



# Standard Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets<sup>1</sup>

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 ( $\epsilon$ ) 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.<sup>2</sup>

1.2 Certain ASTM standards require the use of Test Method D 1928 for compression molding of polyethylene test specimens. Determine whether such requirements exist before using this practice.

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

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

1.5 *This standard does not purport to address all of the safety problems, 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:

D 1928 Test Method for Preparation of Compression-Molded Polyethylene Test Sheets and Test Specimens<sup>3</sup>

## 3. Terminology

### 3.1 Definitions of Terms Specific to This Standard:

3.1.1 *average cooling rate* ( $^{\circ}\text{C}/\text{min}$ ),  $n$ —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* ( $^{\circ}\text{C}/\text{h}$ ),  $n$ —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 *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 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 either from 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 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

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<sup>2</sup> This practice was created as a coalescence of and replacement for Practices D 2292, D 3010, and D 3463.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 08.02.

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.

**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

## 5. Apparatus

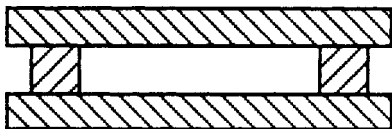
**5.1 Mill**—Any size two-roll mill having chrome-plated rolls, capable of maintaining a constant temperature within  $\pm 2^\circ\text{C}$  ( $\pm 3.6^\circ\text{F}$ ) of the temperature needed for the particular material involved, and being adjustable in speed as needed for the material to be worked,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

### 5.2 Molds:

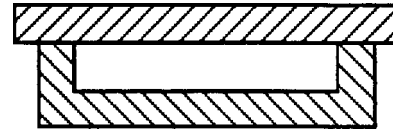
**5.2.1 Mold Types**—Several different types of molds may be used for the compression molding of test specimens of thermoplastics. In general, however, the molds used will fall into one of two categories: a *flash-type* mold (see Figs. 1 and 2) or a *positive-type* mold (see Fig. 3). The characteristics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

**5.2.2 Flash-Type Mold**—The flash-type mold may be of the *picture-frame* type, where a steel chase (the picture frame) is sandwiched between two thin steel ferrotyping 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 steel plate, with a single steel ferrotyping 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.

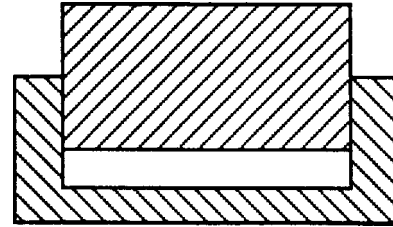
**5.2.3 Positive-Type Mold**—The positive-type mold consists of a cavity machined in a plate or block of steel and a force or plunger which closely mates with the sidewalls of the cavity (see Fig. 3). Like the flash-type mold, the cavity may be built to produce a plaque from which test specimens may be stamped or machined, or to mold a test specimen to finished



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



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



**FIG. 3 Positive-Type Mold**

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, neglected 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.<sup>4</sup> 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:

**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  $240^\circ\text{C}$ , 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  $\pm 5^\circ\text{C}$  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

<sup>4</sup> Mold comparison kits are available from D-M-E Company, 29111 Stephenson Highway, Madison Heights, MI 48071.

other for cooling unless it can be demonstrated that the press used for heating can cool at the specified rate.

NOTE 1—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  $24 \pm 1$  h at  $70 \pm 2^\circ\text{C}$  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.

### 6.2 Molding:

6.2.1 Adjust the mold temperature to within  $\pm 5^\circ\text{C}$  of the molding temperature indicated in the relevant material specification.

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. 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 and the temperature stability of the material must be considered.

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 2—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 3—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  $<40^\circ\text{C}$  if no other instructions are given.

NOTE 4—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; poly(vinyl chloride) (PVC); styrene-butadiene; test specimen preparation; thermoplastics

## 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 ferrotyping 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 ferrotyping 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, rise 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  $180 \pm 2^\circ\text{C}$  ( $360 \pm 3.6^\circ\text{F}$ ), 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 ferrotyping 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  $0.90 \text{ mm} \pm 0.13 \text{ mm}$  ( $0.035 \text{ in.} \pm 0.005 \text{ in.}$ ). The batch size must 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 must 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 (1/4-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 must 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  $\pm 5^{\circ}\text{C}$  ( $\pm 9^{\circ}\text{F}$ ).

#### 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 preheated press ( $205 \pm 30^{\circ}\text{C}$  for granules and  $177 \pm 3^{\circ}\text{C}$  for premilled stock). Bring the platens into contact with the mold, maintaining the minimum observable ram force that will ensure contact between the platens and the mold.

(f) 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.

(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.

## **X1.4 Polyethylene**

X1.4.1 *Method*—A separate standard (Practice D 1928) has been defined for use with polyethylene polymers.

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