



Designation: ~~D4703-07~~ Designation: D4703 – 10

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

This standard is issued under the fixed designation D4703; 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 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 in accordance with ISO Cooling Method B may not be equivalent to Annex A1, Procedure C.

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>3</sup>

D618 Practice for Conditioning Plastics for Testing

D1248 Specification for Polyethylene Plastics Extrusion Materials for Wire and Cable

D3350 Specification for Polyethylene Plastics Pipe and Fittings Materials

D4976 Specification for Polyethylene Plastics Molding and Extrusion Materials

### 2.2 ISO Standard:

ISO 293-1986 Plastics—Compression Moulding Test Specimens of Thermoplastic Materials<sup>4</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 *picture frame mold*,  $n$ —a flat piece of metal, usually of brass or steel, that has a center portion removed to provide the

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.09 on Specimen Preparation. Current edition approved April 1, 2007-2010. Published April 2007-July 2010. Originally approved in 1991. Last previous edition approved in 2003-2007 as D4703 – 03. DOI: 10.1520/D4703-10.7.

<sup>2</sup> This practice was created as a coalescence of and replacement for Practices D1928, D2292, D3010, and D3463.

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>4</sup> ISO/IEC Selected Standards for Testing Plastics, available from ASTM. Also available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

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

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 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  $\pm 2^{\circ}\text{C}$  ( $\pm 3.6^{\circ}\text{F}$ ) 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.

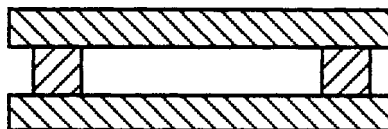
##### 5.2 *Molds*:

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, some mechanical properties may be affected by 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 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



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

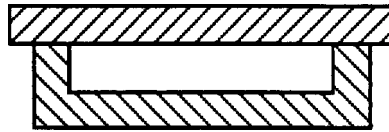


FIG. 2 Flash Mold with Machined Cavity

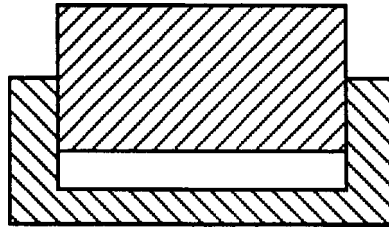


FIG. 3 Positive-Type Mold

degree required by the test for which the specimen is intended. For most tests, a surface quality of SPI-SPE #2 is adequate.<sup>5</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°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

<sup>5</sup> Mold comparison kits are available from D-M-E Company, 29111 Stephenson Highway, Madison Heights, MI 48071.

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

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. 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  $<40^{\circ}\text{C}$  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, and 4 polyethylene plastics (Specification D4976) and Types 0, I, II, III, and IV (Specifications D1248 and D3350). 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 D1928 - 96.

A1.1.1 *Procedure A*, in which the temperature of the initially molten plaque is lowered at a rate of  $5 \pm 0.5^\circ\text{C/h}$ .

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  $15 \pm 2^\circ\text{C/min}$ .

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 1 h at a temperature about  $25^\circ\text{C}$  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 D4976 (Type in D1248 and D3350), have been found to be generally useful:

Class 1 (Type I)	140°C
Class 2 (Type II)	150°C
Class 3 (Type III)	155°C
Class 4 (Type IV)	155°C
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 D1248, D3350 and D4976.

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  $150^\circ\text{C}$  for Class 1 (Type I) and  $177^\circ\text{C}$  for Class 2, 3 and 4 (Type II, III and IV), and  $140^\circ\text{C}$  for 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  $5.0 \pm 0.5^\circ\text{C}$  ( $9.0 \pm 0.9^\circ\text{F}$ )/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.<sup>6</sup> An alternative and equally satisfactory device comprises a controller and a small electric motor geared to lower the oven temperature at the specified rate.<sup>7</sup>

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  $15 \pm 2^\circ\text{C}/\text{min}$ .

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) and 177°C for Class 2, 3 and 4 (Type II, III and IV), and 140°C for 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 two and not more than 10 % of the weight of the final molding. For this purpose, the following densities may be assumed:

Class 1 (Type I)	0.92 g/cm <sup>3</sup>
Class 2 (Type II)	0.93 g/cm <sup>3</sup>
Class 3 (Type III)	0.95 g/cm <sup>3</sup>
Class 4 (Type IV)	0.97 g/cm <sup>3</sup>
Type 0	0.90 g/cm <sup>3</sup>

The densities of most carbon black formulations are about 0.01 g/cm<sup>3</sup> 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

<sup>6</sup> Detail drawings of a suitable device are available from ASTM Headquarters. Request Adjunct No. ADJD1928.

<sup>7</sup> 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.