







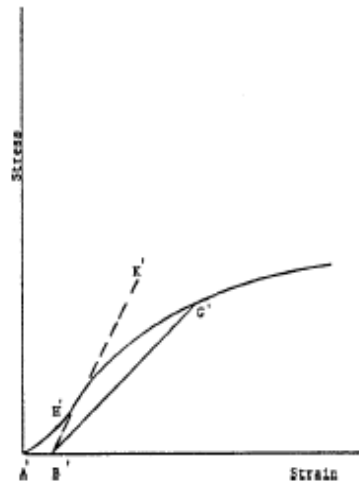
**Tabla 1.4** Especificaciones técnicas de homogeneizador ultrasónico

Nombre del equipo	Homogeneizador Ultrasónico
Modelo	UIP200Ht
Eficiencia	90 % como mínimo
Frecuencia de trabajo	26 kHz
Rango de control	1kHz
Voltaje nominal	240 VAC – 50 Hz
Potencia	200 W

**Tabla 1.5** Especificaciones técnicas de incubadora

Nombre del equipo	Incubadora
Marca	Isotherm
Modelo	IFA – 54 - 8
Rango de temperaturas	32 °C – 100 °C
Voltaje y Potencia máxima	220 – 240 VAC, 50/60 Hz. 1097 W
Dimensiones	620 mm x 530 mm x 840 mm





NOTE 1—Some chart recorders plot the mirror image of this graph.

FIG. A1.2 Material with No Hookean Region

**A2. DETERMINATION OF TENSILE ENERGY TO BREAK**

A2.1 Tensile energy to break (TEB) is defined by the area under the stress-strain curve, or

$$TEB = \int_0^{\epsilon_T} S d\epsilon \quad (A2.1)$$

where  $S$  is the stress at any strain,  $\epsilon$ , and  $\epsilon_T$  is the strain at rupture. The value is in units of energy per unit volume of the specimen's initial gage region. TEB is most conveniently and accurately measured with a tension tester equipped with an integrator. The calculation is then:

$$TEB = (I/K) \quad (A2.2)$$

$$\frac{(\text{full scale load}) (\text{chart speed}) (\text{crosshead speed/chart speed})}{(\text{mean caliper}) (\text{specimen width}) (\text{gage length})}$$

where  $I$  is the integrator count reading and  $K$  is the maximum possible count per unit time for a constant full scale load. This whole calculation is typically done electronically. The results are best expressed in megajoules per cubic metre (or inch-pounds-force per cubic inch).

A2.2 Without an integrator, the area under the recorded stress-strain curve can be measured by planimeter, counting squares, or weighing the cut-out curve. These techniques are time-consuming and likely to be less accurate, since the load

scale on some chart paper is not in round-number dimensions. Moreover, if the curve coordinates are in terms of force and extension instead of stress and strain, the calculated energy, corresponding to the measured area, must be divided by the product of gage length, specimen width, and mean caliper:

$$TEB = \frac{(\text{curve area}) (\text{force per unit chart scale})}{(\text{extension per unit chart travel}) (\text{mean caliper}) (\text{specimen width}) (\text{gage length})} \quad (A2.3)$$

A2.3 For example, if the area under a force-extension curve is 60 000 mm<sup>2</sup>, the load coordinate is 2.0 N/mm of chart scale, the extension coordinate is 0.25 mm of extension per mm of chart travel, and the specimen dimensions are 0.1 mm caliper, 15 mm width and 100 mm gage length, then the calculation for tensile energy to break is:

$$TEB = \frac{(60\,000\text{ mm}^2) (2.0\text{ N/mm}) (0.25 \times 10^{-3}\text{ m/mm})}{(0.1 \times 10^{-3}\text{ m}) (15 \times 10^{-3}\text{ m}) (100 \times 10^{-3}\text{ m})} \quad (A2.4)$$

$$TEB = 200\text{ MJ/m}^3$$

Figura 2.2 Norma ASTM D882, página 10. Especificaciones para el cálculo de la tenacidad.

ANEXO 3

**D3418 – 12<sup>e1</sup>**

often introduce thermal effects because of friction or orientation, or both, and thereby change the thermal history of the specimen.

