Designation: D3012 – 13

Standard Test Method for Thermal-Oxidative Stability of Polypropylene Using a Specimen Rotator Within an Oven[1]

This standard is issued under the fixed designation D3012; 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 provides a means for estimating the resistance of polypropylene, in molded form, to accelerated aging by heat in the presence of air using a forced draft oven.

1.2 The stability determined by this test method is not directly related to the suitability of the material for use when different environmental conditions prevail and shall not be used to predict performance.

NOTE 1—The specified thermal levels in this test method are considered sufficiently severe to cause failure of commercial grades of heat-stable polypropylene within a reasonable period of time. If desired, lower temperatures can be applied to estimate the performance of polypropylene with lower heat stabilities.

1.3 The values stated in SI units are to be regarded as the standard. The values 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.*

NOTE 2—This test method and ISO 4577–1983 are technically similar but different in preparation of test specimens, thickness of test specimen, measurement of the number of air flow changes in the ovens, and the number of air changes per hour required.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D618 Practice for Conditioning Plastics for Testing

D883 Terminology Relating to Plastics

D3641 Practice for Injection Molding Test Specimens of Thermoplastic Molding and Extrusion Materials

D4101 Specification for Polypropylene Injection and Extrusion Materials

D5374 Test Methods for Forced-Convection Laboratory Ovens for Evaluation of Electrical Insulation

E77 Test Method for Inspection and Verification of Thermometers

E220 Test Method for Calibration of Thermocouples By Comparison Techniques

E608 Specification for Mineral-Insulated, Metal-Sheathed Base Metal Thermocouples

E644 Test Methods for Testing Industrial Resistance Thermometers

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E1137/E1137M Specification for Industrial Platinum Resistance Thermometers

E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

2.2 *ISO Standard:*[3]

ISO 4577–1983 Plastics—Polypropylene and Propylene Copolymers—Determination of Thermal Oxidative Stability in Air-Oven Method

ISO 1873 Plastics—Polypropylene and Propylene-Copolymer Thermoplastics:

Part 1: Designation

Part 2: Determination of Properties

3. Terminology

3.1 *Definitions*—The definitions of plastics used in this test method are in accordance with Terminology D883 unless otherwise indicated.

4. Summary of Test Method

4.1 Aging is accelerated by exposing the specimens to an elevated temperature in a forced draft oven equipped with a biaxial or uniaxial rotating specimen holder.

4.2 Visual examination is used to determine the time to failure. The time to failure of the material is taken as the number of days after which the specimen shows localized crazing, crumbling, or discoloration, or a combination thereof.

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials.

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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 American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

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

5. Significance and Use

5.1 Under the severe conditions of this test method, the specimens undergo degradation at a rate that is dependent upon the thermal endurance of the polypropylene material under examination.

5.2 The thermal level of this test method is considered sufficiently severe to cause failure of commercial grades of heat-stable polypropylene within a reasonable period of time. If desired, lower temperatures can be applied to estimate the performance of polypropylene materials with lower heat stability.

5.3 The technique of specimen rotation described in this test method provides an estimate of the life-temperature relationship of polypropylene. If this test method is conducted at different temperatures on the same material, a more reliable estimate of the life-temperature relationship of polypropylene is determined. This test method can be conducted at several temperatures and the data interpreted through use of the Arrhenius relation, by plotting the logarithms of times to failure against the reciprocals of the temperatures in kelvins (K). Temperatures in the range from 100 to 150°C, with intervals of 10°C, are suggested for this purpose.

5.4 The stability as determined under the prescribed test method is not directly related to the suitability of the compound for a use where different conditions prevail.

5.5 The specimen rotation technique of thermal aging increases the probability that all specimens will be exposed similarly and that the effect of temperature gradients in an oven will be minimized.

6. Apparatus

6.1 *Oven,* mechanical convection type for controlled circulation of air, with adjustable air intake and exhaust facilities, and designed for air velocities around 1000 ± 250 mm/s (197 ± 49 ft/min).[4]

6.1.1 The oven shall be equipped with a temperature-control system designed to maintain the test temperature range from 150 ± 1°C (302 ± 1.8°F) and a device to prevent temperature override. With the oven adjusted to the nominal test temperature of 150°C, the override shall be set at 154°C (309°F). A bimetallic-strip temperature switch has been found satisfactory.

