Designation: D1218 – 12 (Reapproved 2016)

Standard Test Method for Refractive Index and Refractive Dispersion of Hydrocarbon Liquids[1]

This standard is issued under the fixed designation D1218; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers the measurement of refractive index, accurate to four decimal places or better, of transparent and light-colored hydrocarbons in the range of 1.3300 to 1.5000 at temperatures from 20 °C to 30 °C by manual (optical-mechanical) or automatic (digital) procedure.

1.2 The manual (optical-mechanical) procedure also covers the measurement of refractive dispersion accurate to the fourth decimal place or better.

NOTE 1—The test method may be suitable for measuring the refractive indices of liquids above 1.5000 and at temperatures both below 20 °C and above 30 °C; however, the test method precision may not apply. Verification of the accuracy of such measurements will depend upon the availability of reliable, certified reference standards that demonstrate the performance of the instrument used under the particular measuring conditions.

1.3 The test method may not be applicable to liquids that are strongly colored, or that have bubble points so near the test temperature that a reading cannot be obtained before substantial weathering takes place. Liquid color should be limited to No. 4 ASTM Color or lighter, as determined by Test Method D1500.

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 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website—http://www.epa.gov/mercury/faq.htm—for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law.

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:*[2]

D1193 Specification for Reagent Water
D1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)
D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance
D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants
E1 Specification for ASTM Liquid-in-Glass Thermometers

2.2 *ASTM Adjuncts:*

Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants, Version 4.0.65[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *refractive dispersion, n*—the difference between the refractive indices of a substance for light of two different wavelengths, both indices being measured at the same temperature.

3.1.1.1 *Discussion*—For convenience in calculations, the value of the difference thus obtained is usually multiplied by 10 000.

---

[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 D02.04.0D on Physical and Chemical Methods.

Current edition approved Oct. 1, 2016. Published November 2016. Originally approved in 1952. Last previous edition approved in 2012 as D1218 – 12. DOI: 10.1520/D1218-12R16.

[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] Out of print. No longer available from ASTM International Headquarters.

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

3.1.2 *refractive index, n*—the ratio of the velocity of light (of specified wavelength) in air, to its velocity in the substance under examination.

3.1.2.1 *Discussion*—It may also be defined as the sine of the angle of incidence divided by the sine of the angle of refraction, as light passes from air into the substance. This is the relative index of refraction. If absolute refractive index (that is, referred to vacuum) is desired, this value should be multiplied by the factor 1.00027, the absolute refractive index of air. The numerical value of refractive index of liquids varies inversely with both wavelength and temperature.

4. Summary of Test Method

4.1 The refractive index is measured using a high-resolution refractometer of an optical-mechanical or automatic digital type with the prism temperature accurately controlled. The instrument principle is based on the critical angle concept.

5. Significance and Use

5.1 Refractive index and refractive dispersion are fundamental physical properties, which can be used in conjunction with other properties to characterize pure hydrocarbons and their mixtures.

PROCEDURE A—MANUAL (OPTICAL-MECHANICAL) PROCEDURE

6. Apparatus

6.1 *Refractometer,* high-resolution optical-mechanical refractometer of the "Abbe" type with suitable measuring range (1.3300 to 1.5000 or higher) and an accuracy/resolution of 0.0001 or better refractive index.

NOTE 2—Prior to 2001, Test Method D1218 was based on a Bausch & Lomb Refractometer. However, this instrument is no longer manufactured. Currently available manual refractometers are of the "Abbe" type (critical angle refractometers, see X1.2.3). There is a variety of refractometers that have been found to be suitable for this measurement. Some instruments offer a wider measuring range but it is important to verify the uniformity of accuracy across the entire measuring range and to ensure suitable calibration materials are available for this verification.

