



Designation: D 4894 – 98a

Standard Specification for Polytetrafluoroethylene (PTFE) Granular Molding and Ram Extrusion Materials¹

This standard is issued under the fixed designation D 4894; 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 specification covers granular resins and test methods for polytetrafluoroethylene (PTFE) that have never been preformed or molded and are normally processed by methods similar to those used in powder metallurgy or ceramics, or by special extrusion processes. These PTFE resins are homopolymers of tetrafluoroethylene, or, in some cases, modified homopolymers containing not more than one percent by weight of other fluoromonomers. The usual methods of processing thermoplastics generally are not applicable to these materials because of their viscoelastic properties at processing temperatures. The materials included herein do not include mixtures of PTFE resin with additives such as colorants, fillers or plasticizers; nor do they include reprocessed or reground resin or any fabricated articles. The methods and properties included are those required to identify the various types of resins. Additional procedures are provided in the Appendix for further characterization of the resins.

1.2 The values stated in SI units as detailed in IEEE/ASTM SI-10 are to be regarded as the standard, and the practices of IEEE/ASTM SI-10 are incorporated herein.

1.3 The following precautionary caveat pertains only to the Specimen Preparation section, Section 9, and the Test Methods section, Section 10, of this specification: *This specification 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.* See Notes 3 and 9 for specific cautionary statements.

NOTE 1—Information in this specification is technically equivalent to related information in ISO 12086-1 and ISO 12086-2.

2. Referenced Documents

2.1 ASTM Standards:

D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing²

¹ This specification is under the jurisdiction of Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.15 Thermoplastic Materials.

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² Annual Book of ASTM Standards, Vol 08.01.

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

D 792 Test Methods for Density Specific Gravity (Relative Density) and Density of Plastics by Displacement²

D 883 Terminology Relating to Plastics²

D 1505 Test Method for Density by the Density-Gradient Technique²

D 1895 Test Methods for Apparent Density, Bulk Factor, and Pourability of Plastic Materials²

D 3295 Specification for PTFE Tubing³

D 3892 Practice for Packaging/Packing of Plastics³

D 4441 Specification for Aqueous Dispersions of Polytetrafluoroethylene^{4,5}

D 4591 Test Method for Determining Temperatures and Heats of Transitions of Fluoropolymers by Differential Scanning Calorimetry⁴

D 4895 Specification for Polytetrafluoroethylene (PTFE) Resins Produced From Dispersion^{4,5}

E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁶

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods⁶

IEEE/ASTM SI 10-1997 Standard for Use of the International System of Units (SI): The Modern Metric System⁶

2.2 ISO Standards:

ISO 12086-1 Plastics—Fluoropolymer Dispersions and Moulding and Extrusion Materials—Part 1: Designation System and Basis for Specification⁷

ISO 12086-2 Test Methods for Fluoropolymers⁷

3. Terminology

3.1 Definitions:

3.1.1 The terminology given in Terminology D 883 is applicable to this specification.

³ Annual Book of ASTM Standards, Vol 08.02.

⁴ Annual Book of ASTM Standards, Vol 08.03.

⁵ Specifications for other forms of polytetrafluoroethylene may be found in Specifications D 4441 and D 4895.

⁶ Annual Book of ASTM Standards, Vol 14.02.

⁷ 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.

3.2 Descriptions of Terms Specific to This Standard:

3.2.1 *bulk density*—the mass (in grams) per litre of resin measured under the conditions of the test.

3.2.2 *extended specific gravity (ESG)*—the specific gravity of a specimen of PTFE material molded as described in this specification and sintered (g.v.) for an extended period of time, compared to the sintering time for the measurement of standard specific gravity (SSG), using the appropriate sintering schedule given in this specification.

3.2.3 *lot, n*—one production run or a uniform blend of two or more production runs.

3.2.4 *preforming*—compacting powdered PTFE material under pressure in a mold to produce a solid object, called a preform, that is capable of being handled. Molding and compaction are terms used interchangeably with preforming for PTFE.

3.2.5 *reground resin*—that produced by grinding PTFE material that has been preformed but has never been sintered.

