



IPC-TM-650 TEST METHODS MANUAL

1.0 Scope This test method establishes a procedure for determining the in-plane coefficient of linear thermal expansion of organic films from 0-200°C using thermal mechanical analysis (TMA).

2.0 Applicable Documents

ASTM D 618 Standard Practice for Conditioning Plastics and Electrical Insulating Materials for Testing

ASTM D 3386 Standard Test Method for Coefficient of Linear Thermal Expansion of Electrical Insulating Materials

3.0 Test Specimen The test specimen shall consist of a strip 15-20 mm long and 2 mm wide with a minimum thickness of 10 µm and maximum thickness of 200 µm.

4.0 Apparatus or Material Perkin-Elmer TMA-7 with a film fixture in extension mode or equivalent equipment capable of handling films less than 25 µm thick.

5.0 Procedure

5.1 The test specimens should be conditioned at 23 ± 2°C and 50 ± 5% relative humidity for not less than 24 hours prior to testing. Refer to ASTM D 618.

5.2 Follow the manufacturer's recommendations for equipment startup and calibration.

5.2 Mount the test specimen in the film holder. The sample length (between the grips) should be between 11-13 mm. Refer to ASTM D 3386.

5.3 Set the force at 30 mN.

5.4 Perform a prescan by heating a rate of 20°C/min. Under inert atmosphere from -10°C to either 10°C above the material glass transition temperature, T_g , or 10°C below the material decomposition limit, T_{max} , determined using nitrogen. T_g may be determined using IPC Test Methods 2.4.24.2, 2.4.24.3, or 2.4.25.

5.5 Hold the temperature for 60 min.

5.6 Cool at a rate of 5°C/min to -10°C.

| | |
|--|----------|
| Number 2.4.41.3 | |
| Subject In-Plane Coefficient of Thermal Expansion, Organic Films | |
| Date 7/95 | Revision |
| Originating Task Group Deposited Dielectric Task Group (C-13a) | |

5.7 Hold the temperature for 10 min.

5.8 Reheat the specimen at a rate of 5°C/min to a maximum temperature of 25°C below the glass transition temperature of the polymer or 10°C below the material decomposition limit, T_{max} , determined under nitrogen. At least two temperature scans of the test specimen should be conducted without disturbing the specimen in the TMA to confirm repeatability of observed test results.

5.9 Calculate the average coefficient of thermal expansion, over the temperature intervals of interest as follows:

$$\alpha = (\Delta L/\Delta T)/L$$

where L is the length of the test specimen between the grips, ΔL is the change in the length of the specimen (in the same units) over the temperature interval ΔT , and ΔT is the temperature interval (normally 200°C) as illustrated in Figure 1. The units are °C⁻¹.

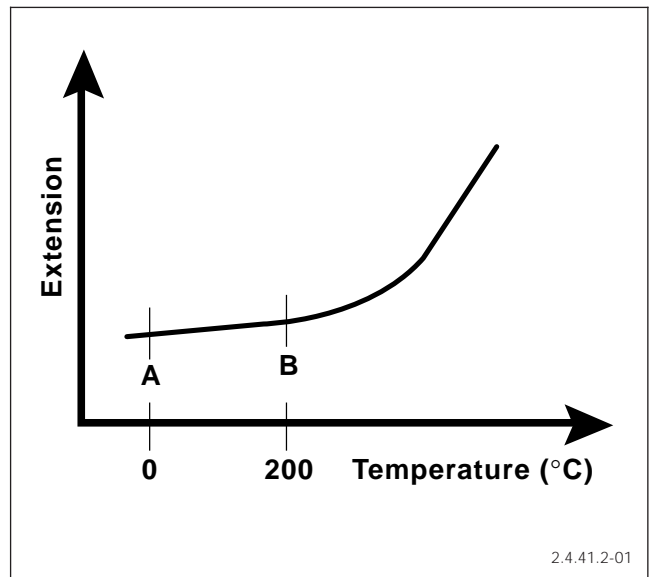


Figure 1

5.10 The coefficient of linear thermal expansion from 0 200°C (below the glass transition) is

$$\alpha = \frac{(\text{Length B} - \text{Length A})}{(\text{Length A}) (\text{Temperature B} - \text{Temperature A})}$$

| IPC-TM-650 | | |
|---------------------------|--|---------------------|
| Number 2.4.41.3 | Subject In-Plane Coefficient of Thermal Expansion, Organic Films | Date 7/95 |
| Revision | | |

5.11 On some instruments ΔL and ΔT may be read directly from the recorder chart. On other instruments, constant factors (from the instrument calibration - see section 6.3) may need to be applied to the chart readings to obtain these values.

6.0 Notes

6.1 Calibration of the instrument must be carried out according to the manufacturer's recommendations. Two calibrations are required, one to establish the baseline and the other to calibrate the TMA relative to a standard.

6.2 A quartz specimen of 11-13 mm in length (between the grips) is run at 5°C/min under inert gas purge (He) from -20 to 400°C to establish a baseline. The baseline is used to eliminate the effects of grip expansion on extension measurements. The coefficient of average thermal expansion of quartz is $0.57 \times 10^{-6}/^{\circ}\text{C}$ (16-500°C)¹. This baseline procedure should be used to either correct the instrument performance to obtain the literature stated value of linear thermal expansion quartz, or, in the event the instrument cannot be adjusted to obtain this value, obtain an estimated correction factor which is then applied to results from test specimens.

6.3 Using a calibration standard with dimensions equivalent to the test specimen, a calibration standard is run between -10 and 200 °C and the observed coefficient of thermal expansion is calculated using the expression:

$$\alpha_{\text{ob}} = (\Delta L/\Delta T)/L$$

where L is the length of the test specimen between the grips. ΔL is the change in the length of the specimen (in the same units) over the temperature interval ΔT , and ΔT is nominally 200 °C. The units of α_{ob} are $^{\circ}\text{C}^{-1}$. An estimated test specimen correction factor, C, is then determined by dividing α_{ob} by the literature value, α_{lit} , for the standard(s). The estimated test specimen correction factor is then as a multiplication factor and applied to the observed linear thermal expansion results for the test specimens.

6.4 The maximum temperature used in this test should be at least 25°C below the glass transition temperature of the material being studied. Heating above the glass transition may alter the morphology of the specimen (e.g., change the molecular orientation) leading to erroneous results. For materials with glass transitions below 250°C, the temperature range over which the coefficient of linear thermal expansion was determined must be noted, e.g., $50 \times 10^{-6}/^{\circ}\text{C}$ (0-150°C).

1. Lange's Handbook of Chemistry, 12th edition, J. A. Dean, ed., McGraw-Hill, New York (1979).