6.2 *Temperature Control Unit,* either an external liquid bath with both heating and cooling capability and pump for maintaining the indicated prism temperature within 0.1 °C, or an internal electronic temperature control system (such as Peltier system). If an external bath is used, the thermostating liquid shall pass the temperature measuring device on leaving, not on entering the prism. The temperature control unit (external or internal) shall have the following control specification:

Stability	±0.1 °C
Uniformity	±0.1 °C
Display Resolution	0.1 °C or better

6.3 *Temperature Measuring Device,* for those apparatus employing mercury in glass thermometer, ASTM Thermometer 17C having a range from 19 °C to 27 °C, and conforming to the requirements of Specification E1. For apparatus using non-mercury in glass thermometer, a platinum resistance probe, thermocouple, or equivalent temperature sensors are acceptable.

6.4 *Light Sources*—The following type of light source can be used in conjunction with an optical-mechanical (Abbe type) refractometer:

6.4.1 *Sodium Arc Lamp,* Na_D line at 589 nm.

6.4.2 *Mercury Arc Lamp,* Hg_c line at 546 nm or Hg_g line at 436 nm.

6.4.3 *Cadmium Arc Lamp,* $Cd_{C'}$ line at 644 nm.

6.4.4 *Mercury-Cadmium Arc Lamp.*

6.4.5 *Helium Discharge Lamp.*

NOTE 3—Measurement of refractive dispersion requires more than one type of light source.

6.5 *Light Filters*—Arc lamps can emit a number of spectral lines that result in multiple-borderlines observed in the refractometer. Filters can be used to eliminate unwanted lines (borderlines). Depending upon instrument design, the manufacturer will recommend and supply a suitable filter or adapt a commercially-available filter (for example, interference filter) to suit the application.

7. Reagents and Materials

7.1 *n-Pentane, 95 mol % Minimum Purity,* for cleaning the prism faces. (**Warning—**Flammable. Harmful if inhaled. Vapors may cause flash fire.)

NOTE 4—Low boiling hydrocarbon fractions with boiling range 50 °C to 100 °C have also been found to be acceptable.

7.2 *Toluene, HPLC Grade,* for cleaning the prism faces. (**Warning—**Flammable. Vapor harmful.)

7.3 *1-Bromonaphthalene, 98 mol % Minimum Purity,* contact liquid when calibrating with solid reference standard. (**Warning—**Toxic when ingested.)

7.4 *Primary Reference Materials,* for calibrating the instrument.

7.4.1 *Solid Reference Standard,* with the value of refractive index engraved on its upper face.

7.4.2 *Distilled or Deionized Water,* conforming to Type II or III of Specification D1193. At 20 °C, $n_D = 1.3330$; at 25 °C, $n_D = 1.3325$; and at 30 °C, $n_D = 1.3319$

7.4.3 *2,2,4-trimethylpentane, 99 mol % Minimum Purity,* at 20 °C, $n_D = 1.3915$; at 25 °C, $n_D = 1.3890$.

7.4.4 *Methylcyclohexane, 99 mol % Minimum Purity,* at 20 °C, $n_D = 1.4231$; at 25 °C, $n_D = 1.4206$.

7.4.5 *Toluene, 99 mol % Minimum Purity,* at 20 °C, $n_D = 1.4969$; at 25 °C, $n_D = 1.4941$. (**Warning—**2,2,4-trimethylpentane, methylcylcohexane, and toluene are all flammable. Their vapor can be harmful.)

NOTE 5—Other pure materials may be used to calibrate the instrument as primary reference material, as long as they can be obtained in 99 mol % minimum purity and accurate values of their refractive indices at specific temperatures are available. The precision of the test method (see 15.1 and 24.1) were obtained using distilled water as the calibrant.

7.5 *Secondary Reference Materials,* for calibrating the instrument.

7.5.1 *Mineral Oil Calibration Standards,* measured and certified by suppliers for specific refractive index ranges and temperatures.

8. Sampling

8.1 A sample of at least 0.5 mL is required. The sample shall be free of suspended solids, water, or other materials that may settle onto the prism surface and affect the measured reading. Water can be removed from hydrocarbons by treatment with calcium chloride followed by filtering or centrifuging to remove the desiccant. The possibility of changing the composition of the sample by action of the drying agent, by selective adsorption on the filter, or by fractional evaporation, shall be considered. (**Warning—**Volatile hydrocarbon samples are flammable.)