3.2.6 *reprocessed resin*—that produced by grinding PTFE material that has been both preformed and sintered.

3.2.7 *sintering*—as it applies to PTFE, a thermal treatment during which the PTFE is melted and recrystallized by cooling with coalescence occurring during the treatment.

3.2.8 *skiving*—a machining operation during which a continuous film of PTFE material is peeled from the lateral surface of a cylindrical sintered molding.

3.2.9 *standard specific gravity (SSG)*—the specific gravity of a specimen of PTFE material molded as described in this specification and sintered using the appropriate sintering schedule given in this specification.

3.2.10 *thermal instability index (TII)*—a measure of the decrease in molecular weight of PTFE material which has been heated for a prolonged period of time.

4. Classification

4.1 This specification covers the following six types of PTFE generally used for compression molding or ram extrusion, or both:

4.1.1 *Type I*—Resin used for general-purpose molding and ram extrusion.

4.1.2 *Type II*—Finely divided resin with an average particle size less than 100 micrometers.

4.1.3 *Type III*—Modified resins, either finely divided or free-flowing, typically used in applications requiring improved resistance to creep and stress-relaxation in end-use.

4.1.4 *Type IV*—Free-flowing resins. Generally made by treatment of finely divided resin to produce free-flowing agglomerates.

4.1.5 *Type V*—Presintered. Resin that has been treated thermally at or above the melting point of the resin at atmospheric pressure without having been previously preformed.

4.1.6 *Type VI*—Resin, not presintered, but for ram extrusion only.

NOTE 2—See Tables 1 and 2 for division of Types by Grades, and footnotes to Tables 1 and 2 (and Table X2.1 in Appendix X2.) for former classifications.

4.2 A one-line system may be used to specify materials covered by this specification. The system uses predefined cells to refer to specific aspects of this specification, illustrated as follows:

Specification				
Standard Number Block	Type	Grade	Class	Special notes
Example: ASTM D 4894 – 97	III	2		

For this example, the line callout would be ASTM D 4894 – 97, III2, and would specify a granular polytetrafluoroethylene that has all of the properties listed for that Type and Grade in the appropriate specified properties, Tables, or both, in this specification. In this case there is no Class item so the cell position for class is left blank. A comma is used as the separator between Standard Number and Type. Separators are not needed between Type, Grade, and Class.⁸ Provision for Special Notes is included so that other information can be provided when required. An example would be in Specification D 3295 where dimensions and tolerances are specified for each AWG size within Type and Class. When Special Notes are used, they should be preceded by a comma.

5. Mechanical Properties

5.1 The resins covered by this specification shall conform to the requirements prescribed in Tables 1 and 2 when tested by the procedures specified herein. Table 1 lists tests to be carried out on resins. Table 2 lists tests requiring specimens molded as described in Section 3.1.1.

⁸ See the ASTM *Form and Style Manual*, available from ASTM Headquarters.

TABLE 1 Detail Requirements for Tests on Resins^A

Type	Grade	Bulk Density, g/L	Particle Size, Average Diameter, μm	Water Content, max, %	Melting Peak Temperature	
					Initial °C	Second °C
I	1	700 ± 100	500 ± 150	0.04	A	327 ± 10
	2	675 ± 50	375 ± 75	0.04	A	327 ± 10
II	<100	0.04	A	327 ± 10
	1	400 ± 125	<100	0.04	A	327 ± 10
III	2	850 ± 50	500 ± 100	0.04	335	327 ± 5
	1	650 ± 150	550 ± 225	0.04	A	327 ± 10
IV	2	>800	...	0.04	A	327 ± 10
	3	580 ± 80	200 ± 75	0.04	A	327 ± 10
V	...	635 ± 100	500 ± 250	0.04	327 ± 10	327 ± 10
VI	...	650 ± 150	800 ± 250	0.04	335	327 ± 5

^A >5°C above the second melting peak temperature.

