Designation: D 4703 – 00

# Standard Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets[1]

This standard is issued under the fixed designation D 4703; 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 practice covers the compression molding of thermoplastic granules and milled stock for the preparation of test specimens.[2]

1.2 While conditions for certain materials are given, the primary source of specific conditions shall be the material specification standards for each type of material.

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.*

NOTE 1—The main body of this practice is equivalent to ISO 293-1986. Annex A1 and ISO 293-1986 differ in some details; however, specimens prepared using Annex A1, Procedure A should be equivalent to those prepared using ISO 293-1986, Cooling Method D. Specimens prepared using Annex A1, Procedure C should be equivalent to those prepared using ISO 293-1986, Cooling Method B. However, due to the greater cooling rate tolerances of the ISO standard, specimens prepared according to ISO Cooling Method B may not be equivalent to Annex A1, Procedure C.

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 618 Practice for Conditioning Plastics for Testing[3]

D 1248 Specification for Polyethylene Plastics Extrusion Materials for Wire and Cable[3]

D 1928 Test Method for Preparation of Compression-Molded Polyethylene Test Sheets and Test Specimens[3]

D 3350 Specification for Polyethylene Plastics Pipe and Fittings Materials[4]

D 4976 Specification for Polyethylene Plastics Molding and Extrusion Materials[5]

ISO 293-1986 Plastics—Compression Moulding Test Specimens of Thermoplastic Materials[6]

## 3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *average cooling rate (°C/min), n*—the cooling rate calculated by dividing the difference between the molding and demolding temperatures by the time required to cool the mold to the demolding temperature.

3.1.2 *cooling rate (°C/h), n*—the rate of cooling obtained by controlling the flow of the cooling fluid in such a way that during each 10-min interval, the deviation from this specified cooling rate shall not exceed the specified tolerance.

3.1.3 *demolding temperature, n*—the temperature of the mold or the press platens at the end of the cooling time, measured in the nearest vicinity to the molded material.

3.1.3.1 *Discussion*—For positive molds, holes are normally drilled in the mold for measuring the temperatures defined in 3.1.3 and 3.1.4.

3.1.4 *molding temperature, n*—the temperature of the mold or the press platens during the preheating and molding time, measured in the nearest vicinity to the molded material.

3.1.5 *molding time, n*—the time during which full pressure is applied while maintaining the molding temperature.

3.1.6 *picture frame mold, n*—a flat piece of metal, usually of brass or steel, that has a center portion removed to provide the specified shape and dimensions of the final molding. The thickness of the metal is dependent on the desired thickness of the finished molding, taking into consideration the shrinkage of the material to be molded. The picture frame mold is sometimes referred to as a chase.

3.1.7 *preheating time, n*—the time required to heat the material in the mold up to the molding temperature while maintaining the contact pressure.

## 4. Significance and Use

4.1 The methods by which sample materials are prepared

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[1] This practice is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.09 on Specimen Preparation.

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[2] This practice was created as a coalescence of and replacement for Practices D 1928, D 2292, D 3010, and D 3463.

[3] *Annual Book of ASTM Standards*, Vol 08.01.

[4] *Annual Book of ASTM Standards*, Vol 08.02.

[5] *Annual Book of ASTM Standards*, Vol 08.03.

[6] ISO/IEC Selected Standards for Testing Plastics, available from ASTM. Also available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

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

and molded influence the mechanical properties of the specimen. Unlike injection molding, the objective of compression molding is to produce test specimens or sheets that are both homogeneous and isotropic. Molded specimens may be made from powder or pellets such as are received directly from a material manufacturer, particles produced in a recycle recovery operation, or from a milled preform or sheet prepared on a two-roll mill. The powder, pellets, particles, preform, or sheet are melted and molded in a mold designed to produce a finished specimen of a given geometry, size, and thickness, or melted and molded in the form of a smooth plaque or sheet of uniform thickness from which desired specimens are cut, punched, or machined. Working a compound on a two-roll mill prior to molding will disperse and distribute the compound additives in a manner that will affect the physical properties of the compound. The need for milling a sample prior to compression molding may be determined by reference to the relevant material specification or the material manufacturer. It is important to treat different samples of the same type of material in the same way: if milling was done prior to molding on a material which is to be used as a standard for comparison, all new materials to be tested against this practice should be prepared and molded in a similar manner.

4.2 The apparatus and exact conditions required to prepare adequate specimens may vary for each plastic material. Apparatus and procedures which should be satisfactory for molding many different plastic materials are given in this practice in Sections 5 and 6. The apparatus and procedures which have been found satisfactory for molding certain specific materials are given in the Appendix. In any case, the apparatus and procedures to be used in producing compression-molded specimens of a given material may be obtained by reference to the relevant material specification and should be agreed upon between the purchaser and the supplier.

## 5. Apparatus

5.1 *Mill*—Any size two-roll mill having chrome-plated rolls, capable of maintaining a constant temperature within ± 2°C (± 3.6°F) of the temperature needed for the particular material involved, and being adjustable in speed as needed for the material to be worked, is adequate. Some recommendations for mills to be used for specific types of materials are given in the Appendix.

5.2 *Molds*:

5.2.1 *Mold Types*—Several different types of molds may be used for the compression molding of test specimens of thermoplastics. In general, however, the molds used will fall into one of two categories: a *flash-type* mold (see Figs. 1 and 2) or a *positive-type* mold (see Fig. 3). The characteristics of the test specimens prepared by using different types of molds are not the same. In particular, some mechanical properties may be affected by the pressure applied to the material during cooling.

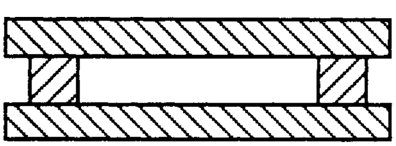

**FIG. 1 Flash Picture-Frame Mold**

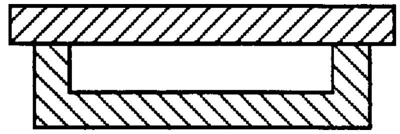

**FIG. 2 Flash Mold with Machined Cavity**

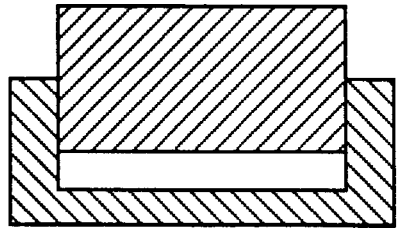

**FIG. 3 Positive-Type Mold**

5.2.2 *Flash-Type Mold*—The flash-type mold may be of the *picture-frame* type, where a metal chase (the picture frame) is sandwiched between two thin metal ferrotype plates (see Fig. 1), or it may be of the machined-cavity type (see Fig. 2), where the mold consists of a cavity machined in a metal plate, with a single metal ferrotype plate used as a top or cover. The cavity, or cavities, in the flash-type mold may be constructed to mold a single plaque from which test specimens may be stamped or machined, or the mold may be built to mold one or more specimens to finished dimensions. Flash molds permit excess molding material to be squeezed out and do not exert molding pressure on the material during cooling. Nevertheless, this type of mold is useful for preparing test specimens or panels of similar thickness or comparable levels of low internal stress.