9. Preparation of Apparatus

9.1 The refractometer shall be kept scrupulously clean at all times. Dust and oil can impair the optical component of the instrument. Thoroughly clean the prism faces with toluene, followed by *n*-pentane (see Note 4) (**Warning—**These materials are extremely flammable. Harmful if inhaled. Vapors may cause flash fire.) using cotton swabs, fresh clean lens tissue, or similar material, in accordance with manufacturer's instructions. Do not dry the prism faces by rubbing with dry cotton.

9.2 Adjust the thermostat bath/circulator settings or the electronic temperature control system so that the temperature indicated by the refractometer temperature measuring device is within 0.1 °C of the desired value. Turn on the light source and allow the refractometer to equilibrate for 30 min.

NOTE 6—The constancy of the prism temperature can be seriously affected by variations in ambient conditions such as air drafts or changes in room temperature. Reasonable precautions should be taken to minimize these factors.

10. Calibration of Refractometer Using Solid Reference Standard

10.1 Thoroughly clean the prism faces and surfaces of the solid reference standard (see 7.4.1). Open the prism assembly. Apply a drop of 1-bromonaphthalene contact liquid, about 1.5 mm in diameter, to the center of the polished surface of the solid reference material. Press the reference standard against the surface of the prism face with the polished end towards the light source.

10.2 Follow the manufacturer's instructions on how to calibrate the instrument using the solid reference standard.

10.3 If the observed refractive index differs from the value engraved on the solid reference standard by more than 0.0001, adjust the refractometer's scale reading to match the certified value, following manufacturer's instructions.

11. Calibration of the Refractometer Using Liquid Reference Standards

11.1 Using the procedure described in Section 12, determine the refractive index of any of the Reference Materials specified in 7.4 or 7.5 for a specific test temperature. If the observed refractive index for the chosen reference material at a specified test temperature differs by more than 0.0001 of the listed value, make adjustment to the instrument following manufacturer's instructions so that the observed refractive index corresponds to the listed value.

11.2 For optimum accuracy, use a reference material whose refractive index is close to the desired refractive index range and temperature to calibrate the instrument.

12. Procedure

12.1 Ensure that the prism faces are clean and dry. Check that the prism temperature is within 0.1 °C of the desired temperature.

12.2 Unlock (if necessary) and open the prism assembly.

12.3 Place one or two drops of the sample on the lower prism face. Close the prism assembly and lock (if necessary). Turn on the light source. Allow 3 min temperature equilibration time.

12.4 Look through the eyepiece and observe the field consisting of a light and dark portion. Follow manufacturer's instructions to adjust the instrument so that the boundary between the light and dark portions of the field is as sharp as possible.

12.5 Following manufacturer's instructions, make any additional adjustment until the sharp boundary line intersects the midpoint of the crosshairs superimposed on the field.

12.6 Read the refractive index on the scale. Repeat 12.5 at least four times, approaching from either side of the sharp boundary line, and average the scale readings.

12.7 Record and report the average refractive index value.

12.8 If instrument is capable of determining refractive dispersion, change the light source to a light source with different wavelength. Determine the refractive index at the secondary wavelength following 12.4 – 12.7.

NOTE 7—When determining refractive dispersion, it is expected that the instrument would have been calibrated at both wavelengths used.

13. Quality Control

13.1 Confirm the performance of the test procedure by analyzing a quality control (QC) sample, which is stable and representative of the sample of interest.

13.1.1 When quality control/quality assurance (QC/QA) protocols are established in the testing facility, these may be used to confirm the reliability of the test result.

13.1.2 When there are no QC/QA protocols established in the testing facility, Appendix X4 can be used to provide guidelines in performing this function.

14. Calculation and Report

14.1 Report the average refractive index to four decimal places and the test temperature at which the test was made, for example:

$$n_D = x.xxxx \text{ at } 20\ °C \text{ or } n_{589} = x.xxxx \text{ at } 20\ °C \tag{1}$$

where *D* or the 589 signifies that the sodium *D* spectral line was used.

14.2 If a refractive dispersion measurement was made, calculate the absolute value of the difference between the refractive indices at the two wavelengths and multiply the difference by 10 000. Report the calculated value and the test temperature, for example:

$$|(n_g - n_D)| \times 10^4 \text{ at } t = 20\,°C \tag{2}$$

where *g* and *D* signifies that the mercury *g* and sodium *D* spectral lines were used in the measurement.