TABLE 2 Detail Requirements for Tests on Molded Specimens

Type	Grade	Thermal Instability Index, max	Standard Specific Gravity		Tensile Strength min		Elongation at break min %
			min	max	MPa	psi	
I	1	50	2.13	2.18	13.8	2000	140
	2	50	2.13	2.18	17.2	2500	200
II	...	50	2.13	2.19	27.6	4000	300
III	1	50	2.14	2.22	28.0	4060	500
	2	50	2.14	2.18	20.7	3000	300
IV	1	50	2.13	2.19	25.5	3700	275
	2	50	2.13	2.19	27.6	4000	300
	3	50	2.15	2.18	27.6	4000	200
V	...	NA ^A	NA ^A	NA ^A	NA ^A	NA ^A	NA ^A
VI ^B	...	NA ^A	NA ^A	NA ^A	NA ^A	NA ^A	NA ^A

^A NA: Not Applicable by molding techniques included in this specification.
^B Extrusions of this resin may show an increased degree of clarity.

6. Other Requirements

6.1 The resin shall be uniform and shall contain no additives or foreign material.
 6.2 The color of the material as shipped by the seller shall be white.

7. Sampling

7.1 The resin shall be sampled in accordance with an adequate statistical sampling program. Adequate statistical sampling prior to packaging shall be considered an acceptable alternative.
 7.2 The producer shall take (and test) sufficient within-lot samples to assure adequate in-process quality control and continuing conformance to the property requirements of this specification.

8. Number of Tests

8.1 Lot inspection shall include tests for bulk density, particle size and standard specific gravity. Periodic tests shall consist of all the tests specified in Tables 1 and 2 and shall be made at least one per year.

8.2 The tests listed in Tables 1 and 2, as they apply, are sufficient to establish conformity of a material to this specification. One set of tests specimens as prescribed in Section 3 shall be considered sufficient for testing each sample. The average of the results for the specimens tested shall conform to the requirements of this specification.

9. Specimen Preparation

9.1 Test Disks:

9.1.1 Use the die shown in Fig. 1 for the molding of test disks (Note 3). The test resin should be near ambient temperature prior to molding (Note 6).

NOTE 3—**Caution:** PTFE can evolve small quantities of gaseous products when heated above 204°C (400°F). Some of these gases are harmful. Consequently, exhaust ventilation must be used whenever the resins are heated above this temperature, as they are during the sintering operations that are a part of this specification. Since the temperature of burning tobacco exceeds 204°C (400°F), those working with PTFE resins should ensure that tobacco is not contaminated.

9.1.2 Screen 14.5 g (for tensile properties) or 7.25 g (for electrical properties discussed in Appendix X1.7) of PTFE resin through a No. 10 hand sieve into the die. Adjust the lower

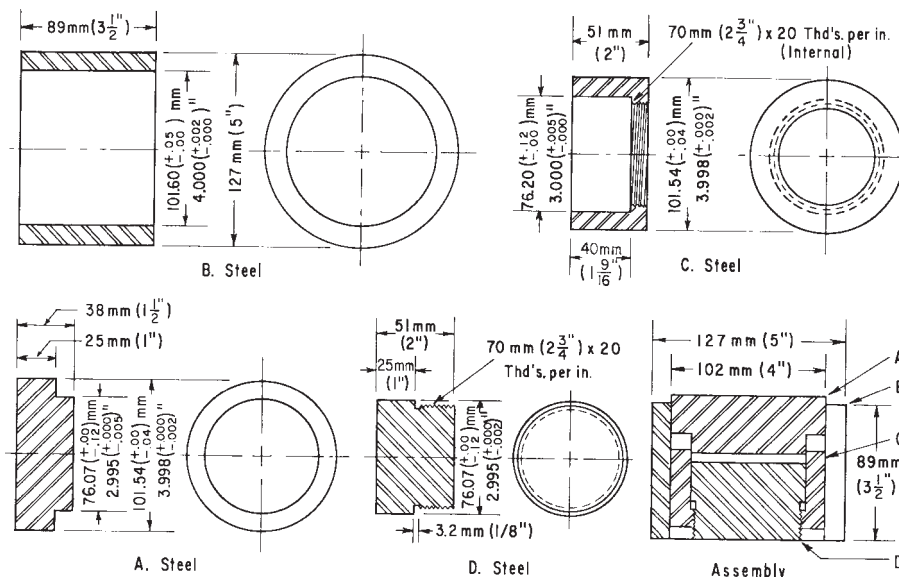


FIG. 1 Assembly and Details of Die for Molding Test Specimens

plug height so that the resin in the die can be leveled by drawing a straightedge in contact with the top of the die across the top of the die cavity. Insert the die in a suitable hydraulic press and apply pressure gradually (Note 4) until a total of 34.5 MPa (5000 psi) is attained. Hold this pressure for 3 min. Remove the disk identification on the disk at this time.

