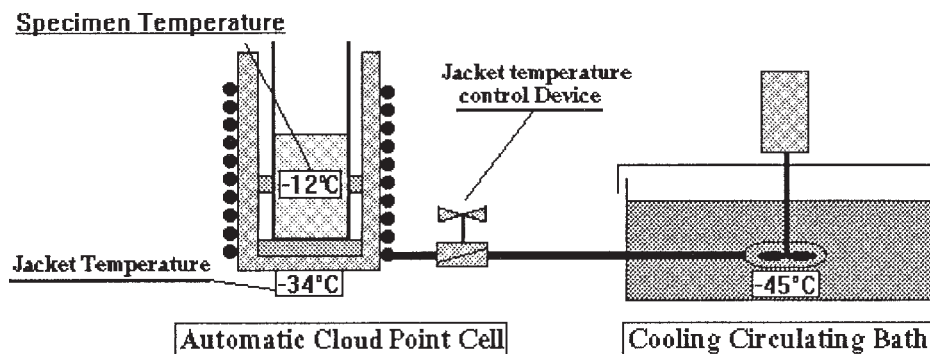


FIG. 3 Test Jar Cooling Chamber and Circulating Bath

which slowly becomes more apparent as the temperature is lowered, is usually due to water in the specimen. Generally this water haze will not interfere with the determination of the wax cloud point. In most cases of interference, filtration through dry lint-free filter papers such as described in 11.2 is sufficient.

8.5 For diesel fuels, if the haze is very dense, a fresh portion of sample must be dried by shaking 100 mL with 5 g of anhydrous sodium sulfate for at least 5 min and then filtering through dry lint-free filter paper. Given sufficient contact time, this procedure will remove or sufficiently reduce the water haze so that the wax cloud can be readily discerned. Drying and filtering should always be done at a temperature at least 14°C above the approximate cloud point, but otherwise not in excess of 49°C.

9. Preparation of Apparatus

- 9.1 Prepare the instrument for operation in accordance with the manufacturer's instructions.
- 9.2 Clean and dry the test head and test jar using suitable solvents as prescribed by the manufacturer.
- 9.3 Adjust the set point of the recirculating cooler to the appropriate temperature to cool the jacket to the required temperature (see Table 1).

10. Calibration and Standardization

- 10.1 Ensure that all of the manufacturer's instructions for calibrating, checking, and operating the apparatus are followed.
 - 10.1.1 A test head simulator with a known resistance is used to calibrate the equipment. Follow the manufacturer's calibration instructions.

NOTE 3—A test head simulator may be obtained from the apparatus supplier which can assist in verification of temperature measurement.

- 10.2 A sample with a mutually agreed upon cloud point such as a sample from a interlaboratory test program can be used to verify performance of the apparatus.

11. Procedure

- 11.1 Set the temperature unit on the apparatus to 1°C or to 0.1°C.

NOTE 4—The temperature unit, in common laboratory practice, is 1°C. If a higher resolution is desired, 0.1°C can be selected.

- 11.2 Using a water bath or oven, bring the sample to be tested to a temperature at least 14°C above the expected cloud point. When moisture is present in the sample, remove the moisture present by a method, such as filtration through dry lint-free filter paper, until the sample is perfectly clear, but make such filtration at a temperature of at least 14°C above the approximate cloud point but otherwise not in excess of 70°C.

- 11.3 Pour the clear sample into the test jar to the level mark.

- 11.4 Place a cork disk at the bottom of the jacket in the appropriate cell and fit a cork ring around the test jar. Use the cork placement guide, if necessary, for the final adjustment. The cork ring must be 25 ± 3.0 mm above the bottom of the test jar.

NOTE 5—A cork placement guide may be obtained from the apparatus supplier which can assist in proper placement of the cork ring.

- 11.5 Place the test jar in the appropriate test cell. Attach the detector head according to the manufacturer's instructions.

- 11.6 Start the test according to the manufacturer's instructions. The instrument shall then automatically adjust the jacket temperature in accordance with Table 1 and begin to optically monitor the specimen for the cloud point. The instrument shall monitor and display the jacket temperature and the specimen temperature during the test procedure.