15. Precision and Bias

15.1 *Precision—Refractive Index:*

15.1.1 The precision for the manual (optical-mechanical) refractive index procedure, as determined by the statistical examination of interlaboratory test results conducted in 1996[4] on eight different samples from ten laboratories, is as follows:

15.1.2 *Repeatability*— The difference between successive test results, obtained by the same operator with the same apparatus, under constant operating conditions, and on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty (95 % confidence level):

$$r_{manual} = 0.0002 \tag{3}$$

15.1.3 *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 the test method, exceed the following values only in one case in twenty (95 % confidence level):

$$R_{manual} = 0.0005 \tag{4}$$

15.1.4 The precision of this test method for automatic and manual refractive index was obtained in accordance with Practice D6300 using the D02 Precision Program (D2PP) software.[3]

15.2 *Precision—Refractive Dispersion:*

15.2.1 *Repeatability* = 0.00012

15.2.2 *Reproducibility* = 0.00012

15.2.3 The precision for refractive dispersion was not obtained in accordance with Practice D6300, and was not part of the 1996 interlaboratory study.

PROCEDURE B—AUTOMATIC (DIGITAL) PROCEDURE

16. Apparatus

16.1 *Refractometer*—Automatic digital refractometer with a suitable measuring range of 1.3300 to 1.5000 or higher, capable of displaying the measured refractive index automatically and digitally. The instrument shall incorporate a solvent-resistant prism seal.

16.2 *Temperature Control Unit*—See 6.2.

16.3 *Temperature Measuring Device*—A platinum resistance probe or equivalent. The accuracy and resolution of the temperature measuring device shall be 0.1 °C or better.

16.4 *Light Source*—Filtered white light such as that obtained using a tungsten-halogen lamp or a light emitting diode (LED), capable of providing the sodium *D* spectral line at 589 nm.

16.5 *Light Filters*—See 6.5.

17. Reagents and Materials

17.1 See Section 7.

18. Sampling

18.1 See Section 8.

19. Preparation of Apparatus

19.1 See Section 9. Ensure that the instrument has a solvent resistant prism seal.

20. Calibration of the Refractometer Using Liquid Reference Standards

20.1 Automatic digital refractometers are normally calibrated using liquid reference standards (see 7.4 and 7.5). They may require a single, two-point, or multi-point calibration. Follow manufacturer's instruction in calibrating the particular automatic digital refractometer being used.

21. Procedure

21.1 Ensure that the prism and the surrounding plate and dish/plate (and presser, if fitted) are thoroughly cleaned as in 9.1. Ensure the prism temperature is correct and the instrument has been properly calibrated.

21.2 Introduce the test sample to the prism ensuring that the prism is adequately covered. Do not introduce an excessive amount because this can slow the thermal equilibration. Lower the sample presser (if fitted).

21.3 Take a succession of readings, noting values, until a steady value is obtained. Record or report, or both, this value as the refractive index of the sample at the test temperature.

NOTE 8—From experience using the instrument, the user will be able to judge with a safe margin the amount of time needed for a sample to thermally equilibrate on the prism. Based on this, it may be possible on some models to set an automatic delay time for readings. This enables a fixed measuring time to be written in to the experimental protocol.

NOTE 9—When successive readings are drifting, the direction of drift will indicate what is happening to the sample. An upward drift means the sample is cooling on the prism; a downward drift means the sample is warming up. This is particularly noticeable when samples are much hotter or cooler than the prism.

22. Quality Control

22.1 See Section 13.

23. Report

23.1 An automatic digital electronic refractometer shall provide a direct and digital reading of refractive index. This can be manually recorded or, if the refractometer is equipped with a computer interface, readings can be sent to a printer or to a computer.

23.2 Report the refractive index to four decimal places and the test temperature, for example:

$$n_D = x.xxxx \text{ at } 20\,°C \text{ or } n_{589} = x.xxxx \text{ at } 20\,°C \tag{5}$$

where *D* or the 589 signifies that the sodium *D* spectral line was used.