NOTE 4—As a guide, increasing the pressure at a rate of 3.45 MPa (500 psi)/min is suggested until the desired maximum pressure is attained.

9.1.3 Sinter the preforms according to Table 3 (Note 5).

9.1.3.1 Use Procedure B for Types I, II and IV and Procedure C for Type III.

NOTE 5—Although the rate of heating application is not critical, the cooling cycle is most important and the conditions cited in these procedures must be followed very closely. If they are not followed, the crystallinity of the disks and the resulting physical properties may be markedly changed. Therefore, the use of a programmed oven is recommended for the most precise sintering cycle control so that the hood window may be left down during the entire sintering procedure, the latter being an important safety consideration.

9.2 Test Specimens for Standard Specific Gravity (SSG) and Extended Specific Gravity (ESG):

9.2.1 A cylindrical preforming die, 28.6 mm (1 1/8 in.) internal diameter by at least 76.2 mm (3 in.) deep, is used to prepare the preforms. End plug clearances should be sufficient to ensure escape of air during pressing. The test resin should be near ambient temperature prior to molding (Note 6).

NOTE 6—For maximum precision, the weighing and preforming operations should be carried out at $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) (the “near ambient” temperature referred to herein). These operations should not be performed at temperatures below about 21°C (70°F) due to the crystalline transition that occurs in PTFE in this temperature region which leads to possible cracks in sintered specimens and differences in specimen density (as well as changes in other physical properties). Problems caused by the effects of temperature on the specific gravity or density of PTFE can be minimized when the measurement is made using immersion procedures if a sensitive thermometer (for example, one reading $\pm 0.1^\circ\text{C}$) is used in the liquid and the temperature is adjusted to be at least 22°C .

9.2.2 Weigh out 12.0 ± 0.1 g of resin and place it in the die. Screen non-free-flowing resins through a No. 10 sieve. Compacted resins can be broken up by hand-shaking cold resin in a half-filled sealed glass container. Condition the resin in the sealed glass container in a freezer or dry-ice chest. After breaking up resin lumps, allow the sealed container to equilibrate to near ambient temperature. Then screen and weigh the

12.0 ± 0.1 -g sample. Insert the die in a suitable hydraulic press and apply pressure gradually (Note 4) until a pressure of 34.5 MPa (5000 psi) is attained. Hold this pressure for 2 min. Remove the preform from the die. A wax pencil may be used to write the sample identification on the preform at this time.

9.2.3 Sinter the preforms according to Table 3 (Note 5).

9.2.3.1 For SSG specimens use Procedure B for Types I, II and IV and Procedure C for Type III.

9.2.3.2 For ESG specimens use Procedure F for Types I, II and IV and Procedure G for Type III.

NOTE 7—Improved precision in SSG and ESG test results has been obtained with the use of an upright, cylindrical oven and an aluminum sintering rack. The cylindrical oven has an inside diameter of 140 mm (5.5 in.) and an inside depth of 203 mm (8 in.) plus additional depth to accommodate a 50.8-mm (2-in.) thick cover, and is equipped with suitable heaters and controllers to sinter specimens according to the Procedures in Table 3. The rack, as shown in Fig. 2, allows preforms to be placed symmetrically in the center region of the oven. Place six preforms on each of the middle oven rack shelves (if six or fewer preforms are to be sintered, place them on the middle rack, filling in with “dummies” as needed). Place “dummies” on the top and bottom shelves. Specimens must be spaced evenly in a circle on each shelf, with none of them touching. An oven load must be no less than 18 pieces including “dummies.” “Dummies” are defined as normal 12-g specimens that have previously been through the sintering cycle. “Dummies” must only be used for an additional two or three thermal cycles, due to eventual loss of thermal stability and physical form.