5.2.3 *Positive-Type Mold*—The positive-type mold consists of a cavity machined in a plate or block of metal and a force or plunger which closely mates with the sidewalls of the cavity (see Fig. 3). Like the flash-type mold, the cavity may be built to produce a plaque from which test specimens may be stamped or machined, or to mold a test specimen to finished dimensions. Because of the manner in which the positive mold operates, it is recommended that this mold type, either for a plaque or a finished specimen, be limited to a single cavity. In the positive-type mold, the full molding pressure, neglecting friction, is exerted on the material during cooling. The thickness, stress and density of the resulting moldings depend on the mold construction, the size of the material charge, and the molding conditions. This type of mold produces test specimens with high density, and it is particularly suitable for obtaining flat surfaces and for suppressing the formation of voids within test specimens.

5.2.4 *Mold Surfaces*—The surfaces of either type of mold that form the flat faces of the specimen should be finished to the degree required by the test for which the specimen is intended. For most tests, a surface quality of SPI-SPE #2 is adequate.[7] The edges of the mold cavity should be visually free of nicks and scratches which could cause premature failure of the specimen during testing.

5.3 *Press*:

[7] Mold comparison kits are available from D-M-E Company, 29111 Stephenson Highway, Madison Heights, MI 48071.

5.3.1 The press shall have a clamping force capable of applying a pressure (conventionally given as the ratio of the clamping force to the area of the mold cavity) of at least 10 MPa (1450 psi), and shall be capable of maintaining pressure within 10 % of the specified pressure during the molding cycle.

5.3.2 The platens of the press shall be capable of being heated to at least 240°C, and being cooled at a rate consistent with the cooling method selected from Table 1.

5.3.3 The platens or mold shall be heated either by high-pressure steam, by a heat-conducting fluid in an appropriate channel system, or by using electric-heating elements. The platens or mold are cooled by a heat-conducting fluid (usually cold water) in a channel system.

5.3.4 The heating and cooling systems in the mold platens shall be such that, when used with a particular mold, they shall be capable of maintaining a temperature difference between points on the mold surfaces of no more than ±5°C during heating or cooling.

5.3.5 For quench cooling (Method C in Table 1), two presses shall be used, one for heating during molding and the other for cooling unless it can be demonstrated that the press used for heating can cool at the specified rate.

NOTE 2—For a specified cooling method, the flow rate of the heat-conducting fluid should be predetermined in a test without any material in the mold.

## 6. Procedure

6.1 *Preparation of Molding Material*:

6.1.1 *Drying of Granular Material*—Dry the granular material as specified in the relevant material specification, or in accordance with the material supplier's instructions. If no instructions are given, dry for 24 ± 1 h at 70 ± 2°C in an oven.

6.1.2 *Preparation of Preforms*—Direct molding of test specimens, plaques, and sheets from granules shall be the standard procedure, provided that a sufficiently homogeneous sheet is obtained. Normally this means that the molded specimen, plaque, or sheet is free from surface irregularities and internal imperfections. Poly(vinyl chloride) (PVC) compounds and chlorinated poly(vinyl chloride) (CPVC) compounds will generally require milling to obtain a preform for the final molding procedure.

6.1.3 *Milling*—Direct molding from powder or granules may sometimes require melt homogenization using a hot-melt milling or mixing procedure to achieve a satisfactory final sheet. Where such is required, a two-roll mill will usually perform satisfactorily. Take the milled material from the mill and cut or shape it to become a preform for the compression mold in which it ultimately will be molded. Use milling conditions that do not degrade the polymer. Recommended conditions for milling the material, particularly the stock temperature and time on the rolls, may be obtained from the relevant material specification or the material manufacturer. The preform prepared by milling should normally be thicker than the specimen, plaque, or sheet to be molded to enable the molding to be done properly.

**TABLE 1 Cooling Methods**

| Cooling Method | Average Cooling Rate (See 3.1.1), °C/min | Cooling Rate (See 3.1.2), °C/min | Remarks |
|---|---|---|---|
| A | 10 ± 5 | | |
| B | 15 ± 5 | | |
| C | 60 ± 30 | | Quench cooling |
| D | | 5 ± 0.5 | Slow cooling |

6.2 *Molding*:

6.2.1 Adjust the mold temperature to within ±5°C of the molding temperature indicated in the relevant material specification. With picture-frame (Fig. 1) molds or large, heavy molds it may not be necessary or desirable to preheat the mold itself. This will then require slight increases in the preheat time of the cycle; the temperature stability of the material shall be considered.

6.2.2 Place a weighed quantity of the material (granules or preforms) in the preheated mold. If granular material is used, make sure that it is evenly spread over the mold surface. The mass of the material shall be sufficient to fill the cavity volume when it is melted and allow about a 10 % loss for a flash mold and about a 3 % loss for a positive mold. With flash molds, cover the mold with the top ferrotype plate (see Figs. 1 and 2) and then place the mold in the preheated press.

6.2.3 Close the press and preheat the material charge by applying a contact pressure for a minimum of 5 min. Then apply full pressure for a minimum of 2 min (molding time, see 3.1.5) and then cool down (see 6.3).

NOTE 3—A preheating time of 5 min is the standardized time for evenly spread material charges sufficient for sheets up to 2 mm in thickness. For thicker moldings, adjust the time accordingly.

NOTE 4—At contact pressure the press is just closed with a pressure low enough to avoid flow of the material. Full pressure means a pressure sufficient to shape the material and squeeze out the excess material.

6.3 *Cooling*:

6.3.1 *General*—With some thermoplastics, the cooling rate affects the ultimate physical properties. For this reason, the cooling methods are specified in Table 1. The method of cooling shall always be stated together with the final physical properties. The appropriate cooling method is normally given in the relevant material specification. If no method is indicated, Method B shall be used.

6.3.2 *Cooling Methods*—The appropriate cooling method shall be selected from Table 1.

6.3.2.1 In the case of quench cooling (see Method C in Table 1), transfer the mold assembly from the heating press to the cooling press as quickly as possible. If the heating press has the capability to cool at the specified rate, it may be used for the cooling step.

6.3.3 The demolding temperature shall be <40°C if no other instructions are given.

NOTE 5—Method D is recommended for producing test specimens free of any internal stress, or for slow cooling after annealing of previously prepared sheets.

## 7. Inspection of the Molded Specimens, Plaques, or Sheets

7.1 After cooling, check the molded specimens, plaques, or sheets for appearance (such as sink marks, shrink holes, discolorations) and for conformance to specified dimensions. Discard any test specimens or sheets having molding defects.

7.2 Make sure there is no degradation or unwanted crosslinking, using the method specified in the relevant material specification, or as agreed upon between the interested parties.

