Designation: D 5224 – 00

Standard Practice for Compression Molding Test Specimens of Thermosetting Molding Compounds[1]

This standard is issued under the fixed designation D 5224; 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 general principles to be followed when compression molding test specimens of thermosetting molding compounds, such as phenolics, aminoplastics, melamine phenolics, epoxies, and unsaturated polyesters.

NOTE 1—This standard is similar in content (but not technically equivalent) to ISO 295-1974 (E).

1.2 Molding conditions are given for amino, phenolic, and allyl molding compounds. Materials specification standards should always be consulted to determine whether the material to be molded has any special requirements.

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

2. Referenced Documents

2.1 *ASTM Standards:*

D 638 Test Method for Tensile Properties of Plastics[2]

D 883 Terminology Relating to Plastics[2]

D 958 Practice for Determining Temperatures of Standard ASTM Molds for Test Specimens of Plastics[3]

2.2 *ISO Standard:*

ISO 295 Plastics—Compression Molding Test Specimens of Thermosetting Materials[4]

ISO 3167 Plastics—Multipurpose—Test Specimens[4]

3. Terminology

3.1 *Definitions*—For definitions of terms pertaining to plastics used in this practice, see Terminology D 883.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *breathe step*, *n*—in plastics molding, the part of the molding cycle in which the mold halves are opened momentarily, prior to curing, to release volatiles from the molded part.

3.2.2 *skin*, *n*—in plastics molding, the thin resin-rich layer (skin) on the surface of the molded part.

3.2.3 *skin effect*, *n*—in plastics testing, the positive or negative effect the skin may have on the results of some standard tests.

4. Summary of Practice

4.1 Compression molded test specimens are produced by loading a mold cavity with some form of the molding material, applying a specified pressure to the mating surface for a specified time and at a specified temperature, and then removing the part from the cavity.

5. Significance and Use

5.1 The conditions at which compounds are molded are known to influence the properties of the specimens. The degree of cure, elimination of knit-lines between particles, density of the part, and degradation of the polymer are among those factors which will be affected by the molding conditions. Thus it is important to hold to a standard set of conditions in order to have a valid comparison of properties between different compounds and different batches of the same compound.

5.2 If the molded specimens show evidence of low-density areas due to trapped gases, they should be discarded. A breathe step may be necessary to eliminate this situation. It is critical that the breathe step be as brief as possible to avoid precuring of the compound before full pressure is applied. This would lead to poorly "knitted" areas and lower strength in the molded specimen.

6. Apparatus

6.1 *Molds*:

6.1.1 The mold shall be made of steel, able to withstand the molding temperatures and pressures. The mold shall be designed such that the compressive mold force is transferred to the molding material with no appreciable loss. The molds shown in Figs. 1 and 2 are recommended for maintaining the maximum force on the material. They are of the three-plate

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

[3] Discontinued 1995; see *1994 Annual Book of ASTM Standards, Vol 08.01*.

[4] *ISO Standards Handbook 21*, Vol 2, Plastics, 2nd Ed., 1990, available from The American National Standards Institute, 11 West 42nd St., 13th Floor, New York, NY 10036.