9.2.4 Remove all flash from each specimen so that no air bubbles will cling to the edges when the specimen is immersed in the solution for weighing during the standard specific gravity and thermal instability index tests. It is recommended for this section and during testing that cotton gloves be worn while handling test specimens.

9.3 Test Billets:

9.3.1 Test specimens cut or skived from billets may be used as alternatives to the test disks described in 9.1 and 9.2 for Types I, II, III and IV resins.

9.3.2 Mold test billets in a mold similar to Fig. 3, having an inside diameter of 57 mm (2.25 in.) and of sufficient height to contain the resin sample. Plug clearance should be sufficient to ensure escape of air during pressing. A 254-mm (10-in.) mold cavity fill depth will produce a billet approximately 76 mm (3 in.) long from a resin charge of 400 ± 50 g. The billet length

TABLE 3 Sintering Procedures

	B	C	D	E	F	G	H	I
Initial temperature, °C (°F)	290 (554)	290 (554)	238 (460)	238 (460)	290 (554)	238 (460)	238 (460)	238 (460)
Rate of heating, °C/h (°F/h)	120 ± 10 (216 ± 18)	120 ± 10 (216 ± 18)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 9)	120 ± 10 (216 ± 18)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 9)
Hold temperature, °C (°F)	380 ± 6 (716 ± 10)	357 ± 8 (675 ± 15)	371 ± 6 (700 ± 10)	360 ± 6 (685 ± 10)	380 ± 6 (716 ± 10)	357 ± 8 (675 ± 5)	380 ± 6 (716 ± 10)	371 ± 6 (700 ± 10)
Hold time, min	30 + 2, -0	30 + 2, -0	240 ± 15	240 ± 15	360 ± 5	240 ± 15	960 ± 15	120 ± 5
Cooling rate, °C/h (°F/h)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 9)
Final or second hold temperature, °C (°F)	294 ± 6 (561 ± 10)	294 ± 6 (561 ± 10)	238 ± 6 (460 ± 10)	238 ± 6 (460 ± 10)	294 ± 6 (561 ± 10)	238 ± 6 (460 ± 10)	238 ± 6 (460 ± 10)	238 ± 6 (460 ± 10)
Second hold time, min	24 + 0.5, -0	24 + 0.5, -0	NA ^A	NA ^A	24 + 0.5, -0	NA ^A	NA ^A	NA ^A
Period to room temperature, min, h	1/2	1/2	6	6	1/2	6	6	6

^A NA, Not applicable.

9.4 Conditioning Test Specimens:

9.4.1 For tests of tensile properties and all tests requiring the measurement of specific gravities condition the test specimens in general accordance with Procedure A of Practice D 618, with the following deviations therefrom: a) the aging period shall be a minimum of 4 h immediately prior to testing, b) the laboratory temperature shall be $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$), and c) there shall be no requirement respecting humidity. The other tests require no conditioning of the molded test specimens.

9.5 Test Conditions:

9.5.1 Tests shall be conducted at the standard laboratory temperature of $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$). See Note 6 for additional details. Since these resins do not absorb water, the maintenance of constant humidity during testing is not required.

10. Test Methods

10.1 Melting Characteristics by Thermal Analysis:

10.1.1 Significance and Use—Most of the PTFE resins that fall within the scope of this specification have never been melted (the only exception is Type V resin). These resins have higher melting peak temperatures on initial melting than on second or subsequent meltings. Since PTFE resins that have been melted prior to use behave differently from those that have not, the melting characteristics of resins provide important distinctions among them. Melting peak temperatures (see Fig. 5) are used to make these distinctions, and determine conformance of a resin to the melting peak temperature requirements given in Table 1 of this specification. A resin that has been melted is not compatible with this specification, except for Type V.