**8.2 Molded or Pelleted Specimens**—Cut the specimens with a microtome, razor blade, hypodermic punch, paper punch, or cork borer (Size No. 2 or 3) or other appropriate means to appropriate size, in thickness or diameter and length that will best fit the specimen containers as in 7.1.5 and will approximately meet the desired weight in the subsequent procedure.

**8.3 Film or Sheet Specimens**—For films thicker than 40  $\mu\text{m}$ , see 8.2. For thinner films, cut slivers to fit in the specimen capsules or punch disks, if the circular specimen capsules are used.

**8.4** Use any shape or form listed in 8.1 – 8.3 except when conducting referee tests that shall be performed on films as specified in 8.3.

**9. Calibration**

9.1 The purge gas shall be used during calibration.

9.2 Calibrate the DSC temperature signal using Practice E967 and the same heating rate to be used in this test method preferably 10°C/min or 20°C/min (see Note 5). (See Section 10 for details.)

9.3 Calibrate the DSC heat flow signal using Practice E968 and the same heating rate as in 9.2 (see Note 5).

9.4 Some instruments allow for the temperature and heat flow calibration to be performed simultaneously. In such cases, use the same heating rate for this method and follow the manufacturer’s instruction. Report the heating rate. (See 12.1.3.)

**NOTE 5**—Use of other heating rates is permitted. However, test results are affected by the heating rate. It is the responsibility of the user of other rates to demonstrate equivalency to this test method.

**10. Procedure**

**10.1 For First-Order Transition (melting and crystallization):**

10.1.1 The purge gas shall be used during testing. The flow rate of the gas shall be the same as used in the calibration (9.1).

10.1.2 Use a specimen mass appropriate for the material to be tested. In most cases a 5-mg specimen mass is satisfactory. Avoid overloading. Weigh the specimen to an accuracy of  $\pm 10 \mu\text{g}$ .

10.1.2.1 Intimate thermal contact between the pan and specimen is essential for reproducible results. Crimp a metal cover against the pan with the sample sandwiched in between to ensure good heat transfer. Take care to ensure flat pan bottoms.

10.1.3 Perform and record a preliminary thermal cycle by heating the sample at the same rate used for testing from at least 50°C below to 30°C above the melting temperature to erase previous thermal history.

10.1.4 When the effect of annealing is studied, selection of temperature and time are critical. Minimize the time of exposure to high temperature to avoid sublimation or decomposition. In some cases it is possible that the preliminary thermal cycle will interfere with the transition of interest, causing an incorrect transition or eliminating a transition. Where it has been shown that this effect is present, omit the preliminary thermal cycle.

10.1.5 Hold the temperature for 5 min (10.1.3).

**NOTE 6**—In cases that high-temperature annealing cause polymer degradation, the use of shorter annealing times is permitted but shall be reported.

10.1.6 Cool to at least 50°C below the peak crystallization temperature using the same rate that was used for heating and record the cooling curve.

10.1.7 Hold the temperature for 5 min.

10.1.8 Repeat heating at the same rate used in 10.1.3 (10°C/min or 20°C/min) and record the heating curve. Use this curve to calculate the enthalpies of transition.

10.1.9 Measure the temperatures for the desired points on the curves:  $T_{\text{elim}}$ ,  $T_{\text{pm}}$ ,  $T_{\text{efm}}$ ,  $T_{\text{eic}}$ ,  $T_{\text{pc}}$ , and  $T_{\text{cic}}$  (see Fig. 1). Report two  $T_{\text{pm}}$ 's or  $T_{\text{pc}}$ 's if observed.

10.1.10 In case of dispute determine  $T_m$  and  $T_c$  at a heating rate of 10°C/min.

where:

$T_{\text{elim}}$  = melting extrapolated onset temperature, °C,  
 $T_{\text{efm}}$  = melting extrapolated end temperature, °C,  
 $T_{\text{pm}}$  = melting peak temperature, °C,  
 $T_{\text{eic}}$  = crystallization extrapolated onset temperature, °C,  
 $T_{\text{pc}}$  = crystallization peak temperature, °C, and  
 $T_{\text{cic}}$  = crystallization extrapolated end temperature, °C.