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

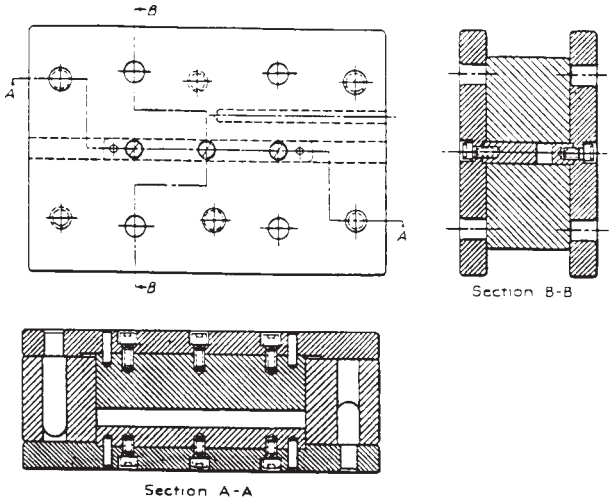

FIG. 1 Single-Cavity Positive-Compression Mold for Bar Test Specimens

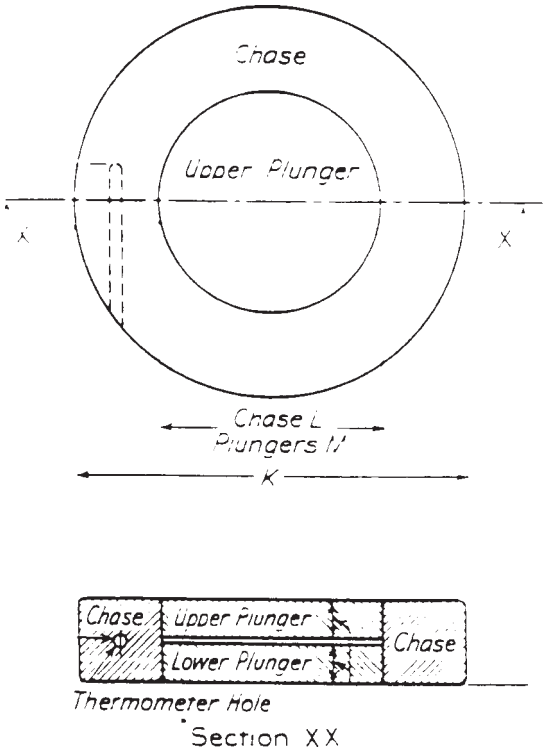

FIG. 2 Compression Mold for Disk Test Specimens

design; consisting of a shell or floating plate, with upper and lower compression plates. Molds may be of single or multiple cavity design.

NOTE 2—Semi-positive molds may be used, and for materials such as amino compounds, may even be preferred.

6.1.2 Although the actual mold cavity may have various forms, the majority of tests will use bars 12.7 mm (0.5 in.) in width by 127 mm (5 in.) or 64 mm (2.5 in.) in length, discs 51 mm (2 in.) or 102 mm (4 in.) in diameter or an appropriate tensile bar as described in Test Method D 638. The multi-purpose design from ISO 3167 may also be used. The mold shall be capable of molding thickness from 1.5 mm (0.06 in.) to 12.5 mm (0.5 in.). If the specimens are to be used for flame testing, even thinner specimens may be needed. In all cases the ASTM Standard Test Procedure to be used shall be consulted for the dimensions of the required test specimens.

NOTE 3—If at all possible, specimens shall be molded directly to dimension, rather than machined from a plaque. This maintains the integrity of any skin effect.

NOTE 4—If specimens are to be machined from plates or plaques, they should not be taken from the edge of the plaque. A minimum margin of 10 mm (0.5 in.) is recommended.

6.1.3 A cavity draft angle not exceeding 3° may be used to facilitate specimen removal.

6.1.4 The clearance between the vertical wall of the cavity and that of the force shall not exceed 0.1 mm (0.004 in.).

6.1.5 Mold surfaces should be finished to a roughness of 0.4 um to 0.8 um (SPI-SPE #2 or equivalent[5]), unless it is known that the particular test is not affected by a coarser surface finish. Chrome plating is recommended but not necessary. All cavity surfaces should be draw polished in the direction parallel to the force to facilitate specimen removal.

6.1.6 If ejector pins are used, they shall not deform the specimens and their placement shall be such that the pin marks are not in the area of test.

6.1.7 The mold shall have a loading chamber of sufficient volume to allow the introduction of the entire charge of material in a single loading. Preforms may be used to decrease the required loading volume of high bulk materials. The conditions of such preforming shall be included in the report.

6.1.8 As the specimen surface facing the lower die is heated for a longer time and at a higher temperature in the time interval between filling and compression, it is recommended that a mark be placed on one cavity face in such a position that it will not interfere with the testing. When reporting the results of tests that affect the surfaces unequally, the tested surface shall be indicated.

