Designation: D7399 – 08

Standard Test Method for Determination of the Amount of Polypropylene (PP) in Polypropylene/LDPE Mixtures Using Infrared Spectrophotometer (FTIR)[1]

This standard is issued under the fixed designation D7399; 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 test method uses an infrared approach for determining the presence of polypropylene (PP) physically mixed in with low density polyethylene – usually for recycling purposes.

NOTE 1—Quantitative determinations require several standard mixtures in the concentration range of interest and well defined baseline anchoring points.

1.2 *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 2—There is no known ISO equivalent to this standard.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D883 Terminology Relating to Plastics

D1600 Terminology for Abbreviated Terms Relating to Plastics

D2238 Test Methods for Absorbance of Polyethylene Due to Methyl Groups at 1378 cm^{-1}

D5033 Guide for Development of ASTM Standards Relating to Recycling and Use of Recycled Plastics (Withdrawn 2007)[3]

E131 Terminology Relating to Molecular Spectroscopy

E168 Practices for General Techniques of Infrared Quantitative Analysis

E932 Practice for Describing and Measuring Performance of Dispersive Infrared Spectrometers

E1421 Practice for Describing and Measuring Performance of Fourier Transform Mid-Infrared (FT-MIR) Spectrometers: Level Zero and Level One Tests

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

3. Terminology

3.1 *Definitions*—For definitions of plastics terms used in this test method, see Terminology standards D883, D1600, and D5033.

3.2 *Terminology*—Units, symbols, and abbreviations used in this test method appear in Terminology E131 or IEEE/ASTM SI-10.

4. Summary of Test Method

4.1 Thin films representing a typical portion of the material are analyzed with an IR spectrophotometer using two absorbance bands—one characteristic for polypropylene (PP) and one for low density polyethylene (LDPE). The ratio of these two absorbencies is used to assess the presence of polypropylene.

4.2 For quantitative determinations, several standards with known concentrations of PP in LDPE bracketing the range of interest are needed to develop a calibration curve. Also, baseline placement shall be defined to produce repeatable results.

5. Significance and Use

5.1 In recycling PP and LDPE, it is important to the end product properties as well as to the processing conditions to know what the purity of the mixture is. In many cases, it is vital to know that the recycled material is reasonably free of a contaminant.

6. Interferences

6.1 Presence of materials other than PP or LDPE in a sample might cause error in measurement.

6.2 Inhomogeneous sample is known to cause false reading. It is recommended to verify sample homogeneity by measuring different areas of the sample.

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods.

Current edition approved Sept. 1, 2008. Published September 2008. DOI: 10.1520/D7399-08.

[2] 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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

7. Apparatus

7.1 *Infrared Spectrophotometer,* either double beam or a Fourier transform (FTIR).

7.1.1 *Double-Beam Infrared Spectrophotometer,* capable of a 4 cm^{-1} spectral resolution as defined in Practice E932. The instrument shall be capable of scale expansion along the wavelength axis.

7.1.2 *Fourier Transform Infrared Spectrometer,* capable of 4 cm^{-1} resolution. The instrument shall be capable of scale expansion along the wavelength axis. Also, see Practice E1421 for testing procedures.

7.2 *Film Mounts,* with apertures at least 6 by 27 mm, to hold the film specimens in the infrared spectrophotometer.

NOTE 3—Attenuated Total Reflectance (ATR) methodology is acceptable as an alternative to the transmission mode. For best results, sample shall be tested using the same mode as the calibration curve.

8. Hazards

8.1 The optical bench of the FTIR spectrometer contains a laser. To avoid eye injury, do not look directly into the laser beam.

9. Procedure

9.1 *Sample Preparation:*

9.1.1 Use film specimens that give absorbencies 1.5 units or less on IR scan. Thickness of approximately 0.025 mm (1 mil) has been found adequate.

NOTE 4—For samples not available in the proper thickness range, the films can be produced on any suitable cast or blown film unit using appropriate conditions that will produce films of uniform thickness and appearance.

9.2 *Spectral Measurements:*

9.2.1 Place the sample in the infrared spectrophotometer.

9.2.2 Set the controls of the infrared spectrophotometer to give a good signal to noise ratio. A spectral resolution of 4 cm^{-1} and a Beer-Norton medium or Happ-Genzel apodization function have been found to be appropriate.

9.2.3 Record the infrared spectrum from 4000 cm^{-1} to 500 cm^{-1}.

9.3 Determine the absorbance of bands at 1373 cm^{-1} and 1465 cm^{-1}—one characteristic for polypropylene (PP) and one for low density polyethylene (LDPE). Draw baseline between 1200 cm^{-1} and 1600 cm^{-1} and use it for both peaks.

NOTE 5—The 1373 cm^{-1} and 1465 cm^{-1} bands present in both spectra, but, within limits, the ratio of the two can be used for differentiation.

NOTE 6—There is a valley between these two peaks. If preferred, draw baseline for individual peak.

9.4 Several standards with known concentrations of PP are needed to develop a calibration curve. The absorbance ratio of 1373 cm^{-1} versus 1465 cm^{-1} is plotted against PP concentration from which the PP content of the unknown sample is obtained. Use the average of three measurements for calibration curve. (See Fig. 1)

9.5 Measure the absorbance ratio of unknown sample and calculate the amount of PP from the calibration curve using the same baseline anchoring points as used for calibration curve.

10. Report

10.1 Report the following information:

10.1.1 Complete identification of material tested including name, manufacturer, lot number and physical form when sampled.

10.1.2 Date of test.

10.1.3 Any sample or spectral anomalies observed during the measurement.

11. Precision and Bias

11.1 An unknown sample was measured ten times. The average content of PP is 26.0 % and the standard deviation is 1.2 %. The coefficient of variation is 4.7 %.

12. Keywords

12.1 infrared spectrophotometer; low density polyethylene; polypropylene

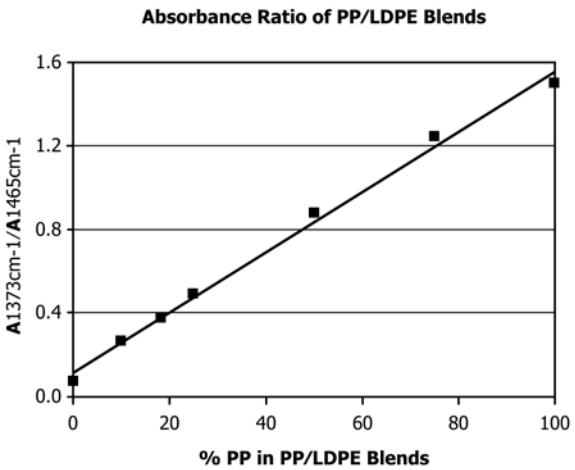

FIG. 1 Example of the Calibration Curve