**NOTE 7**—The actual temperature displayed on the temperature axis depends upon the instrument type (for example, specimen temperature, program temperature, or specimen-program temperature average). Follow any recommended procedures or guidelines of the instrument manufacturer to obtain specimen temperature at the point of interest.

**10.2 For Glass Transition:**

10.2.1 The purge gas shall be used during testing. The flow rate of the gas shall be the same as used in the calibration (9.1).

10.2.2 Use a specimen mass appropriate for the material to be tested. In most cases, a 10-mg specimen mass is satisfactory. Weigh the specimen to an accuracy of  $\pm 10 \mu\text{g}$ .

10.2.3 Perform and record a preliminary thermal cycle by heating the sample at a rate of 20°C/min from at least 50°C below to 30°C above the melting temperature to erase previous thermal history.

10.2.4 Hold the temperature for 5 min. (See Note 6.)

10.2.5 Quench cool to at least 50°C below the transition temperature of interest.

10.2.6 Hold the temperature for 5 min.

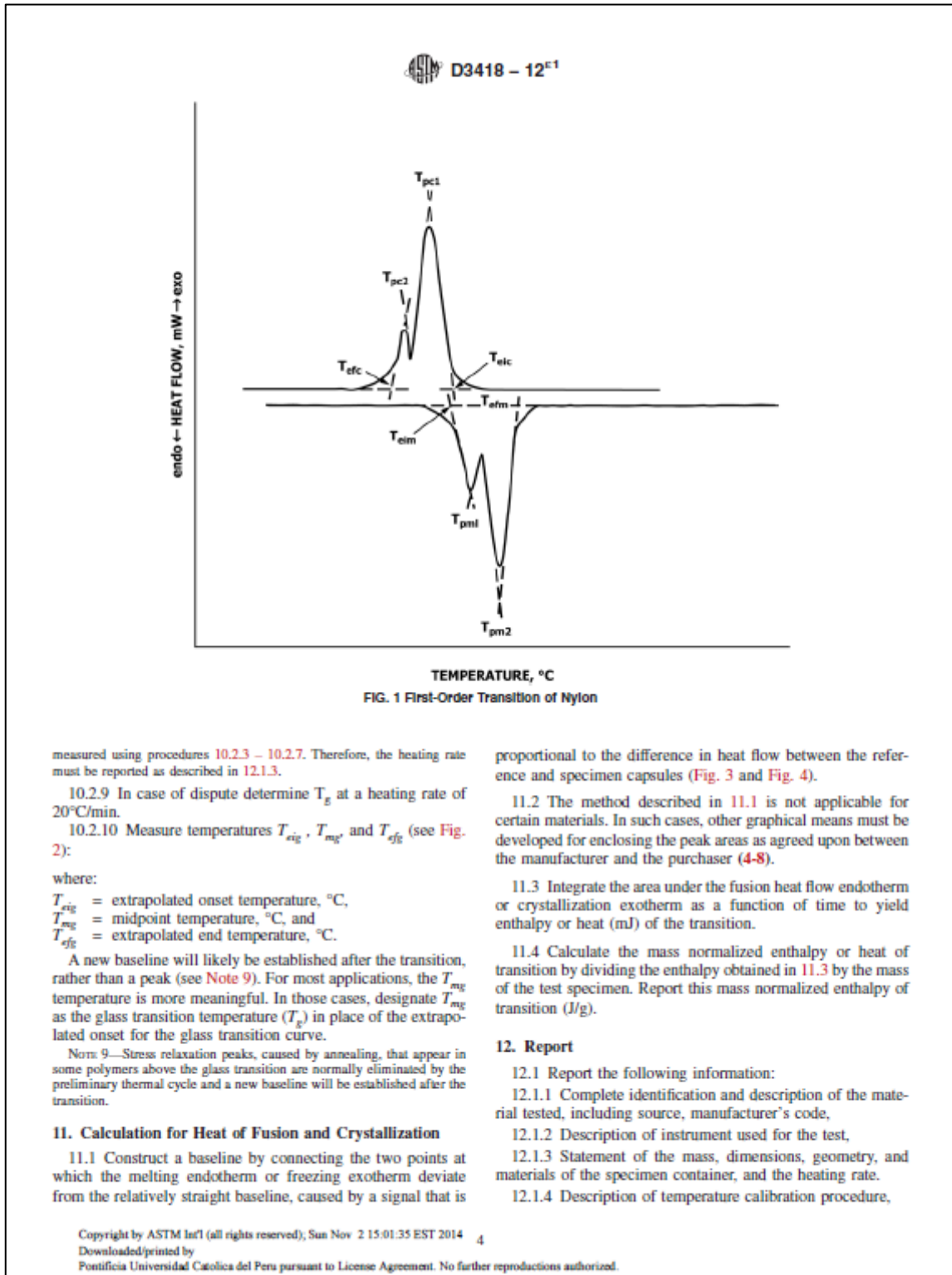
10.2.7 Repeat heating at a rate of 20°C/min, and record the heating curve until all desired transitions have been completed. (See Note 5.)