---

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1493.

24. Precision and Bias

24.1 *Precision—Refractive Index:*

24.1.1 The precision for the automatic digital refractive index procedure, as determined by the statistical examination of interlaboratory test results conducted in 1996[4] on eight different samples from ten laboratories, is as follows:

24.1.2 *Repeatability*—The difference between successive test results, obtained by the same operator with the same apparatus, under constant operating conditions, and on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty (95 % confidence level):

$$r_{automatic} = 0.0002 \qquad (6)$$

24.1.3 *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 the test method, exceed the following values only in one case in twenty (95 % confidence level):

$$R_{automatic} = 0.0005 \qquad (7)$$

24.1.4 The precision of this test method for automatic refractive index was obtained in accordance with Practice D6300 using the D02 Precision Program (D2PP) software.[3]

24.2 *Relative Bias*—The 1996 interlaboratory study did not indicate any relative bias between automatic and manual refractive index instruments.

25. Keywords

25.1 refractive dispersion; refractive index; refractometer

APPENDIXES

(Nonmandatory Information)

X1. FACTORS THAT AFFECT PRECISION AND ACCURACY

X1.1 *Temperature* —Refractive index varies inversely and non-linearly with temperature. The relative change in refractive index with temperature (temperature coefficient) is different for each liquid under test. Hydrocarbon liquids have a higher temperature coefficient than water and aqueous solutions. For example, at 20 °C the temperature coefficient for water is -9×10^{-5} units of refractive index per °C. For hydrocarbon liquids, the coefficient will typically be in the range -3×10^{-4} to -5×10^{-4}.

X1.2 Wavelength (Light Source)

X1.2.1 Refractive index also varies inversely and non-linearly with the wavelength of light. Refractive index should, therefore, be measured using light of a single wavelength (monochromatic light). Most measurements traditionally have been made at the sodium *D* line (589.3 nm) because of the purity and availability of a sodium source such as an arc lamp. Other light sources include mercury, cadmium, helium, and hydrogen where filters are used with the refractometer to eliminate unwanted emission lines.

X1.2.2 The effect of wavelength on refractive index is called dispersion. Different substances have varying degrees of refractive dispersion (sometimes called dispersive power).

X1.2.3 Optical-mechanical refractometers (often called Abbe refractometers or critical angle refractometers) can be used with a variety of spectral light sources. Some instruments can utilize a white light source, but often these instruments are limited in accuracy because of the light source. High accuracy Abbe refractometers require a pure spectral light source such as a sodium arc lamp (see 6.4).

X1.2.4 Modern electronic digital refractometers function at a single wavelength, invariably that of sodium light (589 nm). The refractive index at this wavelength is denoted as n_D.

X1.2.5 One of two types of light source tends to be used in digital refractometers: (*1*) filtered white light source such as that obtained using a tungsten-halogen lamp, or (*2*) light emitting diode (LED).

X1.2.6 With both types of light source, the wavelength is not a single value but is a narrow band spanning typically 10 nm or more about the nominal value (589 nm). The degree of impurity (band width) will cause a dispersion of the detected light from the sample. Instrument software can be used to compensate for and thus minimize this error source but it will nevertheless be a factor in determining the limiting accuracy of the instrument. The dispersion error will however differ according to the substance measured. This is why instrument manufacturers may specify the instrument accuracy as *typically* or *better than* in order to cover a broad application scope.

NOTE X1.1—Sodium actually emits a doublet of lines at 589.1 nm and 589.6 nm. However, for practical purposes, because of the proximity of the lines, it is regarded as a single wavelength source and the average value of 589.3 nm is often cited.

X2. INSTRUMENT CALIBRATION

X2.1 A refractometer must be calibrated using one or more reliable calibration standards. The optical system in a refractometer is subject to microscopic movement as materials expand and contract with fluctuating ambient thermal conditions. This effect leads to calibration drift, which must be periodically corrected to ensure measurement reliability.