6.2 *Oven Temperature Measurement System,* consisting of a thermocouple, thermometer, or resistance thermometer as the sensor, together with its associated conditions and readout instrumentation covering at least the temperature range from 0 to 200°C (32 to 372°F).

6.2.1 The thermometer must cover the range in one-degree subdivisions. It must be tested for bulb stability and standardized, in accordance with Test Method E77.

6.2.2 The secondary standard shall be ASTM Thermometer S67C–03 of Specification E2251.

6.2.3 Thermocouples shall comply with the requirements of Specification E608 and shall be calibrated in accordance with Method E220.

6.2.4 Resistance thermometers shall comply with the requirements of Test Methods E644 and Specification E1137/E1137M, and be calibrated in accordance with NIST Special Publication 250-22.[5,6]

6.3 *Molding Press,* designed to operate at 200 ± 5°C (392 ± 9°F).

6.4 *Injection Molding Unit,* meeting the requirements of Practice D3641.

6.5 *Mold:*

6.5.1 *Compression Mold,* comprised of the following:

6.5.1.1 *Compression Molding Chase,* having a blanked-out area of suitable size (Note 3) and capable of producing a plaque 1.00 ± 0.05 mm (0.039 ± 0.002 in.) thick.

NOTE 3—A 152.4 by 152.4-mm (6 by 6-in.) blanked-out section has been found satisfactory.

6.5.1.2 *Backing Plates,* large enough to cover this chase and strong enough to resist warping or distortion, under the molding conditions. Polished steel plates, 3 mm (0.1 in.) thick, are satisfactory.

6.5.2 *Injection Mold,* designed using the guidelines specified in Practice D3641. The mold shall be capable of producing either a plaque having a thickness of 1.00 ± 0.05 mm (0.039 ± 0.002 in.) from which 50 by 10 by 1.00-mm (2 by 0.4 by 0.039-in.) specimens can be die cut, or mold a standard test specimen, 50 by 10 by 1.00 mm.

6.6 *Parting Sheets*—Fluoropolymer, polyester, or other film that will not affect the long-term thermal stability of polypropylene, 0.05 to 0.20 mm (0.002 to 0.008 in.) thick. The film must be free of wrinkles and foreign matter, such as lubricants and oils.

6.7 *Cutting Die,* to produce 50 by 10-mm (2 by 0.4-in.) specimens from either a compression-molded or injection-molded plaque. The die must be sharp and free of nicks.

6.8 *Specimen Holder*—The specimen holder can be biaxially rotated or uniaxially rotated provided that the test specimens are in a stream of air having a relative velocity about 1000 mm/s (197 ft/min). Illustrations of suitable apparatus for biaxially and uniaxially rotated specimen holders are shown in Figs. 1 and 2, respectively. Biaxial rotation increases the probability that all specimens will be exposed similarly. In the case of dispute, the use of biaxial rotation shall be the reference method.

6.8.1 *Biaxially Rotated Specimen Holder* (see Fig. 1)[7]. The frequency of rotation about the horizontal and vertical axes shall be 1 to 3 min^{-1}.

6.8.2 *Uniaxially Rotated Specimen Holder* (see Fig. 2). The drum peripheral velocity shall be such that the air stream impinges on the flat section of the test specimens at about 1 m/s.

[4] A Precision Scientific Freas Model 835 B, or Blue M POM-206C-1, or equivalent, can be used.

[5] Mangum, B. W., "Platinum Resistance Thermometer Calibration," NBS Special Publication 250-22 (1987).

[6] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 3460, Gaithersburg, MD 20899-3460.

[7] Standard Scientific Supply Company, Model CS191, or equivalent, can be used.