## 8. Report

8.1 Provide the following information in the processing report:

8.1.1 Reference to this practice and the relevant material specification,

8.1.2 Dimensions of the specimen and its intended use,

8.1.3 Complete identification of molding material (type, designation, etc.),

8.1.4 Preparation of molding material:

8.1.4.1 Drying conditions for granules and powder, and

8.1.4.2 Processing conditions used in the preparation of preforms and their average thickness,

8.1.5 Type of mold and plates used,

8.1.6 Molding conditions:

8.1.6.1 Preheating time,

8.1.6.2 Molding temperature, pressure, and time,

8.1.6.3 Cooling method used, and

8.1.6.4 Demolding temperature,

8.1.7 State of specimen, if applicable, and

8.1.8 Any other observations.

## 9. Precision and Bias

9.1 No statement is made about either the precision or the bias of this practice for preparation of compression-molded test specimens since there is no numerical result.

## 10. Keywords

10.1 acrylonitrile-butadiene-styrene (ABS); chlorinated poly(vinyl chloride) (CPVC); compression molding; polyethylene (PE); poly(vinyl chloride) (PVC); styrene-butadiene; test specimen preparation; thermoplastics

# ANNEXES

**(Mandatory Information)**

## A1. PROCEDURES FOR POLYETHYLENE

A1.1 *Scope*—This annex covers the preparation of compression-molded test sheets of Class 1, 2, 3, 4, and 5 polyethylene plastics (Specification D 4976) and Types 0, I, II, III, and IV (Specifications D 1248 and D 3350). This annex includes both branched and linear polyethylenes. Two of the procedures given provide for compression-molded sheets to be conditioned by first heating each material above its melting point for a period of time sufficient to erase its prior thermal history and then cooling it from the melt state at a controlled rate while maintaining the required dimensions of the sheet. The third procedure provides for molded test sheets to be prepared by cooling the platens of the compression press, and hence the molten polyethylene plastic, at a controlled rate. Three cooling schedules are provided for as follows:

NOTE A1.1—The specimen preparation procedures in this annex were originally published as ASTM Test Method D 1928-96.

A1.1.1 *Procedure A*, in which the temperature of the initially molten plaque is lowered at a rate of 5 ± 0.5°C/h.

NOTE A1.2—It is recognized that Procedure A may not be applicable to material containing carbon black due to difficulties, at times, with sheets containing voids. If it is not possible to mold a void-free sheet, Procedure B or C should be selected.

A1.1.2 *Procedure B*, in which the initially molten plaque is chilled very rapidly under specified conditions in water, and

A1.1.3 *Procedure C*, in which the temperature of the platens of the molding press, and hence of the initially molten plaque, is lowered at a rate of 15 ± 2°C/min.

A1.2 *Significance and Use*—The conditions under which a polyethylene plastic is formed into a test sheet, particularly the rate of cooling, influence some of the properties of test specimens taken from the sheet. It is, therefore, necessary to control the cooling rate of the test sheet. These procedures are intended to minimize interlaboratory variability of test results on compression-molded specimens arising from differences in rate of cooling.

A1.2.1 Procedures A and B are designed also to erase moderate differences in the prior thermal history by employing a conditioning period of l h at a temperature about 25°C above the melting point of the polyethylene plastic prior to cooling at a controlled rate. For Procedures A and B, the conditioning temperature may have to be raised above this minimum value in some cases to promote adhesion to the aluminum foil parting sheets. The following temperatures, which depend on the Class of polyethylene plastic as classified in Specification D 4976 (Type in D 1248 and D 3350), have been found to be generally useful:

| | |
|---|---|
| Class I (Type I) | 140°C |
| Class 2 (Type II) | 150°C |
| Class 3 (Type III) | 155°C |
| Class 4 (Type IV) | 155°C |
| Class 5 (Type 0) | 140°C |

A1.2.2 Values obtained on specimens from test sheets prepared in accordance with Procedure C of this annex are useful for the identification of type or class of polyethylene plastics in accordance with Specifications D 1248, D 3350 and D 4976.

A1.2.3 Results obtained on specimens from test sheets prepared by any of the procedures described do not necessarily reflect the properties of articles fabricated by other methods, such as extrusion and injection molding.

A1.3 *Apparatus:*

A1.3.1 *Two-Roll Mill*, that can be heated to a temperature high enough to flux the materials to be tested. Heating may be by steam, electricity, or other suitable means.

A1.3.2 *Press*, with platens that can be heated to at least 150°C for Class 1 (Type I), 160°C for Class 2 (Type II), and 177°C for Class 3 and 4 (Type III and IV), and 140°C for Class 5 (Type 0) polyethylene plastics.

NOTE A1.3—Pressure is not a key parameter of the molding of polyethylene. Available data shows no additional variability imparted to low, medium or high density polyethylene density measurements when ram force was varied from ten tons to twenty-eight tons.

A1.3.3 *Molds*, of the flash picture-frame variety (also known as chases) or flash molds with machined cavities shall be used.

A1.3.4 *Backing Plates*, flat, for the chases. The backing plates should be strong enough to resist warping or distortion under the molding conditions. Plates made from high thermal conductivity materials such as aluminum, copper or bronze plates or polished steel, of 3.2 to 12.7 mm thick, are suitable.

A1.3.5 *Aluminum Foil*, 0.05 to 0.2 mm thick, for use as a parting agent in the molding operation.

NOTE A1.4—It is necessary to use aluminum foil of the specified thickness as a parting agent in the molding operation in Procedures A and B. Much thinner foil is not stiff enough to resist the tendency of the plastic surface to wrinkle on fluxing, while thicker foil does not conform well enough to the plastic surface. Aluminum alloy 1100, Temper O, is suitable.

A1.3.6 *Oven*, capable of maintaining a temperature of at least 175°C. Temperature variation within the oven should be less than 4°C, and the oven should be large enough to conveniently accommodate a one day production of molded sheets.

A1.3.7 *For Procedure A Only*:

A1.3.7.1 *Device* for lowering the oven temperature at a rate of 5.0 ± 0.5°C (9.0 ± 0.9°F)/h.

NOTE A1.5—A suitable device may be made by connecting a motor-driven thermoregulator or a programmer through a relay to the oven heaters.[8] An alternative and equally satisfactory device comprises a controller and a small electric motor geared to lower the oven temperature at the specified rate.[9]

A1.3.8 *For Procedure B Only*:

A1.3.8.1 *Cooling Tanks*—Tanks into which water may run and continuously overflow and which are large enough to accommodate the chases but small enough to fit into a laboratory sink. Each tank shall contain a mesh basket to hold the chases suspended in the cooling water. The basket should be made with as open a construction as possible to facilitate circulation of cooling water over all surfaces of the molding.

A1.3.9 *For Procedure C Only*:

A1.3.9.1 *Polyester Film*, or aluminum foil for use as a parting agent in the molding operation.

NOTE A1.6—Studies have shown that the type of parting sheet, that is, polyester film or aluminum foil, may cause minor but measurable differences in certain properties. In cases of dispute, the type of parting sheet should be agreed upon by the parties involved.

A1.3.9.2 *Device* for lowering the temperature of the platens at a rate of 15 ± 2°C/min.

