X-Ray Diffraction Analysis of Tapes

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1 INTRODUCTION

This document describes the sample preparation and suggested instrumental parameters for the X-ray diffractometry (XRD) analysis of pressure sensitive adhesive tapes.

2 SCOPE

This procedure applies to Chemistry Unit caseworking personnel who analyze paints and polymers via XRD.

3 EQUIPMENT

- X-ray Diffractometer system (e.g., Malvern Panalytical Empyrean, Rigaku MiniFlex, or equivalent) and accompanying software
- Sample holder equipped with low background substrate
- Stereo microscope (~6X to ~50X) with appropriate lighting
- General laboratory supplies (e.g., glass microscope slides, scalpel with blades)
- Solvents for cleaning (e.g., acetone, chloroform)

4 STANDARDS AND CONTROLS

A. Manufacturer-supplied and commercially available tapes, polymers, and adhesives are maintained in the FBI Laboratory. These materials are used in casework in accordance with CHEM-100.

B. Performance checks for the Trace Evidence Unit (TEU) XRD instrument are conducted by personnel in the Geology discipline. Refer to both the instrument logbook as well as GEO-538 to determine if this check is current.

C. The performance check for the Rigaku Miniflex XRD instruments is conducted according to the guidance set forth in IOSS-773.

5 SAMPLING

Refer to PP-800 for guidance on sample(s) selection. Record the samples selected for analysis in the case notes.

6 PROCEDURE

A. Sample preparation will depend on sample type, size, and condition. If the sample is contaminated or too limited in size, record as such in the case notes. All tapes are generally analyzed intact. Additionally, the film backing can be analyzed separately.

1. Prior to analysis, clean the tape backings using a cotton swab or disposable wipe and as necessary, an appropriate solvent (e.g., methanol, acetone).

2. To analyze an intact specimen, obtain a sample area large enough to fill the sample holder. Using the sample holder as a template, cut a sample of the specimen with a scalpel blade. If the specimen is a partial roll of tape, the roll should be unwound approximately three to four inches before sampling in order to eliminate stretching or distorting the film.

3. The same sample preparation should be used for the analysis of the tape backing. The adhesive and scrim fabric, if present, is removed using a suitable solvent (e.g., hexane or chloroform for rubber-based adhesives, acetone for acrylic-based adhesives).

4. Place the specimens on the sample holder in a manner that results in a flat, uniform surface for X-ray beam interaction. Specimens containing adhesive can be affixed directly onto the XRD sample holder. When only the film backing is analyzed, a small amount of adhesive can be applied to affix the sample to the sample holder outside the sampling area where the X-ray beam impinges.

B. Analyze the sample(s). If the compositions of two specimens are to be compared, analyze them both under the same instrumental conditions.

C. Ensure the instrument identification and the operating parameters are recorded on the printed spectra or elsewhere in the case notes.

D. If phase identification in the resulting diffraction pattern is necessary, use the Powder Diffraction File, or analyze an appropriate reference material under the same operating conditions as the unknown sample.

E. Upon completion of the analysis, remove all samples from the sample chamber and, if necessary, return the instrument to standby conditions.

7 INSTRUMENTAL CONDITIONS

The following instrumental conditions serve as a guide for XRD analysis of tape and adhesive standards and samples using the Malvern Panalytical Empyrean:

Generator Settings:

Standby:	30 kV 10 mA
Analysis:	45 kV 40 mA
Anode material:	Copper (Cu)
Filter type:	Nickel (Ni)

Incident Beam Path:

Soller slits incident beam:	0.03 rad.
Soller slits diffracted beam:	0.04 rad.
Fixed divergence slit:	¼°

Fixed anti-scatter slit:	¼°
Fixed beam mask:	14 mm
Secondary beam mask:	14 mm
Scan range:	8 – 80 ° 2Θ
Step size:	0.0131 ° 2Θ (~5 mins/sample)
Scan type:	Continuous

The following instrumental conditions serve as a guide for XRD analysis of tape and adhesive standards and samples using the Rigaku MiniFlex:

Anode material:	Copper (Cu)
Scan range:	5 – 75 ° 2Θ
Scan width:	0.0200 ° 2Θ
Scan speed:	2.000 degrees/min (slow scan, ~ 15mins/sample)
Scan type:	Continuous

8 ACCEPTANCE CRITERIA

A. If exclusionary differences are observed between the diffraction patterns of two (or more) samples being compared, then it is concluded that the specimens differ from one another.

B. If no exclusionary differences are observed between the diffraction patterns of two (or more) samples being compared, then it is concluded that they are indistinguishable from one another by XRD.

C. XRD pattern comparison is one part of a multi-analytical comparative approach. XRD data alone can be used to distinguish the sources of compared samples but is otherwise not used independent of data obtained from other analytical techniques to reach an overall opinion regarding the potential relationship between the sources of the samples. An overall opinion that sources are indistinguishable is only reported when no exclusionary differences are observed in the analytical techniques that were applied.

D. If XRD is being used to characterize a component of the tape (e.g., polyethylene, calcite), the diffraction pattern of that material should compare favorably to a corresponding reference powder diffraction file or known duct tape analyzed in-house.

E. Proper sample preparation is critical for XRD analysis. The following are some common sample preparation problems encountered in XRD analysis:

    1. The displacement of the surface of the specimen away from the diffraction axis is the primary source of error in the measurement of diffraction peak

positions. The sample must be spread flat on the specimen holder without extending above or below the instrument's diffraction axis.

2. Any sample preparation technique should minimize introducing stress and orientation effects ("preferred" orientation).

9 LIMITATIONS

A. On average, the lower limit of detection of a component in a mixture is approximately 1%. This limit will vary depending on composition, degree of crystallinity, and crystallite size.

B. Limited sample size can preclude analysis by this technique.

10 PRECAUTIONARY STATEMENTS

A. As with any procedure involving trace evidence, ensure actions minimize the potential for loss or contamination of the sample.

B. If components in a sample yield a similar diffraction pattern, the peaks can overlap one another; therefore, comparing the composite pattern with known powder diffraction patterns of individual compounds can be difficult.

C. The elasticity inherent in some polymers can cause the flattened backing sample to curl up if insufficient mounting media is used to hold the sample flat and stationary.

11 SAFETY

Personal radiation monitors (dosimeters) are administered by the Health and Safety group to monitor exposure to ionizing radiation. Operators should familiarize themselves with the specific User's Guide safety section of the instrument prior to use.

12 REVISION HISTORY

Revision	Issued	Changes
06	10/28/2020	Updated to reflect newest TEU instrument and SOP throughout. Removed grade of reagents used in equipment list. Minor edits as needed to conform to LOM changes. Minor grammatical edits. Removed need to refer to instrument manuals in Section 5. Edited Section 7 for clarity. Edited Section 10 to remove limitation previously covered in 10iv.
07	08/15/2022	Reformatted to conform with LAB-100 revisions. Minor grammatical changes throughout. Simplified and updated equipment and limitations. Moved references to training manual.