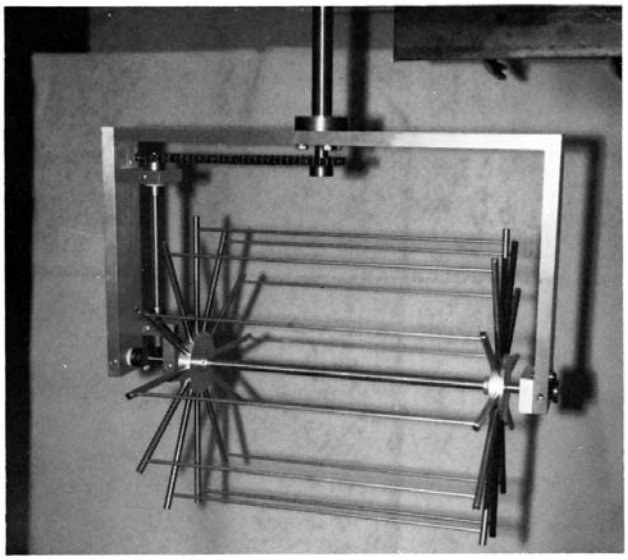

FIG. 1 Biaxial Ferris-Wheel-Type Rotator

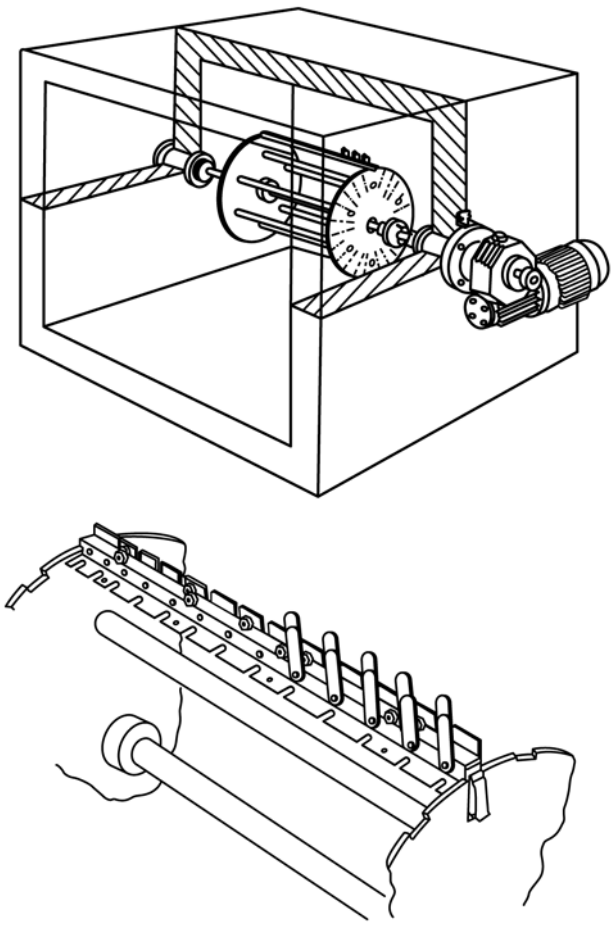

FIG. 2 Uniaxially Rotated Specimen Holder—Drum Rotator

6.9 *Air Velocity Meter,* nondirectional resistance wire type for measuring the air velocity in the oven.

6.10 *Anemometer,* with a circular vane, for determining the frequency of air changes in the oven. The anemometer shall be positioned directly in front of the oven outlet. Based on the design of the outlet and the anemometer, every effort shall be made to position the anemometer's electronics in a way as to minimize the degree of exposure to the heated air exiting the oven.

6.11 *Metal Clips,* lined with fluorocarbon film or other materials that have no adverse effect on the oxidative thermal stability of polypropylene.