A1.4 *Roll Milling:*

A1.4.1 Compression moldings can be made directly from the granules, pellets, or powders. However, if insufficient flow or inadequate adhesion between particles is observed, materials can be roll milled to ensure homogeneity.

NOTE A1.7—During the milling operation, the addition of an antioxidant is desirable if the molded test sheet is not used subsequently for tests of thermal, oxidative, or environmental stress cracking stability where such additives will interfere.

A1.4.2 Mill rolls should be hot enough to flux materials, but not so hot as to cause them to drip. The crepe should be slashed or turned frequently to promote mixing. Materials should not be milled for more than 5 min after gelling to minimize oxidative and thermal changes.

A1.5 *Compression Molding Procedure:*

A1.5.1 A double pressing technique may sometimes be required to squeeze entrapped air out of mill-massed material; if the air is not removed, conditioned sheets may develop voids. A single pressing is usually sufficient, however, to produce void-free test sheets from most polyethylene plastics.

A1.5.2 A flash-type molding operation is involved. The subsequent conditioning step will not give good results unless excess material is squeezed out on all sides and both surfaces of the chase.

NOTE A1.8—The presence of sink marks in the molded sheet, or the failure of the aluminum foil to adhere tightly all around the chase, usually indicates that an insufficient amount of material has been charged. Failure of the aluminum foil to stick to the plastic can often be remedied by pressing and subsequently conditioning at a higher temperature.

A1.5.3 The press temperature should be warm enough to result in good adhesion between the plastic and the aluminum foil. Recommended minimum temperatures for the press platens are 150°C for Class 1 (Type I), 160°C for Class 2 (Type II), and 177°C for Class 3 and 4 (Type III and IV), and 140°C for Class 5 (Type 0) polyethylene plastics.

A1.5.4 Weigh out the amount of plastic necessary to fill the blanked-out volume of the chase and provide an excess, for flash, of not less than 2 and not more than 10 % of the weight of the final molding. (For this purpose, densities of 0.92, 0.93, 0.95, 0.97 and 0.90 may be assumed for Class 1, 2, 3, 4, and 5 (Type I, II, III, IV and 0) polyethylene plastics, respectively. The densities of most carbon black formulations are about 0.01 g/cm$^3$ higher than those of the natural materials.)

A1.5.5 *For Procedures A and B*:

A1.5.5.1 Compression molding is performed using parting sheets of aluminum foil. The foil should be cleaned by wiping with acetone and then dried with clean, absorbent paper or cloth. Any wrinkles in the foil should be smoothed out before use. While the foil is usually not suitable for reuse, it may be used again if undamaged.

A1.5.5.2 *Safety Precautions*—See acetone Material Safety Data Sheets for specific hazards information on these materials. The OSHA requirements for 8-h time weighted average

[8] Detail drawings of a suitable device are available from ASTM Headquarters. Request Adjunct No. 12–419280–00.

[9] Components of this alternative device may be purchased from the Haydon Corp., Tarrington, CT, and the West Instrument Corp., 4363 W. Montrose Ave., Chicago, IL 60641. The West Guardsman Model JP Controller has been found satisfactory as the controller.

and acceptable ceiling concentration are found in 29 CFR, CH XVII, Table Z-2.[10]

A1.5.5.3 Place the polyethylene plastic in the chase between cleaned aluminum foil parting sheets backed by smooth backing plates. With the platens at the correct temperature, insert the assembly between the platens. Allow a heating period of 5 min with the platens closed and contact pressure applied. Then bring to full pressure as quickly as possible. The dwell time at full pressure and cooling rate are optional. After the molding has been formed, remove the assembly from the press. Carefully pry the backing plates off without disturbing the aluminum foil, which should be adhering tightly to the chase and polyethylene plastic sheet. Handle the sheet, chase, and adhering aluminum foil as a unit from this point.

A1.5.5.4 If a second pressing is desired (see A1.5.1), proceed as follows: after molding as in A1.5.5.3, remove the assembly from the press (take off the aluminum foil) and knock the molded sheet out of the chase. Discard the flash from this sheet. Cut the sheet into at least four pieces of approximately equal size. Replace the pieces within the chase and between aluminum foil parting sheets. The pieces shall not be spread over the open volume of the mold but should be stacked so the press can squeeze out any air. Add enough milled crepe to make up for the flash lost in the first molding. Put smooth backing plates behind the aluminum parting sheets and repeat the molding procedure specified in A1.5.5.3.

A1.5.6 *For Procedure C*:

A1.5.6.1 Compression molding is performed using parting sheets of uncoated polyester film or aluminum foil. Any wrinkles in the parting sheets should be smoothed out before use. While foil is usually not suitable for re-use, it may be used again if undamaged.

A1.5.6.2 Place the polyethylene plastic in the chase between polyester film or aluminum foil backed by smooth backing plates. With the platens at the correct temperature insert the assembly between the platens. Allow a heating period of 5 min with the platens closed and no pressure applied, but with the platens in contact with the mold assembly. Then bring to full pressure as quickly as possible. After full pressure has been applied, continue the heating from 3 to 5 min. At the end of this heating period, turn off the heat and cool the press platens at a rate of 15 ± 2°C/min until a platen temperature of 76°C is reached for Class 1 and 2 (Type I and II) polyethylene plastics and 95°C for Class 3 and 4 (Type III and IV) and 45°C for Class 5 (Type 0). Continue to cool the platens until they are warm to the touch. Then remove the assembly from the press (Notes A1.9 and A1.10).

NOTE A1.9—The platen cooling rate can be varied by controlling the flow of water through the platens. The flow of water can be controlled by the use of needle valves on the coolant inlet lines or in the coolant exit lines.

A1.5.6.3 The cooling rate of the platens may be determined by inserting a thermometer (the bulb wrapped with metallic wool to ensure contact) or a thermocouple into a hole drilled into the platen and measuring the temperature change of each platen during the cooling cycle. Record the temperature each 30 s in the following temperature ranges:

| | |
|---|---|
| Class I (Type I) | 150°C to 76°C |
| Class 2 (Type II) | 177°C to 76°C |
| Class 3 (Type III) | 177°C to 76°C |
| Class 4 (Type IV) | 177°C to 76°C |
| Class 5 (Type 0) | 140°C to 45°C |

On linear graph paper plot temperature on the Y-axis versus time on the X-axis for the following temperature ranges:

| | |
|---|---|
| Class I (Type I) | 121°C to 76°C |
| Class 2 (Type II) | 150°C to 90°C |
| Class 3 (Type III) | 150°C to 90°C |
| Class 4 (Type IV) | 150°C to 90°C |
| Class 5 (Type 0) | 121°C to 45°C |

For each 30 s interval, the cooling rate shall be 15°C ± 2°C/min.

A1.5.6.4 Uniformity of temperature over the surface of the platens may be checked conveniently by using small temperature probes to measure the surface temperature of the platens or the temperature of the plastic itself. With suitable platen design, temperature differences within a particular cavity or picture frame can be kept to within 2°C.