- 11.7 The instrument shall continue to monitor and adjust the jacket temperature according to Table 1. At no point shall the time to move from one jacket temperature level to the next jacket temperature level exceed 90 s.

- 11.8 The instrument shall detect the cloud point at the bottom of the test jar and record and display the temperature as selected in 11.1. The instrument shall have an audible alarm, such that when a cloud point is detected, it immediately notifies the operator. This function allows the operator to remove the specimen from the instrument and visually inspect the cloud formation and verify the result, if desired.

11.9 After the determination, the instrument shall adjust the jacket temperature to $23 \pm 2^{\circ}\text{C}$.

12. Report

12.1 If 0.1°C was selected in 11.1, report the temperature recorded in 11.9 as automated cloud point Test Method D 5771.

12.2 If 1.0°C was selected in 11.1, report the temperature recorded in 11.9 as Test Method D 2500 equivalent cloud point Test Method D 5771.

13. Precision and Bias

13.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory test results^{6,7} 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 2.2°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 3.9°C only in one case in twenty.

13.1.3 The precision statements were derived from a 1997 interlaboratory cooperative test program.⁷ Participants analyzed eleven sample sets as blind duplicates, comprised of various distillate fuels and lubricating oils with a temperature range from $+34$ to -56°C . Ten laboratories participated with the automatic machines and eight laboratories participated with the manual Test Method D 2500/IP 219. 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 D 2500/IP 219. Although statistically significant bias was observed, the magnitude was determined small enough (-0.56°C) to be of little practical significance.

13.4 *Precision for Biodiesel Products* —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 1.2°C only in one case in twenty.

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 2.7°C only in one case in twenty.

NOTE 6—The precision for blends of biodiesel in diesel samples comprised cloud points from about -2 to $+10^{\circ}\text{C}$.

13.4.3 The precision statements were derived from a 2001 interlaboratory cooperative test program.⁸ Participants analyzed eleven sample sets comprised of two petroleum distillate fuels, diesel and kerosine, with various biodiesel fuels with a temperature range from $+10$ to -45°C . Nine laboratories participated with the automatic machines, and ten laboratories participated with the manual Test Method D 2500/IP 219. Information on the type of samples and their average cloud points are in the research report.

NOTE 7—One of the outcomes of the interlaboratory study was the selection of the sample types used in the study contributed to a difficulty in determining the precision statement. Kerosine is a sufficiently different fuel type from biodiesel to cause some slight separation of phases upon cooling when in B20 blends. Also, the particular kerosine sample used was atypical, which complicated the study further. Therefore data from the blends of kerosine in biodiesel were not used in the precision statement. In addition, the diesel fuel used in the research report was high cloud point material. Due to the cloud point of the base diesel material, the temperature range of the precision statement was limited.

NOTE 8—A future interlaboratory cloud study will be done including a wider range of biodiesel fuels with various distillate blend stocks.

13.5 *Bias for Biodiesel Products*—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 D 2500/IP 219 has not been determined for blends of biodiesel in diesel samples.

14. Keywords

14.1 automatic cloud point; cloud point; petroleum products

⁶ The sole source of supply of the ISL Model CPP97-6 and CPP97-2 known to the committee

⁶ Supporting data have been filed at this time in ISL North America, Stevensville, MD or ISL SA, BP 40, 14790 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, which you may attend, and may be obtained by requesting Research Report RR: D02-1373.

⁷ 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: D02-1373; D02-1508.

⁸ Supporting data (the results of the 1997 2001 interlaboratory cooperative test program) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1508/24.

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D 5771–023) that may impact the use of this standard.

- ~~(1) Revised Scope to incorporate the 1997 round robin sample temperature range.~~
- ~~(2) Incorporated the 1997 round robin repeatability.~~
- ~~(3) Incorporated the 1997 round robin reproducibility.~~
- ~~(4) Revised 13.1.3 to reflect wording pertaining to the 1997 interlaboratory test program.~~
- ~~(5) Deleted Notes 6 definition and 7, because they were redundant with the completion discussion of the 1997 cloud-round robin point.~~

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