7. Specimen Preparation

7.1 The test specimens shall be cut from either a compression–molded plaque or injection-molded plaque prepared from granules or other homogeneous molding material. Plaques shall be prepared as follows:

7.2 *Compression Molding:*

7.2.1 Adjust the temperature of the platens to 200 ± 5°C (392 ± 9°F).

7.2.2 Place a smooth, clean parting sheet on a backing plate and center the chase on it. Put enough of the sample into the cavity to fill it completely when molded. A slight excess of material is desirable. Cover the loaded chase first with a clean parting sheet and then a backing plate. Finally, put the assembled mold on the lower platen and close the press carefully until both platens are in contact with the assembly. When the material has melted, apply sufficient pressure to form a void-free plaque in the 1.00 ± 0.5 mm (0.039 ± 0.002 in.) thick and record this pressure. Leave the polypropylene in the heated press under pressure for 3 to 4 min at 200 ± 5°C (392 ± 9°F). Flash cool the mold assembly by transferring to a water-cooled press or by water quenching.

7.3 *Injection Molding:*

7.3.1 Plaques or test specimens shall be injection-molded in accordance with the requirements of Specification D4101.

7.4 Prepare a minimum of five specimens per material sample by die-cutting specimens from the plaque or directly molding the standard test specimen. The standard specimen shall be 10 mm wide, 50 mm long, and 1.00 ± 0.05 mm thick. Edges shall be smoothed, if necessary, to remove imperfections introduced by cutting.

7.5 Test specimens prepared directly by injection molding without die cutting or specimens cut from polypropylene products can be used as agreed upon between the interested parties. In all cases the referee method will be based on compression molded die cut specimens.

NOTE 4—Failure test times for compression-molded and injection-molded specimens are not necessarily comparable due to the skin surface effect and the distinctness or sharpness of the specimen edge.

7.6 Specimens of other thicknesses can be used as agreed upon between the interested parties.

7.7 In cases of dispute, the referee specimens shall be die-cut only from compression-molded specimens, unless the test specimen is otherwise agreed upon.

7.8 In handling the plaques and cut specimens, use clean gloves or tongs to prevent contamination of the test specimens.

8. Conditioning

8.1 Condition the test specimens in accordance with Specification D4101. When unsure of the material formulation, condition the test specimen at 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 10 % relative humidity for not less than 40 h prior to test in accordance with Procedure A of Practice D618. In cases of disagreement, the tolerances shall be ±1°C (±1.8°F) and ±5 % relative humidity.

9. Oven Parameters

9.1 Unless otherwise specified, the oven temperature shall be 150°C (302°F) and shall not vary more than ±1°C, measured at a single location within the oven, or more than ±1.5°C throughout the oven.

9.1.1 To monitor temperature, the oven temperature measurement sensor should be conveniently located about 80 mm (3 in.) from the top liner of the oven, in the vicinity of the exhaust port.

9.2 In the event that the average time to failure at 150°C is less than seven days, the test report shall indicate "less than seven days at 150°C" and the test shall be repeated at an oven temperature of 140 ± 1°C, and, if necessary, at lower temperatures at intervals of 10°C until time to failure of seven days is achieved.

NOTE 5—If it is preferred to perform the test at 140°C, this is permitted provided that this is stated in the test report. This is applicable even for materials lasting more than seven days at 150°C.

9.3 The air velocity shall be 1000 ± 250 mm/s (197 ± 49 ft/mm) during the course of the test as measured at the center of the oven.

NOTE 6—The air velocity in the oven is dependent on the design of the oven and will not vary a great deal. It should be checked occasionally to determine if the blowers and other moving parts are functioning properly.

NOTE 7—The air velocity should be measured at +23°C for non-thermally corrected anemometers. A thermally-corrected anemometer can be used in the range from +20 to +80°C. In no case should an anemometer be used to measure air flow at the test temperature. Typically an anemometer will have a 25 % error for each 35°C above its maximum calibrated temperature point.

9.4 The air intake and exhaust ports shall be adjusted to allow approximately 1 ± 15 % air volume change/min.

9.4.1 The number of air changes that take place in the oven should be determined by the power consumption technique described in Test Methods D5374.

9.4.2 An anemometer can be used to measure the number of air exchanges also. This method is described in Appendix X1. If used, this method should be verified with the power consumption technique, and the anemometer value must be within 15 % of the number of air changes observed with the power consumption method. The advantages of the anemometer method is that is it quick, easy, and can be conducted on a more frequent basis to ensure that no changes have occurred in the settings or conditions in the oven. It is not an absolute measure.

