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An American National Standard

Standard Guide for Using Pyrolysis Gas Chromatography and Pyrolysis Gas Chromatography-Mass Spectrometry in Forensic Polymer Examinations[1]

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INTRODUCTION

The forensic analysis of polymers (for example, fibers, paints and coatings, tapes and adhesives) using pyrolysis gas-chromatography (PGC) or pyrolysis gas-chromatography/mass spectrometry (PGC/MS) is a destructive technique that provides detailed organic chemical information about such samples. This information augments that obtained from other analytical techniques such as Fourier transform infrared spectroscopy (FTIR), polarized light microscopy (PLM), scanning electron microscopy-energy dispersive X-ray spectroscopy (SEM-EDS), and X-ray fluorescence spectroscopy (XRF).

1. Scope

1.1 This guide covers information and recommendations for the selection and application of various PGC and PGC/MS procedures and methods in the forensic examination of polymeric materials (for example, fibers, paint, tape). PGC and PGC/MS methods are used for the identification and comparison of the organic components of these materials. Refer to Practice D3452 for further information on the preparation of the pyrolysis system for polymeric analyses.

1.2 This guide is to be used in conjunction with a broader analytical scheme such as Guides E1610 or E3260, or the SWGMAT Forensic Fiber Examination Guidelines.

1.3 This standard is intended for use by competent forensic science practitioners with the requisite formal education, discipline-specific training (see Practices E2917, E3233, E3234), and demonstrated proficiency to perform forensic casework.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D16 Terminology for Paint, Related Coatings, Materials, and Applications

D123 Terminology Relating to Textiles

D3452 Practice for Rubber—Identification by Pyrolysis-Gas Chromatography

E355 Practice for Gas Chromatography Terms and Relationships

E1492 Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Science Laboratory

E1610 Guide for Forensic Paint Analysis and Comparison

E1732 Terminology Relating to Forensic Science

E2917 Practice for Forensic Science Practitioner Training, Continuing Education, and Professional Development Programs

E3233 Practice for Forensic Tape Analysis Training Program

E3234 Practice for Forensic Paint Analysis Training Program

E3260 Guide for Forensic Examination and Comparison of

[1] This guide is under the jurisdiction of ASTM Committee E30 on Forensic Sciences and is the direct responsibility of Subcommittee E30.15 on Trace.

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[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.

Pressure Sensitive Tapes

2.2 *Other Documents:*

ISO/IEC 17025 General requirements for the competence of testing and calibration laboratories[3]

SWGMAT Forensic Fiber Examination Guidelines[4]

SWGMAT Trace Evidence Quality Assurance Guidelines[4]

SWGMAT Trace Evidence Recovery Guidelines[4]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this guide, see Terminologies D16, D123, and E1732, and Practice E355.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *Curie point, n*—the temperature at which a ferromagnetic metal loses its ferromagnetic properties.

3.2.2 *exclusionary difference, n*—a difference in a feature or property between compared items that is substantial enough to determine that they did not originate from the same source.

3.2.3 *interface temperature, n*—the temperature of the heated zone between the pyrolysis unit and the GC.

3.2.4 *pyrogram, n*—a chromatogram obtained from the pyrolysis products of a material.

3.2.5 *pyrolysis, n*—the thermal fragmentation of a substance in an inert atmosphere.

3.2.6 *pyrolysis temperature, n*—the temperature (set or ramped) at which the pyrolysis of the sample is performed.

3.2.7 *pyrolyzate, n*—the product of the pyrolysis process.

3.2.8 *traceable reference standard, n*—a sample acquired or prepared with documented origin that has known properties for the purpose of calibrating equipment and/or for use as a control.

4. Significance and Use

4.1 This guide provides guidance in the selection of appropriate sample preparation methods and instrumental parameters for the analysis, comparison, or identification of various polymeric materials by PGC and PGC/MS.

4.1.1 PGC/MS can differentiate between classes of fibers (for example, acrylic, polyester, nylon) and within classes of fibers (for example, acrylics) (**1-3**).[5]

4.1.2 Paint binders are differentiated based upon the variety of monomers used in paint formulations which could be difficult to identify by other analytical techniques. In addition, some additives can be detected or identified.

4.1.3 Differentiation can be achieved by the separation and identification of organic components in the adhesive portion of tapes (**4, 5**) and in the backings of electrical tapes (**6**).

4.1.4 PGC/MS can provide additional discrimination for other types of polymers such as automotive lenses, automotive body fillers, cosmetics, plastics, and rubbers (**7-9**).

4.2 Pyrolysis breaks a large molecule into many smaller molecules in a reproducible fashion through the breaking of bonds by means of the application of thermal energy. Analytical pyrolysis is used to provide chemical information on organic-containing solids that cannot be dissolved or otherwise introduced into a chromatographic system. It is also used to analyze and compare solvents bound in a solid material (such as tape adhesives) (**10**). When analyzed using a separation technique such as gas chromatography, the smaller molecules produced through the action of pyrolysis form a pattern of separated fragments. Mass and structural information indicative of the original molecule are also available when a mass spectral detector is used.

4.3 Although a destructive method, and therefore often placed at the end of an analytical scheme, the pyrograms produced from different polymer compositions form characteristic patterns that are useful for both identification of polymer type and comparisons between samples (**4, 6, 9-23**). When used for comparison purposes, the goal is to determine whether any exclusionary differences exist between the samples.

5. Sample Handling

5.1 *General Considerations:*

5.1.1 Practice E1492 as well as the relevant portions of the SWGMAT Trace Evidence Quality Assurance Guidelines and Trace Evidence Recovery Guidelines are followed for the general collection, handling, documentation, and tracking of specimens and samples.

5.1.2 Since PGC is a destructive technique, considerations are made prior to analysis to ensure that the entire sample is not consumed.

5.1.3 Prior to sampling, any foreign debris or contaminants that are visually observed by stereomicroscopy should be scraped away or otherwise removed.

5.1.4 Except where sample size or condition makes it prohibitive, samples for PGC or PGCMS analysis are prepared in a manner that permits resolution and analysis of individual layers. This allows the organic components from each layer to be effectively characterized and attributed to the appropriate layer. When samples are too small to isolate individual layers, samples from multiple layers can be analyzed simultaneously. Samples for comparison are prepared under the same conditions and in the same manner whenever possible.

5.1.5 Sample size can vary in accordance with polymer type, pyrolysis method, column type, chromatographic conditions, and detection methods. As a general approach, varying amounts of a known polymeric material can be analyzed to determine the minimum amount that provides sufficient signal to identify the characteristic components. Once optimal sample size is determined, samples of similar size and shape should be used for analyzing replicates and for comparison purposes. Instrument manufacturer recommendations and published studies of like materials can be used for guidance in determining approximate sample size.

5.1.6 Samples are positioned on or within appropriate sample holders to maximize pyrolysis. More specific guidance on sample holders is discussed in 6.7.1.

[3] Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, https://www.iso.org.

[4] Available from Scientific Working Group for Materials Analysis (SWGMAT), http://swgmat.org.

[5] The boldface numbers in parentheses refer to a list of references at the end of this standard.