6.2 *Press*—The hydraulic press shall have a range of pressures sufficient to insure that the specified pressure is applied and maintained during the entire molding operation, and of maintaining that pressure within ±1.5 MPa (±218 psi).

6.2.1 In order to prevent precure, the press shall be capable of closing within 15 s after the placement of the material in the mold. A two-speed press is preferred for this purpose. The fast approach speed can be in the range of 200 to 400 mm/s (8 to 16 in./s) while the slow closing speed of 5 mm/s (0.2 in./s) is used to prevent gas entrapment.

6.3 *Heating System*—The molds may be heated by conduction from heated platens, heaters inserted into the mold itself, or by hot fluids circulated through passageways in the mold. The heating system shall be capable of controlling the mold temperature to ±3°C (±5°F) from point-to-point on the mold and for the duration of the molding time.

6.3.1 If the mold is heated directly, it shall be insulated from the press platens with a sheet of insulating material.

NOTE 5—It is generally preferable to heat the mold electrically.

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

6.4 *Temperature-Measurement System*:

6.4.1 *Mold Temperature*—A thermometer or pyrometer shall be used in accordance with Practice D 958.

6.4.2 *Preheated Compound Temperature*— If the compound is to be preheated to a definite temperature, a needle-probe pyrometer sensitive to ±2°C shall be used.

7. Conditioning

7.1 Except for referee testing and preparation of samples for electrical tests, prior conditioning of the material is not required.

7.2 *Referee Testing: Phenolic and Amino Molding Compounds*:

7.2.1 For the referee testing of all but electrical specimens, place a sufficient quantity of material in an open tray to a maximum depth of 13 mm (0.5 in.) for 72 h in a standard laboratory atmosphere (23 ± 1°C, 50 ± 2 % relative humidity).

7.2.2 For the preparation of electrical test specimens, place a sufficient sample of the molding material in an open tray to a maximum depth of 13 mm (0.5 in.). Dry in an air-circulating oven for 30 min at 90 ± 3°C. Mold the material immediately after conditioning.

7.3 *Referee Testing: Allyl Molding Compounds*:

7.3.1 For referee testing of all test specimens, place a sufficient quantity of material in an open tray to a depth of 25 mm (1 in.) for 72 h in a standard laboratory atmosphere (23 ± 1°C, 50 ± 2 % relative humidity).

7.4 *Electronic Preheating*:

7.4.1 When electronic preheating is to be used (refer to Molding Condition, Table 1), the objective is to attain a particular temperature as rapidly as possible. Determine the conditions necessary to attain that temperature with spare preforms which are then discarded. Do not use this type of preheating when preparing electrical test specimens. The material shall be transferred to the mold immediately after preheating to prevent cooling or premature curing.

7.5 *Preplastification*:

7.5.1 Preplastification is permissible to insure thermal and mechanical homogenization of the material. The preplasticized material shall be molded immediately after preplastification to prevent cooling or premature curing. Preplastification conditions shall be the subject of an agreement between the interested parties and shall be included in the molding report.

8. Procedure

8.1 Mold test specimens under the conditions listed in Table 1.

8.1.1 The "breathe step," if used, is done by opening the mold for a few seconds to release volatiles generated by the curing process. The mold opening should be slight and is done after it has already been closed at the molding temperature.

8.1.2 Release agents are not normally needed as molding compounds have internal lubricants to facilitate mold release. If release agents are used it shall be shown that they have no influence on the test specimen properties. They shall be noted in the report.

8.1.3 Cooling fixtures may be used to prevent warpage upon removal of the specimens from the mold.

8.2 *Phenolic Molding Compounds*:

8.2.1 Use conditions A or B from Table 1 for impact, flexure, tension, water absorption, heat deflection, heat-aging tests.

8.2.2 Use Condition C from Table 1 for electrical test specimens.

8.3 *Amino Molding Compounds*:

8.3.1 For urea-formaldehyde compounds use Condition D from Table 1.

8.3.2 For melamine-formaldehyde compounds use Condition E from Table 1.

8.4 *Allyl Molding Compounds*—Use conditions F, G, or H from Table 1.