10. Procedure

10.1 The metal clips with fluoropolymer lining shall be cleaned beforehand with trichloroethylene or any other suitable solvent to remove all traces of oil.

10.2 Attach the five specimens to the biaxial rotator by suitable metal clips lined with fluoropolymer film. Space the specimens 30 to 40 mm (1.2 to 1.6 in.) from each other on the rack. (**Warning—**Handle specimens with caution using clean gloves to avoid contamination.)

10.2.1 Avoid direct contact of the specimens with the metal clips or metal parts. Certain metals, such as aluminum, are known to affect with the long-term thermal endurance of some polypropylenes.

10.3 Inspect the specimens at least once a day, turning off the biaxial rotator during inspection.

10.4 Failure by this test method for polypropylene is visual evidence of localized discoloration and crumbling, crazing or pin holes that open when sample is flexed on any part of the specimen directly exposed to the air flow. The area within 5 mm of the clips shall not be included in this evaluation.

NOTE 8—When testing polypropylene containing recycled material localized discoloration often appears on the surface of the specimen within a few days of exposure to the heat. This should not be interpreted as a failure unless the criteria of failure specified in 10.4 are met.

NOTE 9—Visual inspection of the deterioration of the specimen surface, as a reliable evaluation of oxidative aging endurance, is justified by the fact that oxidation of propylene thermoplastics usually takes place on the surface of the material before propagating inward.

10.5 Record the time to failure of each specimen in days.

11. Report

11.1 The report shall include the following information:

11.1.1 A complete identification of the sample,

11.1.2 The average time to failure, in days at 150°C[8] of the five specimens,

11.1.3 The range of times to failure in days at 150°C[8] of the five specimens,

11.1.4 The preparation or history of the specimen,

11.1.5 The thickness of the specimen,

11.1.6 The type of rotator,

11.1.7 The test temperature, if other than 150°C,

11.1.8 Any departures from the specified test conditions, and

11.1.9 Details of conditioning (temperature and humidity) if appropriate.

12. Precision and Bias

12.1 Table 1 is based on a round robin conducted in 1970 in accordance with Practice E691, involving three materials tested by seven laboratories. Each material was supplied from one source, but the individual specimens were prepared at the laboratory that tested the specimens. Test specimens were 1.25 mm (0.50 in.) in thickness. Each of two test results that was obtained by each laboratory was the average of five individual determinations. (**Warning—**The following explanations of *r* and *R* (12.2 – 12.2.3) are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Table 1 should not be rigorously applied

[8] If the time to failure is less than seven days at 150°C, report as "less than seven days at 150°C." In addition, report the time (range of times) to failure at 140°C (or at other temperatures used).

TABLE 1 Precision Data for Oxidative Stability of Polypropylene

Material	Average Time to Failure, days	S_r [A]	S_R [B]	r [C]	R [D]
L	14.0	0.765	2.63	2.16	7.44
M	34.9	3.08	6.95	8.72	19.6
H	63.4	5.42	18.7	15.3	52.9

[A] S_r = within-laboratory standard deviation of the average.
[B] S_R = between-laboratory standard deviation of the average.
[C] $r = 2.8 \times S_r$.
[D] $R = 2.8 \times S_R$.

to acceptance or rejection of material, as those data are specific to the round robin and are not necessarily representative of other lots, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of 12.2 – 12.2.3 would then be valid for such data.)

12.2 *Concept of r and R*—If S_r and S_R have been calculated from a large enough body of data, and for test results that were averages from testing five test specimens:

12.2.1 *Repeatability (r)*—In comparing two test results for the same material, obtained by the same operator using the same equipment on the same day, the two test results should be judged not equivalent if they differ by more than the I_r value for that material.

12.2.2 *Reproducibility (R)*—In comparing two test results for the same material, obtained by different operators using different equipment on different days, the two test results should be judged not equivalent if they differ by more than the I_R value for that material.

12.2.3 Any judgment in accordance with 12.2.1 and 12.2.2 would have an approximate 95 % (0.95) probability of being correct.

12.3 *Bias*—There are no recognized standards on which to base an estimate of bias for this test method.