A1.5.6.5 If non-uniform temperature distribution is observed, it may be minimized by the use of plates made from high thermal conductivity materials such as aluminum. copper or bronze plates or polished steel, of 3.2 to 12.7 mm thick. On some presses, a region of the outside perimeter of the mold may not be usable due to poor design of cooling channels. In extreme cases, a better distribution of cooling water channels in the platen may be required.

NOTE A1.10—If regular tap water is used as the platen coolant. it will be necessary to flush the scale and rust from the platen channeling periodically. This can be done by circulating boiler treater solution through the platen channels. The frequency of scale cleaning can be reduced by using air to purge water from the platens prior to preheating.

A1.5.6.6 If a second pressing is desired (see A1.5.1) proceed as follows: after molding as in A1.5.6.2, remove the assembly from the press (take off the parting sheets) and knock the molded sheet out of the chase. Discard the flash from this sheet. Cut the sheet into at least four pieces of approximately equal size. Replace the pieces within the chase and between the parting sheets of uncoated polyester film, cellophane, or aluminum foil. The pieces shall not be spread over the open volume of the mold but should be stacked so the press can squeeze out any air. Add enough milled crepe to make up for the flash lost in the first molding. Put smooth backing plates behind the parting sheets and repeat the molding procedure specified in A1.5.6.2. Test values obtained from stress-crack tests will be at a substantially different level than samples prepared by Procedures A or B.

NOTE A1.11—Available data indicate that the reproducibility of stress-crack tests on specimens from test sheets, prepared using Procedure C, may be variable. Results on specimens from test sheets, prepared by one of the other procedures may be less variable.

A1.6 *Conditioning (for Procedures A and B):*

A1.6.1 *General*—Place the unit produced in accordance with A1.5.6.2 on a smooth thin metal plate on a rack in the oven. The oven temperature shall be not less than 140°C for Class 1 (Type I). 150°C for Class 2 (Type II), 155°C for

[10] Available from Superintendent of Documents, U.S. Government Printing Office, North Capital and "H" Sts., NW Washington, DC 20401.

Classes 3 and 4 (Type III and IV), nor 140°C for Class 5 (Type 0) polyethylene plastics. In no case shall the temperature be greater than 175°C. Units intended for cooling by Procedure B should not be stacked. Units for Procedure A may be stacked, with a light, smooth metal plate between each pair (Note A1.13).

NOTE A1.12—Units shall reach the specified temperature and be maintained in the oven at the specified temperature for at least 1 h, and thereafter shall be cooled according to Procedure A or B.

NOTE A1.13—Care should be taken that all the units stacked for eventual cooling by Procedure A reach the oven temperature before cooling is started. The ability of the oven to heat all units to the required temperature in 1 h should be checked with suitably placed thermocouples. The use of low pressures during the conditioning procedure may assist in preventing the formation of bubbles and voids in black or other samples which show this tendency. Low pressures can be applied by the use of a metal following plate which rests on the sample and not on the metal chase.

A1.6.2 *Procedure A*—Leave the unit or units in the oven and switch on the thermo-regulating device to cool the oven at a rate of 5°C/h. Continue cooling at the specified rate and remove the samples at a temperature of 50°C or less.

NOTE A1.14—Most properties of polyethylene plastics are quite stable after conditioning by Procedure A. However, the use of Procedure A may not be suitable for preparation of test sheets for the mechanical tests of materials that are embrittled excessively by the slow cooling rate.

A1.6.3 *Procedure B*—The temperature of the water in the tank shall be 15 to 20°C. Running cold tap water or stirred, refrigerated water should be used (Note A1.15). After the unit at its specified temperature has been in the oven for 1 h remove it from the oven, place it in the wire tray, and lower the whole unit quickly into the cooling tank. The entire operation should be completed as quickly as possible. For this reason, the cooling tank should be located near the oven. The unit should be in the cold water within 10 to 15 s after its removal from the oven. Only one unit at a time should be in the cooling tank unless it has been shown that the presence of more than one does not affect the results of subsequent tests. Units shall remain immersed in the tank for at least 15 min. At the end of the cooling period remove the unit, strip off the aluminum foil, and press the test sheet out of the chase.

NOTE A1.15—The effect of a variation in the water temperature in the cooling bath depends on the material and the property being measured. For example, a change from 1 to 20°C has a noticeable effect on the density of almost all materials, whereas brittleness temperatures of most resins would be unaffected. For this reason, Procedure B is strictly confined to quenching in 15 to 20°C water.

NOTE A1.16—Procedure B is not recommended for density measurements that are to be compared between laboratories, because this property changes too rapidly with elapsed time after conditioning. Some of the other properties of interest do not appear to change as rapidly as does density, and Procedure B has been found useful for preparing test sheets for such tests as brittleness temperature and environmental stress cracking. The stability of tensile and stiffness properties will depend on the polyethylene plastic.

A1.7 *Test Specimen Preparation:*

A1.7.1 Prepare test specimens from test sheets by any technique which will not disturb the thermal history introduced during sheet preparation and which will provide test specimens of acceptable quality as judged by visual examination as described in A1.7.2. Where possible, use one of the five techniques described in Annex A2-Annex A7.

A1.7.2 Inspect prepared test specimens visually and use the following criteria for acceptability:

A1.7.3 *Cut*—Examine the freshly cut edges of the specimen. These should be smooth, substantially unbeveled. and free of feathery edges. For Class 3 and 4 (Type III and IV) materials, some whitening is not unusual and should not be considered a defect. If edge defects are seen, sharpening of the blanking die or of the machining tool is indicated.

A1.7.4 *Surface*:

A1.7.4.1 Examine the top and bottom flat surfaces of the specimen adjacent to cut edges for evidence of cracking. No cracks should be visible. If cracks are seen. use of another specimen preparation technique is indicated.

A1.7.4.2 Examine the top and bottom fiat surfaces of the specimen for evidence of scratching or marring. Surfaces should be generally blemish-free. Blemishes can arise from such things as blemish-transfer from the cover sheet when a blanking die is used, careless or improper technique in removing the specimen from the blanking die, or careless handling in the machining operation. When such blemishes are seen, immediate steps should be taken to correct the situation.

A1.7.5 *Form*—Examine the specimen for evidences of bending or warping. None should be seen. Such deformations can arise through poor technique in removing specimens from blanking dies.

A1.8 *Report:*[11]

A1.8.1 Report the following information:

A1.8.2 Identification of the material,

A1.8.3 Date of preparation of test sheets and test specimens,

A1.8.4 Procedure used to prepare test sheets (Procedure A, B, or C),

A1.8.5 Technique used to prepare test specimens (Technique 1, 2, 3, 4, or 5 of the Annexes; or details of the particular technique employed), and

A1.8.6 Deviations, if any, from the standard conditioning of Practice D 618, Procedure A.

[11] Round-robin data for this practice may be obtained from ASTM Headquarters. Request RR: D20-1047.