10.2.8 The glass transition is more pronounced at faster heating rates. A heating rate of 20°C/min is the preferred heating rate for  $T_g$  measurements. The instrument shall be calibrated at the same heating rate used for testing. If both first- and second-order transitions ( $T_m$  and  $T_g$ , respectively) are to be determined in the same run, use the same heating rate as used in 10.1.8 for both transitions and determine results from the second heating step (10.1.8). Report the heating rate. (See 12.1.3.)

**NOTE 8**— $T_g$  obtained using Procedure 10.1 will be different from  $T_g$

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Figura 3.1 Norma ASTM D 3418, página 3. Procedimiento y recomendaciones para la ejecución del ensayo de calorimetría diferencial de barrido.



measured using procedures 10.2.3 – 10.2.7. Therefore, the heating rate must be reported as described in 12.1.3.

10.2.9 In case of dispute determine  $T_g$  at a heating rate of 20°C/min.

10.2.10 Measure temperatures  $T_{eig}$ ,  $T_{mg}$ , and  $T_{efg}$  (see Fig. 2):

where:

$T_{eig}$  = extrapolated onset temperature, °C,  
 $T_{mg}$  = midpoint temperature, °C, and  
 $T_{efg}$  = extrapolated end temperature, °C.

A new baseline will likely be established after the transition, rather than a peak (see Note 9). For most applications, the  $T_{mg}$  temperature is more meaningful. In those cases, designate  $T_{mg}$  as the glass transition temperature ( $T_g$ ) in place of the extrapolated onset for the glass transition curve.

NOTE 9—Stress relaxation peaks, caused by annealing, that appear in some polymers above the glass transition are normally eliminated by the preliminary thermal cycle and a new baseline will be established after the transition.

**11. Calculation for Heat of Fusion and Crystallization**

11.1 Construct a baseline by connecting the two points at which the melting endotherm or freezing exotherm deviate from the relatively straight baseline, caused by a signal that is

proportional to the difference in heat flow between the reference and specimen capsules (Fig. 3 and Fig. 4).

11.2 The method described in 11.1 is not applicable for certain materials. In such cases, other graphical means must be developed for enclosing the peak areas as agreed upon between the manufacturer and the purchaser (4-8).

11.3 Integrate the area under the fusion heat flow endotherm or crystallization exotherm as a function of time to yield enthalpy or heat (mJ) of the transition.

11.4 Calculate the mass normalized enthalpy or heat of transition by dividing the enthalpy obtained in 11.3 by the mass of the test specimen. Report this mass normalized enthalpy of transition (J/g).

**12. Report**

12.1 Report the following information:

12.1.1 Complete identification and description of the material tested, including source, manufacturer's code,  
 12.1.2 Description of instrument used for the test,  
 12.1.3 Statement of the mass, dimensions, geometry, and materials of the specimen container, and the heating rate.  
 12.1.4 Description of temperature calibration procedure,

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Figura 3.2 Norma ASTM D 3418, página 4. Procedimiento para el cálculo de la entalpía de fusión.