X2.2 Calibration materials are available as two types: solids (test plates) and liquids. Solid plates are often preferred for their stability (shelf life), but have the disadvantage of limited accuracy in use because of the errors associated with the placing of the plate on the refractometer prism. Typically, a plate may not allow an accuracy better than ±0.0001 units of refractive index. Test plates are particularly difficult to apply to sapphire prisms of the type typically used in digital electronic refractometers. This is because the micro-roughness of sapphire is greater than that of softer (optically flatter) glasses used in optical (Abbe) refractometers. Invariably, liquid reference standards will be preferred and will be recommended by instrument manufacturers for this reason.

X2.3 An optical-mechanical instrument may require calibration at only one point on the scale. Automatic digital refractometers may require a single (zero), two-point, or multi-point calibration. For each calibration a suitable calibration standard should be used.

X2.4 The frequency of calibration will depend upon a number of factors. When an electronic instrument is first switched on and the liquid bath circulation is started, a period of thermal adjustment is required to allow the internal environment, and particularly the optical system, to adjust to the new condition. Calibration should not take place until the instrument is in a steady-state. Experience will demonstrate that this may be typically up to an hour. Similarly, if the circulating liquid temperature is changed, a new calibration sequence will be required. In circumstances where the instrument is used continuously and in a constant thermal condition, the need for recalibration should be minimal.

X2.5 For each calibration point, a calibration standard is required. Any calibration fluid could be used. However, it is best practice to select fluids with a refractive index close to the intended measuring range or, where a pair or group of standards is needed, select liquids that adequately span the working range.

X2.6 Whether an electronic digital instrument or an optical-mechanical instrument is being used, the laboratory needs to adopt a strict protocol for calibration frequency and procedure. This will entail regular checks on measurement accuracy (verification) using reliable standards. The calibration should only be reset when measured values are outside of the stated tolerance. The protocol should also include procedures that govern the control of calibration materials: storage (shelf-life), contamination-prevention and staff competence (approved users).

X3. REFLECTED VERSUS TRANSMITTED LIGHT

X3.1 Critical angle refractometers can function by using light transmission through the sample (usually a thin film sandwiched between two prisms) or by light reflection. A light reflection mechanism has the advantage that effects of absorption (highly colored samples) are minimized. A disadvantage of reflectance is the difficulty of observing the borderline in optical-mechanical (Abbe) instruments because of the low light intensity and poor contrast.

X3.2 Digital electronic refractometers usually work with a reflected light mechanism.

X4. GUIDELINES FOR QUALITY CONTROL

X4.1 The performance of the instrument or the test procedure should be confirmed by analyzing a quality control (QC) sample.

X4.2 It is recommended that the QC sample be representative of the material routinely analyzed. However, pure materials may be used, if so desired. An ample supply of QC sample material should be available for the intended period of use, and should be homogeneous and stable under the anticipated storage conditions.

X4.3 Prior to monitoring the measurement process, the average value and control limits of the QC sample need to be established (see Practice D6299 and ASTM MNL 7[5]). When a new QC sample material is required, control limits for the new material should be established before the old QC sample is exhausted.

---

[5] *ASTM MNL7*, “Manual on Presentation of Data Control Chart Analysis,” 6th Edition, available from ASTM International Headquarters.

X4.4 The QC results should be recorded, and analyzed by control charts or other statistically equivalent techniques to ascertain the statistical control status of the total testing process (see Practice D6299 and ASTM MNL 7[5]). Any out of control data should trigger an investigation of root cause(s). The results of such investigation may, but not necessarily, result in instrument recalibration.

X4.5 In the absence of explicit requirements given in the test method, the frequency of QC testing is dependent on the criticality of the quality being measured, the demonstrated stability of the testing process, the length of time required to do the test, and customer requirements. The QC testing frequency should be increased if a large number of samples are routinely analyzed. However, when it is demonstrated that the test method is under statistical control, the QC testing frequency may be reduced. The QC sample precision should be checked against the ASTM method precision to ensure quality of results.

X4.6 Consult Practice D6299 and ASTM MNL 7[5] for further guidance on QC and control charting techniques.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the Copyright Clearance Center, 222 Rosewood Drive, Danvers, MA 01923, Tel: (978) 646-2600; http://www.copyright.com/