13. Keywords

13.1 aging; biaxial rotator; polypropylene; thermal aging; thermal oxidative stability

APPENDIX

(Nonmandatory Information)

X1. SECONDARY METHOD FOR DETERMINING THE NUMBER OF AIR EXCHANGES

X1.1. Scope

X1.1.1 The purpose of this appendix is to provide a secondary method for determining the number of air exchanges that occur within the forced draft oven used for measurement of thermal oxidative stability.

X1.2. Summary of Test Method

X1.2.1 This method uses an anemometer to measure the linear velocity of the air exiting the heated oven through the oven exhaust outlet. Knowing the volume of the interior of the oven and the cross-sectional area of the exhaust outlet and linear velocity of the air, the number of air volume changes occurring in the oven over a 1-h period can be calculated.

X1.3. Apparatus

X1.3.1 *Anemometer,* with circular vane. The vane shall be of equal or smaller diameter than the inside diameter of the exhaust outlet. The electronics within the anemometer shall be below the level of the heated exhaust to prevent the electronics from being heated and errors in calibration occurring. The unit should have a digital display of the air velocity.

X1.3.2 *Temperature Measuring Devices,* to determine the temperature in the room and in the oven.

X1.4. Procedure

X1.4.1 Ensure that the anemometer has been properly calibrated before using.

X1.4.2 Position the vane of the anemometer at the exhaust outlet, ensuring that it is centered on the outlet.

X1.4.3 Monitor the digital display of the anemometer until the reading stabilizes. Record this value as the linear air velocity in metres per minute. Move the vane away from the exhaust port and allow it to cool. Once cool, repeat the air velocity measurement. Ensure that the anemometer is cooled between each measurement to prevent heat buildup that might effect the electronics. Make a minimum of three measurements of the air velocity.

X1.4.4 Determine the volume of the interior of the oven in cubic metres.

X1.4.5 Determine the inside cross-sectional area of the exhaust outlet, in square metres.

X1.4.6 Measure the air temperature within the laboratory to the nearest degree, in degree centigrade.

X1.4.7 Measure the air temperature within the oven, in degree centigrade.

X1.5. Calculation

X1.5.1 Determine the average linear air velocity from data collected in X1.4.3.

X1.5.2 Convert the oven temperature and the room temperature from degree centigrade to degree Kelvin (°C + 273 = K)

X1.5.3 Using the Perfect Gas Law, PV = *n*RT, and assuming pressure remains constant, the volume of air once heated in the oven that will exit the exhaust outlet can be determined as follows:

$$V_1/V_2 = T_1/T_2 \quad \text{(X1.1)}$$

where:

V_1 = volume of air in the oven at room temperature, m^3,
V_2 = volume of air in the oven at the set temperature, m^3,
T_1 = temperature of the room, K, and
T_2 = temperature of the oven, K.

NOTE X1.1—Should V_1 be more than 3° from +23°C, it is required that you calculate V_1 first using the following equation:

$$V_1 = V_0 \times (T_1/296) \quad \text{(X1.2)}$$

where:

V_0 = volume of oven calculated from dimensions at +23°C, m^3, and
296 = 296K = conversion of 23°C to K (kelvin).

X1.5.4 Calculate the air volume discharged from the oven as follows:

$$CMH = V_3 \times A \times (V_1/V_2) \times 60 \quad \text{(X1.3)}$$

where:

CMH = air volume exiting the exhaust outlet per hour, m^3/h,
V_3 = air velocity of air exiting the exhaust outlet, m/min,
A = cross-sectional area of exhaust outlet, m^2, and
60 = conversion from minutes to hours.

X1.5.5 Calculate the number of air changes per hour as follows:

$$AC = CMH/VO \quad \text{(X1.4)}$$

where:

AC = number of air changes per hour.

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D3012 - 07) that may impact the use of this standard. (April 1, 2013)

(1) Editorially corrected air velocity specification in 6.1.
(2) Updated conditioning requirements in 8.1
(3) Moved old Warning Note 8 to the end of 10.2.
(4) Edited permissive language.