## A2. SPECIMEN PREPARATION FOR POLYETHYLENE

A2.1 Five techniques are described for preparing test specimens from test sheets. These are designated 1 through 5 and are generally in the order of choice for use as the task of preparing acceptable test specimens becomes progressively more difficult.

A2.1.1 *Technique 1* employs a blanking die and impact curing of the sheet at 23°C.

A2.1.2 *Technique 2* employs a blanking die and relatively slow curing of the sheet at 23°C by means of an arbor press or other device providing suitable mechanical advantage.

A2.1.3 *Technique 3* employs a blanking die and impact curing of the heated sheet.

A2.1.4 *Technique 4* employs a blanking die and curing of the heated sheet by use of an arbor press, or equivalent.

A2.1.5 *Technique 5* involves machining operations at 23°C.

A2.2 The necessity for having more than one technique comes from the fact that problems sometimes arise in attempting to prepare acceptable specimens from sheets which exhibit either some degree of brittleness or exceptional toughness. As a general rule, the simplest and most direct approach is preferable. Technique 1 or 2 would be used where possible, Technique 3 or 4 (heating of the sheet prior to cutting) would be used only when 1 or 2, or both, did not produce acceptable specimens, and Technique 5 would be used only if none of the other four produced satisfactory results.

A2.2.1 Techniques 1, 2, and 5 are applicable for preparing specimens from sheets prepared by any one of the three Procedures, A or B or C. However, precautions shall be taken when applying Techniques 3 and 4.

A2.2.1.1 Techniques 3 and 4 are applicable for sheets prepared by Procedure A, since the limited heating cycles for sheets do not change the thermal history introduced by Procedure A significantly.

A2.2.1.2 Techniques 3 and 4 are not applicable for sheets prepared by Procedure B, since the heating treatment would invalidate the thermal history introduced by Procedure B.

A2.2.1.3 Techniques 3 and 4 are applicable for sheets prepared by Procedure C provided it can be demonstrated that the properties of interest have not been altered significantly by the sheet-heating process.

A2.2.2 For Class 1, 2, and 5 (Type I, II, and 0) polyethylene plastics, all of the techniques will ordinarily produce acceptable specimens, so Technique 1 or 2 would normally be used.

A2.2.3 For Class 3 and 4 (Type III and IV) polyethylene plastics, wherein brittle or exceptionally tough sheets are encountered more often, some amount of trial and error may be required before an acceptable technique is found. Technique 1 or 2 would be the first choice, Technique 3 or 4 (subject to the limitations described in A2.2.1.1-A2.2.1.3 above) the second choice, and Technique 5 the last choice.

**TABLE A2.1 Heating Schedules for Sheets**[A]

| Class (Type) Polyethylene Plastic | Temperature | Heating Time for Oven Type | |
|---|---|---|---|
| | | Gravity | Recirculating |
| 1, 2, and 5 (I, II, and 0) | 76°C | 10 ± 2 min | 5 ± 1 min |
| 3 and 4 (III and IV) | 95°C | 10 ± 2 min | 5 ± 1 min |

[A] These heating schedules are for sheets no thicker than 3.2 mm (⅛ in.) nominal. Thicker sheets may require a longer time at the temperatures shown in order to produce acceptable specimens.

## A3. APPARATUS FOR SPECIMEN PREPARATION OF POLYETHYLENE

A3.1 *Blanking Dies*—Suitable blanking dies shall be used. The dimensions of each shall be as given in the applicable test method for the specimen to be prepared. The inside faces of each shall be polished and perpendicular to the plane formed by the cutting edges for a depth of at least 5 mm (0.2 in.).

A3.2 *Support*—For Techniques 1, 2, 3, and 4, the top surface of the support for the sheet should be smooth, planar, and solidly supported. A rectangular hardwood block about 75 mm (3 in.) thick is suitable.

A3.3 *Cover Sheet*—The top surface of the support should be provided with. a suitable cover sheet to protect it from physical damage during use. One suitable material is ultra-high molecular weight Class 3 or 4 (Type III or IV) polyethylene plastic sheeting nominally 3 mm (⅛ in.) thick. The cover sheet is laid on the support surface so that it may be moved about readily to provide an unmarred fresh surface for each cutting. Both sides of a cover sheet may be used and cover sheets are replaced as required.

A3.4 *Striker*—For Techniques 1 and 3, a striker is required to effect impact cutting in a manual operation. A heavy mallet or maul with a rawhide head is suitable.

A3.5 *Arbor Press*—For Techniques 2 and 4, an arbor press or equivalent, either manually operated or hydraulically driven, is required to produce downward force sufficient to cut specimens from sheets. Commercial devices[12] are available for this purpose, complete with interchangeable dies and provisions for automatic knockout of specimens.

A3.6 *Oven*—For Techniques 3 and 4, a constant temperature oven, either gravity convection or mechanical recirculating, is required for the heating of sheets.

A3.7 *Machining Equipment*—For Technique 5, a suitable cutter (hacksaw, bandsaw, or the like) and suitable machining equipment (a router and suitable templates afford a simple approach) are needed.

[12] One such manual device is the PUNCH-PRESS, NAEF, Model B, from MS Instrument Co., Castleton-on-Hudson, NY 12033.

## A4. MAINTENANCE AND PREPARATION OF EQUIPMENT FOR POLYETHYLENE

A4.1 The cutting edges of the blanking dies shall be sharp and free of nicks in order to prevent ragged edges on the specimen. Careful maintenance of die cutting edges is of extreme importance and can be obtained by light daily honing and touching up with a jeweler's hard Arkansas honing stone, or equivalent. Resharpening the die cutting edges, to remove nicks should be done by a qualified machinist.

A4.2 For Techniques 2 and 4, set the stop on the arbor press in a position so that specimens will be cut out cleanly but that minimum penetration into the cover sheet will occur to lengthen the life of the cover sheet.

A4.3 For Techniques 3 and 4, set the air oven to control at either 76 ± 2°C, for Class 1, 2, and 5 (Type I, II, and 0), or at 95 ± 2°C for Class 3 and 4 (Type III and IV) polyethylene plastics.

A4.4 For Technique 5, keep the cutting edges of the tool sharp.

## A5. SAMPLING FROM POLYETHYLENE SHEET

A5.1 When taking specimens from a sheet, avoid the 12-mm (½-in.) perimeter when property results might be affected. For example, this would apply to the entire density specimen and to the center section of the tensile specimen but not to the tab ends of the tensile specimen.

A5.2 Avoid minor bad areas (for example, voids, sinks, thick or thin areas) when taking specimens from a sheet. In extreme cases, it may be necessary to remold or to mold another sheet from the same material.

A5.3 Take specimens of the same type from different areas of the sheet to obtain an average property result representative of the sheet.

## A6. SPECIMEN PREPARATION AND CONDITIONING

A6.1 *Conditioning*—Condition the test specimens at 23 ± 2°C and 50 ± 5 % relative humidity for not less than 40 h prior to test in accordance with Procedure A of Practice D 618, for those tests where conditioning is required. In cases of disagreement, the tolerances shall be ± 1°C and ± 2 % relative humidity.