8.5 *Molding Pressures*:

8.5.1 The molding pressures shown in Table 1 are recommended and shall be used for all referee molding and testing. If other pressures are to be used, the oil pressure p_0 in megapascals (MPa), to be displayed on the pressure gage in order to obtain the specified pressure p, in megapascals, is given by the equation:

$$p_0 = (p \times A_1)/A \quad (1)$$

where:

A = the area of the press piston head, m^2, and

TABLE 1 Molding Conditions for Compression Molding of Thermoset Molding Compounds

Molding Condition	A	B	C	D	E	F	G	H
Charge	powder	preform[A]	powder or preform[B]	powder, pill, or granular	powder or preform[B]	powder	powder	preform
Preheating	none	electronic	air oven		air oven	none	air oven	electronic
Preheat temperature		104 to 115°C	90°C		90°C		120°C	107°C
Preheat time			30 min		60 min		10 min	25 to 30 s
Molding temperature[C]	170°C	170°C	170°C	150°C	150°C	160°C	160°C	160°C
Molding pressure[D,E]	17 MPa[E]	17 MPa	17 MPa	28 MPa	28 MPa	20 MPa	20 MPa	20 MPa
Molding time								
3.2 mm thickness	5 min	3 min	5 min	4 min	5 min	5 min	3 min	3 min
6.4 mm thickness				5 min				
12.7 mm thickness				5 min				
Breathing	allowed	allowed	allowed	allowed	allowed	when necessary (keep brief)		

[A] A single preform, preferably approximating the molded shape, shall be used. If more than one preform is used, weld lines may affect the test value of the specimen.
[B] When using preforms, the material should be preheated in powder form, then preformed and molded immediately.
[C] Molding temperatures should be held to within 3°C of the specified level.
[D] Molding pressure should be held to within 2 MPa of the specified level.
[E] Highly plastic materials may flash excessively under the molding pressure specified. If that occurs, molding pressure may be reduced to a minimum of 10 MPa, in which case the pressure used shall be reported.

A_1 = the total area of the cavities, m^2.

9. Report

9.1 Report the following information:

9.1.1 Type and description of material used,

9.1.2 Description of the mold,

9.1.3 State of material molded (as received or with "referee test" conditioning),

9.1.4 Molding condition used (A, B, C, D, E, F, G, or H) and any variations from the listed condition, and

9.1.5 Whether or not a "breathe step" was used during molding.

9.1.6 The report form (Fig. 3) may be used and identifies all pertinent information.

10. Precision and Bias

10.1 Inasmuch as this practice does not generate a numerical result, a precision and bias estimate is not possible. However, the procedures used during molding of test specimens will affect the numerical results from tests done on the

Compression Molding Report			
Date:		Material:	
		Plasticity:	
Molded by:		Form:	
		Batch Number:	
Conditioning	Oven Preheating	Without	
		Time	
		Temperature	
	Preforming	Pressure	
		Temperature	
		Weight	
		Dimensions	
	High Frequency Preheating	Preheater Power	
		Time	
		Amperage	
		Number of Preforms	
		Preform Temperature	
	Preplasification	Barrel Temperature	
		Dynamic Pressure	
		Screw Speed	
		Material Temperature	
Compression Molding		Temperature	
		Pressure	
		Cure Time	
		Breathing	
Mold		Release Agents	
		Type	
		Number of Cavities	
		Chrome Plated	
		Heating Device	

Notes:

FIG. 3 Compression Molding Report

specimens. Thus variabilities in the molding procedure will contribute to variability in the final test results.

11. Keywords

11.1 allyl molding compound; amino molding compound; compression molding; phenolic molding compound; plastics; thermoset

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 5224 – 00:

(*1*) Added editorial changes for clarification; removed reference to discontinued standards.

(*2*) Added new terms under Terminology.

(*3*) Rewrote practice to move from a mold design format to a good compression molding practice format.

(*4*) Added preplastification as a means of preheating the material.

(*5*) Added mold temperatures to Conditions G, H, and I of Table 1.

(*6*) Added report format form (Fig. 3).