10.1.2 Apparatus—Use apparatus described in Test Method D 4591.

10.1.3 Procedure:

10.1.3.1 Measure melting peak temperatures according to the procedures given in Method D 4591. An initial melting peak temperature above the melting peak temperature obtained on the second and subsequent melting (defined as the second

melting peak temperature) indicates that the resin was not melted before the test. The second melting peak temperature occurs at about 327°C (621°F). Usually the difference between the initial and second melting peak temperatures is greater than 5°C (9°F), but may be slightly less, as can be seen in Table 1. If peak temperatures are difficult to discern from the curves, that is, if the peaks are rounded rather than pointed, straight lines should be drawn tangent to the sides of the peak. Where these lines intersect beyond the peak should be taken as the peak temperature. Multiple peaks during the initial melting test may occur. Where more than one peak occurs during the initial melting test, the presence of any peak corresponding to the second melting peak temperature may indicate the presence of some previously melted material.

10.2 Bulk Density:

10.2.1 Significance and Use—Bulk density gives some indication of how a resin may perform during feeding of molding and ram extrusion equipment. PTFE resins have a tendency to compact during shipment and storage, and even though the material is broken up by screening or some other means, original “as produced” results may not be duplicated. Because of this tendency to pack under small amounts of compression or shear, Test Method D 1895 is not applicable to these resins. The procedure given in the following paragraphs must be used to measure this property.

10.2.2 Apparatus:

10.2.2.1 Funnel—A funnel arrangement as shown in Fig. 6.

10.2.2.2 Feeder⁹—A feeder with a No. 8 wire screen placed over approximately the top two-thirds of the trough. The funnel shall be mounted permanently in the feeder outlet.

10.2.2.3 Controller¹⁰

10.2.2.4 Volumetric Cup and Cup Stand (Fig. 7)—The volumetric cup shall be calibrated initially to 250 mL by filling it with distilled water, placing a planar glass plate on top, drying the outside of the cup, and weighing. The net weight shall be 250 ± 0.5 g. The top and bottom faces of the volumetric cup and the cup stand shall be machined plane and parallel.

10.2.2.5 Leveling Device—The leveler (Fig. 8) shall be affixed permanently to the table and adjusted so that the sawtooth edge of the leveler blade passes within 0.8 mm ($1/32$ in.) of the top of the volumetric cup.

10.2.2.6 Work Surface—The work surface for holding the volumetric cup and leveler shall be essentially free from vibration. The feeder, therefore, must be mounted on an adjoining table or wall bracket.

10.2.2.7 Balance—The balance having an extended beam shall have a capacity of 500 g and a sensitivity of 0.1 g or equivalent.

10.2.3 Procedure:

10.2.3.1 Place the clean, dry volumetric cup on the extended beam of the balance and adjust the tare to zero. Select about 500 mL of the resin to be tested, place it on the feeder screen

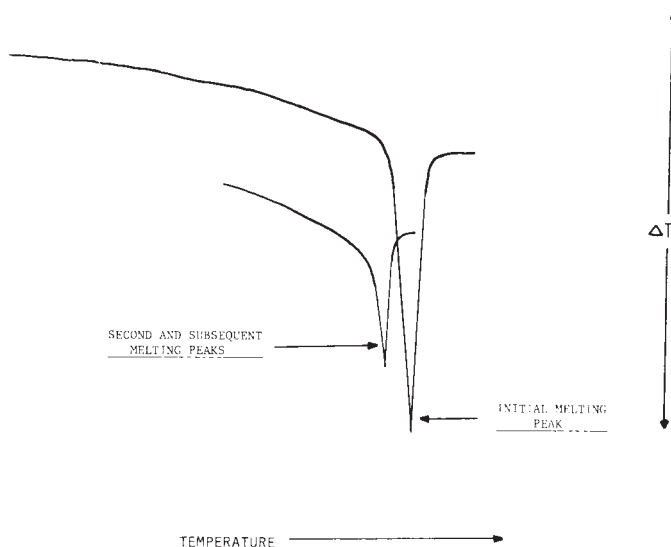


FIG. 5 Melting Characteristics by Thermal Analysis

⁹ A “Vibra-Flow” Feeder, Type FT01A, Available from FMC Corporation, Material Handling Division, FMC Building, Homer City, PA 15748, has been found satisfactory for this purpose.

¹⁰ A “Syntron” controller, Type SCR1B, available from FMC Corporation, address as shown in footnote 12, has been found satisfactory for this purpose.