A6.2 For a sheet prepared by any one of the Procedures A, B, or C—prepare specimens within 8 h after sheet preparation.

NOTE A6.1—For special purposes, one may wish to obtain property data for specimens under nonstandard conditioning procedures. A non-standard conditioning procedure particularly recommended for sheets prepared by Procedure B is to prepare specimens within 1 h after sheet preparation and test the specimens within 4 h after sheet preparation.

## A7. PROCEDURES

A7.1 *Technique 1:*

A7.1.1 Place the test sheet on the cover sheet, hold the blanking die vertically in contact with the top surface over the selected area with one hand, then wield the striker manually so as to contact the top of the die handle with a force sufficient to cut out the specimen with one blow while penetrating a minimum depth into the cover sheet.

A7.1.2 Remove the specimen from the die, taking care to bend or twist the specimen as little as possible. Use of a suitable pushout tool, soft enough to avoid marring the surface of the specimen, will be helpful in this process.

A7.1.3 Repeat steps A7.1.1 and A7.1.2 to prepare additional specimens.

A7.1.4 Visually inspect the specimens to make sure they conform to the requirements of A1.7.2.

A7.2 *Technique 2:*

A7.2.1 Follow step A7.1.1 above, except, use an arbor press instead of a striker to achieve the necessary cutting force. Make the cut in one motion.

A7.2.2 Remove the specimen as in A7.1.2 (not applicable when a commercial device with automatic specimen knockout is employed).

A7.2.3 Repeat steps A7.2.1 and A7.2.2 to prepare additional specimens. Visually inspect the specimens to assess conformity to the requirements of A1.7.2.

A7.3 *Techniques 3 and 4:*

A7.3.1 Heat the test sheet in a flat position on a wire mesh support in an air oven according to the schedule given in Table A2.1.

A7.3.2 Remove the sheet and prepare specimens by Technique 1 or 2, cutting specimens as rapidly as possible.

A7.3.3 If necessary, repeat the heating-cutting steps above until the necessary number of specimens have been obtained.

A7.3.4 Visually inspect the specimens to assess conformity to the requirements of A1.7.2.

A7.4 *Technique 5:*

A7.4.1 By use of a hacksaw, bandsaw, or a similar instrument, cut the sheet into pieces whose sizes are appropriate for preparing specimens by machining.

A7.4.2 Using a router and suitable templates, or other suitable technique, prepare the necessary number of each specimen type required for subsequent testing.

A7.4.3 Visually inspect the specimens to assess conformity to the requirements of A1.7.2.

A7.5 *Dimensions:*

A7.5.1 All dimensions should be measured until the relationship between die dimensions and part dimension has been established. This relationship will change with different procedures and different materials, thus the part should be measured at 23°C prior to test.

## APPENDIX

### (Nonmandatory Information)

### X1. SUMMARIES OF PROCEDURES FOR CERTAIN THERMOPLASTIC MATERIALS

#### X1.1 Styrene-Butadiene Molding and Extrusion Materials

X1.1.1 *Apparatus*—Flash picture-frame type or flash cavity-type mold; hydraulic press with heating and cooling means in the press or in the mold.

X1.1.2 *Recommended Procedure*:

X1.1.2.1 Place a sheet of aluminum foil on the bottom ferrotype plate and place the mold chase on the foil sheet. This will help eliminate localized sink marks.

X1.1.2.2 Place a measured amount of granules in the mold, distributing it evenly in the cavity or cavities, and then cover the mold with another sheet of aluminum foil and the top ferrotype plate. Determine the approximate starting weight of the material to be used by multiplying the resin specific gravity by the mold volume (in cubic centimeters).

X1.1.2.3 Place the mold in a preheated press at 175 to 180°C (350°F) (see Note X1.1). Bring the platens into contact with the mold, maintaining the minimum observable ram force that will ensure contact between the platens and the mold.

NOTE X1.1—If acceptable plaques or specimens cannot be molded at this temperature, a suitable temperature should be used and should be reported.

X1.1.2.4 After 5 min, raise the ram force slowly to a value of approximately 7 MPa (1015 psi) over a period of approximately 1 min and maintain at this ram force for an additional 5 min.

X1.1.2.5 Turn off the heat and cool the press to 65°C (150°F) or lower at a reasonably uniform rate of approximately 10°C (18°C) per min, maintaining the ram force as indicated previously.

X1.1.2.6 Slow cooling is necessary to minimize orientation and eliminate bubbles and voids. Remove the excess material from around the mold before the temperature drops below 120°C (250°F).

X1.1.2.7 Remove the mold from the press. Remove the specimens or plaque from the mold and remove the flash, taking care to avoid contact with oils or other stress-cracking materials.

NOTE X1.2—Test specimens can be rough-cut from the plaque with a band saw and then machined with a milling cutter to standard dimensions.

#### X1.2 Rigid Poly(Vinyl Chloride) Compounds

NOTE X1.3—Chlorinated poly(vinyl chloride) (CPVC) compounds will likely require conditions and procedures different from those described below. Refer to the supplier's recommendations before molding such materials. CPVC compounds will require milling at temperatures of 180 to 230° C. Also, certain high heat distortion PVC compounds and those containing glass fibers are not amenable to compression molding and should be injection molded.

X1.2.1 *Apparatus*—Any size two-roll mill capable of maintaining a constant temperature of 180 ± 2°C (360 ± 3.6°F), a hydraulic press capable of maintaining a pressure of 7 MPa (1015 psi) on the material during the molding cycle, with platens having provision for both heating and cooling, a picture-frame compression molding chase having a blanked-out area of suitable size for producing plaques from which the required test specimens can be made, and two polished chromium-plated ferrotype plates at least 0.60 mm (0.024 in.) thick and of adequate surface area to cover the molding cavity.

X1.2.2 *Recommended Procedure*:

X1.2.2.1 *Milling*:

(*a*) Set the mill rolls at the temperature recommended by the compound supplier (see Note X1.3). Use a calibrated band pyrometer to verify the temperature. Set the clearance between the mill rolls to band the material as rapidly as possible. After banding, adjust the mill rolls to a clearance of 0.90 mm ± 0.13 mm (0.035 in. ± 0.005 in.). The batch size shall be sufficient to maintain a 10 to 20 mm (0.39 to 0.79 in.) rolling bank on the mill. The processing time on the mill will be recommended by the supplier. The supplier may recommend that the material be processed to a given stock temperature rather than specify a time. Measure this recommended stock temperature with a calibrated band pyrometer while the mill rolls are moving.

NOTE X1.4—This practice is applicable to compounds with a variety of optimum processing temperatures specific for each compound and depending upon the application for which the compound is to be used. The supplier shall specify the mill and mold temperatures and time cycle that will be satisfactory for the evaluation of the supplier's compound.

