

XRF Analysis of Electrical Tape Backings

Table of Contents

1	INTRODUCTION	2
2	SCOPE	2
3	EQUIPMENT	2
4	STANDARDS AND CONTROLS	2
4.1	Controls.....	2
4.2	Performance Check	2
5	SAMPLING	2
6	PROCEDURE	3
6.1	Sample Preparation.....	3
6.2	Analytical Procedure	3
6.3	Instrumental Conditions.....	3
7	ACCEPTANCE CRITERIA.....	4
8	LIMITATIONS	4
8.1	Precautionary Statements.....	4
9	SAFETY	5
10	REFERENCES.....	5
11	REVISION HISTORY	6

XRF Analysis of Electrical Tape Backings

1 INTRODUCTION

This document describes the sample preparation and suggested instrumental parameters for the X-ray Fluorescence (XRF) Spectroscopy analysis of electrical tape backings.

2 SCOPE

This procedure applies to Chemistry Unit caseworking personnel who analyze electrical tape backings via XRF.

3 EQUIPMENT

- A. Energy Dispersive X-ray Fluorescence spectrometer: Bruker M4 Tornado spectrometer (or equivalent)
- B. Energy Dispersive X-ray acquisition software (Bruker M4 software or equivalent)
- C. Glass microscope slides
- D. Square petri dish with lid
- E. Graphite planchet
- F. Carbon tape
- G. Disposable wipes
- H. Cotton tipped applicators
- I. Tweezers
- J. Scissors
- K. Scalpel handle with blades
- L. Acetone (reagent grade or equivalent)
- M. Hexane (reagent grade or equivalent)
- N. Methanol (reagent grade or equivalent)

4 STANDARDS AND CONTROLS

4.1 Controls

Manufacturer-supplied and commercially available electrical tapes are maintained in reference collections within the FBI Laboratory.

4.2 Performance Check

Refer to the *Compositional Analysis by Energy Dispersive X-Ray Fluorescence Spectrometry (EDXRF)* and *Operation of the Bruker M4 Tornado X-Ray Fluorescence Spectrometer* technical procedures for details on the necessary performance checks and supplies to operate the instrument.

5 SAMPLING

Refer to the current version of *the General Approach for Tape Casework* technical procedure for guidance on sampling. Record the sample(s) selected for analysis in the case notes.

6 PROCEDURE

6.1 Sample Preparation

- A. Prepare samples in as similar a manner as possible.
- B. Samples are first examined with a stereomicroscope. If extraneous (contaminant) materials are present, remove using the tip of a scalpel blade, by taking a series of thin peels, or with a cotton-tipped applicator moistened with a suitable solvent (e.g., methanol, acetone). When extraneous materials cannot be removed, note their location during light microscopy and avoid these areas during subsequent XRF analysis.
- C. Adhesive removal is preferred. Use a small amount of solvent (e.g., acetone, hexane) as needed. Adhesive should be removed in an area large enough to account for repeat measurements with a 1cm x 1cm area.
- D. Samples should be mounted on a graphite planchet (or other appropriate medium) with carbon tape at the edges to hold them in place.
- E. Place the mounted sample on the stage and close the chamber door.

6.2 Analytical Procedure

- A. Within the instrument software, use the x, y, and z coordinates to position the sample, the 10x magnification to course focus the sample, and then the 100x magnification to fine focus and analyze the sample. To generate replicates, move the stage or select different spots using the instrument software.
- B. Once an X-ray spectrum is collected, perform a spectral peak identification in order to determine the elements present.
 1. The presence of an element is considered unequivocal only when a distinctive set of lines is produced, or when an element's response is a single peak that occurs at an energy where it cannot be mistaken for another element or artifact. Label the peak(s) with the corresponding elemental symbol.
 2. Unequivocal identification may not be possible if an element is present in low concentration or if lines required for confirmation are overlapped with the lines of (an)other element(s). When identification is probable, but not unequivocal, the operator can manually add the elemental symbol in parentheses on the printout.
- C. Qualitative analysis of data includes a spectral overlay following peak identification. This is done to compare samples for presence/absence of elemental peaks.

6.3 Instrumental Conditions

- A. The following operating conditions are meant as general guidelines. The following instrumental conditions are not intended to be prescriptive nor exhaustive. Minor modifications to the conditions may be used as needed and without authorization, provided the same conditions are used for all inter-compared samples. The utilized conditions will be recorded and retained with the case notes.

- B. Actual requirements can vary as determined by specific analytical needs.
 - o Atmospheric condition: Vacuum
 - o X-Ray tube: Rh target
 - o Tube Voltage/Current: 45 kV/400 μ A
 - o Acquisition Time: \geq 60 live seconds
 - o Mounting Material: graphite planchet
 - o Minimum number of replicates: 3
 - o Stage: If sample is placed in the center of the stage on a graphite planchet, set the stage to the following position locations.
 - x: 100
 - y: 100
 - z: 115

7 ACCEPTANCE CRITERIA

- A. When exclusionary differences are observed between compared spectral features, the sources of the samples are considered distinguishable by XRF. Exclusionary differences in spectral comparisons: 1) are outside the variability of spectra originating from the same source; and 2) cannot be explained by considerations such as sample heterogeneity, contamination, different sample conditions, or different sample histories.
- B. When no exclusionary differences are observed between compared spectral features, the sources of the samples are considered indistinguishable by XRF. Differences that are not considered exclusionary: 1) are within the variability of spectra originating from the same source; or 2) can be explained by considerations such as sample heterogeneity, contamination, different sample conditions, or different sample histories.

8 LIMITATIONS

- A. The information available from a specimen can diminish as its size is reduced and its condition degrades. Individual replicates may no longer be representative of the original material.
- B. No electrical tape standard reference material currently exists. As a result, there is no direct comparison to known element concentrations in order to perform quantitative measurements.
- C. Overlap of peaks can occur. The following are some commonly occurring overlaps encountered in electrical tape backing spectra: CaK α /SbL α , BaL α /TiK α . Describe limitations of the technical procedure.

8.1 Precautionary Statements

- A. As with any procedure involving trace evidence, ensure actions minimize the potential for loss or contamination of the sample.
- B. Consider analyzing the planchet if there is concern for potential adverse contribution from the planchet.

- C. If spectral differences are detected, it is likely that the materials that produced them are not similar in composition; however, several alternative explanations are possible. These include heterogeneity of the sample and X-ray contribution from extraneous material.

9 SAFETY

Use standard precautions for the handling of potentially biohazardous materials, chemicals, or sharps. Operators should wear a personally assigned dosimeter to monitor exposure to ionizing radiation when in proximity to the working instrument.

10 REFERENCES

Compositional Analysis by Energy Dispersive X-Ray Fluorescence Spectrometry (EDXRF), FBI Laboratory, Chemistry Unit- Metallurgy

General Approach for Tape Casework, FBI Laboratory, Chemistry Unit- Paints and Polymers

Operation of the Bruker M4 Tornado X-Ray Fluorescence Spectrometer, FBI Laboratory, Chemistry Unit- Metallurgy

11 REVISION HISTORY

Revision	Issued	Changes
00	12/02/2021	Original document issued.