(*b*) Open the mill rolls to the sheet thickness desired. Determine the sheet thickness from swatches cut from both ends of the mill and measure with a sheet-thickness gage or a micrometer. Remove the entire batch from the mill and cool on a smooth surface under a suitable weight to maintain a flat sheet. A 6.4-mm (¼-in.) thick metal plate has been successfully used. It will be necessary to identify the mill direction of the sheet so that tensile, deflection and Izod test specimens can be prepared with the mill direction parallel to their long dimension.

(*c*) Cut the necessary number of sample sheets to be used for compression molding. Trim the rough edges of the milled batch before cutting. Do the cutting with a band saw shearing tool. The dimensions of the sheets shall be between 6 and 3 mm (0.24 and 0.12 in.) less than the dimensions of the mold. Set all sample sheets in the mold cavities with the mill direction parallel.

X1.2.2.2 *Molding*:

(*a*) Place the picture-frame chase plate on top of one of the clean ferrotype molding plates. Introduce into the molding cavity sufficient milled samples to fill the blanked-out area completely when molded. A slight excess of material is necessary. Place the other ferrotype plate on top of the molding-cavity (chase) plate. Insert this assembly between the platens of the molding pressure previously heated to the temperature recommended by the supplier. Preheat the material for such time as recommended by the supplier. This is done by bringing the platens into contact with the mold plates, with no pressure registered on the gage. After the preheat cycle, apply pressure to the mold at 7 MPa (1015 psi) for a period of time recommended by the supplier. At the end of the pressing cycle, turn off the heat and immediately turn on the full cooling system. Do not adjust the pressure on the mold during the cooling cycle. (The pressure will diminish during the cooling cycle.)

NOTE X1.5—Some compounds produce sink marks if cooled without maintaining pressure. If this is the case, it may be possible to avoid the sink marks by maintaining pressure during the cooling cycle. This is permissible, but if pressure is used on any of the samples, the same pressure cycle shall be maintained in cooling all samples of any given compound used for determining tensile strength or deflection temperature, or both.

(*b*) Remove the cooled samples from the press mold. Cut specimens from the cooled plaques in accordance with the dimensions given in the test methods.

### X1.3 Rigid Acrylonitrile-Butadiene-Styrene (ABS) Plastics

X1.3.1 *Apparatus*—A two-roll mill with chrome-plated rolls (a 305-mm (12-in.) mill with 152-mm (6-in.) diameter rolls, having a friction ratio of 1.4 to 1 and a slow roll speed of 25 r/min, has been found to be satisfactory), a flash picture-frame type or flash cavity-type mold (see Note X1.5), a hydraulic press capable of maintaining a pressure of 7 MPa (1015 psi) on the material during the molding cycle, with platens having provision for both heating and cooling (see Note X1.6), and sheets of aluminum foil, equal in size to the ferrotype plates of the mold and 0.1 mm (0.004 in.) thick.

NOTE X1.6—Recommended mold types include: (*1*) picture-frame compression mold, consisting of a chase having a blanked-out area of suitable size, capable of producing a plaque plus two polished chromium-plated ferrotype plates at least 1.0 mm (0.040 in.) thick with adequate surface area to cover the molding chase; or (*2*) a suitable flash cavity-type mold.

NOTE X1.7—Heating and Cooling Systems—Any convenient method of heating and cooling the press platens, molds, and mill rolls may be used, provided that the heat source is constant enough to maintain the temperatures within ±5°C (±9°F).

X1.3.2 *Recommended Procedure*:

X1.3.2.1 *Milling*:

(*a*) Set the roll clearance as low as possible. Pour material into the mill bite of a mill preheated to 165 to 170 °C (329 to 338°F) and spread evenly across the mill. Allow 60 s to warm the sample thoroughly, then start the mill and flux the material. Gradually open the mill roll bite until an even bank is obtained.

(*b*) Cut and blend the sample thoroughly to ensure complete fluxing and dispersion of the resin. This should be accomplished in 3 min.

(*c*) At the end of 3 min, adjust the roll bite to the desired thickness of sheet. The desired thickness will be that which is required to obtain the necessary molded sheets for tensile, impact, and other tests. Stop the mill and cut off the resin sheet. Make the cut as near to the bank as possible.

(*d*) Place the milled sheet on a clean, smooth, flat surface. Before the sample cools, cut the desired blanks for molding.

NOTE X1.8—For some ABS materials, mill direction may affect properties. This should be determined for each material and reported if it exists.

X1.3.2.2 *Molding*:

(*a*) ABS is a hygroscopic material and should be dried at a temperature of approximately 85°C for 2 h if the sample has been exposed to the air for more than a few minutes.

(*b*) Be sure that the mold is clean. Precondition the mold with a mold-release agent. Fluorocarbon release agents have been found satisfactory.

(*c*) Place a sheet of aluminum foil on the bottom ferrotype plate and place the mold chase on the foil sheet. This will help eliminate localized sink marks.

(*d*) Place a measured amount of granules or premilled stock in the mold, distributing it evenly in the cavity or cavities, and cover the mold with another sheet of aluminum foil and then the top ferrotype plate. Flash shall not exceed 0.075 mm (0.003 in.). Determine the approximate starting weight of material to be used by multiplying the resin specific gravity by the mold volume (in cubic centimeters).

(*e*) Place the mold in a press preheated to one of the following temperatures appropriate for the type of material being molded:

| | |
|---|---|
| FR grades | 200 ± 5°C |
| General grades | 220 ± 5°C |
| High heat grades | 240 ± 5°C |

(*f*) After 5 min, raise the ram force slowly to a value of approximately 4 ± 0.5 MPa (580 ± 73 psi) over a period of approximately 1 min and maintain at this ram force for an additional 5 min.

(*g*) Turn off the heat and cool the press to 65°C (149°F) or lower at a reasonably uniform rate of approximately 10°C (18°F) per min, maintaining the ram force as indicated previously. Slow cooling is necessary to minimize orientation and eliminate bubbles and voids. Remove the excess material from around the mold before the temperature drops below 120°C (250°F).

(*h*) Remove the mold from the press. Remove the plaque or specimens from the mold and remove the flash, taking great care not to disturb the molded surfaces. In handling the plaque or specimens, take care to avoid contact with oils or other stress-cracking materials.

## SUMMARY OF CHANGES

This section identifies the location of selected changes to this practice. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this practice. This section may also include descriptions of the changes or reasons for the changes, or both.

D 4703–00:

(*1*) Deleted paragraph about Test Method D 1928 (former 1.2).

(*2*) Modified ISO equivalency statement due to addition of Annex A1.

(*3*) Added new referenced documents.

(*4*) Added *picture frame mold* to definitions.

(*5*) Added "powder" to forms of materials that can be molded.

(*6*) Changed references to "steel" frames to "metal".

(*7*) Corrected Table 1 cooling rate ranges from + to ±.

(*8*) Added "polyethylene" to keywords.

(*9*) Added Annex A1 covering procedures for polyethylene and made editorial changes as needed.

(*10*) Deleted Appendix X1.4 (reference to Test Method D 1928, polyethylene molding procedure).

(*11*) Changed ABS molding conditions to conform to ISO ABS standard.