



# Elemental analysis and characterization of electrical tape backings by LA-ICP-MS



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## ABSTRACT

Adhesive tapes are commonly found as physical evidence in cases involving violent crimes and national security threats. This research evaluated the utility of Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry (LA-ICP-MS) for the characterization of chemical signatures of electrical tapes for forensic comparison and provenance purposes. The backings of 90 black electrical tapes, previously characterized by microscopical examination, Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS), Fourier Transform Infrared Spectroscopy (FTIR) and Pyrolysis-Gas Chromatography-Mass Spectrometry (Py-GC-MS), were analyzed by LA-ICP-MS to evaluate the ability of the technique to discriminate samples originating from different sources and to associate pieces of tapes originating from the same roll. The results showed that LA-ICP-MS is a useful tool that complements current protocols for the organic and inorganic characterization and comparison of electrical tapes and results in improved discrimination and superior characterization. The developed LA-ICP-MS method alone provided 94% correct discrimination of the tapes known to originate from different rolls and 100% correct association of the tapes known to originate from the same roll. Moreover, LA-ICP-MS captured a large amount of compositional information, as the use of elemental profiles alone provide similar discrimination and classification capabilities as all the conventional methods together. The analytical sensitivity of LA-ICP-MS provides a means for the classification of tapes to support investigations as well as the potential for database searching capabilities in the future.

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## 1. Introduction

Adhesive tapes are submitted to forensic laboratories as evidence related to the construction of improvised explosive devices, illicit drug packaging, kidnappings, murders, ligatures, and other criminal activities. Electrical tapes can be recovered as part of the pre-blast and/or post-blast evidence. Adhesive tapes are normally composed of a backing and an adhesive layer. In the case of electrical tapes, the backing layer is typically made of polyvinyl chloride (PVC), although polyethylene and polypropylene are also used [1]. Stabilizers, fillers, colorants, and flame-retardants may also be present in the backing [2]. In addition, plasticizers are added to pro-

vide flexibility to PVC, which is normally rigid at room temperature. The adhesive layer consists of an elastomer base, tackifying resins, plasticizers, additives, and fillers. The major component of the adhesive is the elastomer, which is usually natural rubber; a copolymer of some combination of styrene, isoprene, and butadiene; or acrylate [3]. In both backings and adhesives, fillers may be used to increase the bulk and lower the manufacturing cost without adversely affecting the tape performance.

The analysis of tapes typically involves the comparison of two or more pieces of tape using physical and microscopical examinations and chemical identification of organic and inorganic constituents.

The most common methods used for the organic analysis of electrical tapes are FTIR and Py-GC-MS [1,2,4]. IR spectroscopy is a quick universal technique that provides identification of organic compounds without the need for sample preparation. However, in some instances, FTIR presents some limitations for the analysis of electrical tape backings because primary components of the plasticizer may mask the detection of other components [5]. Py-GC-MS

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is capable of providing chemical information on a wide range of organic materials difficult to be analyzed by IR. Py–GC–MS is therefore complementary to infrared spectroscopy, and it provides separation (retention time) and identification (mass spectrometry) of organic compounds [2]. However, Py–GC–MS is destructive and time consuming and therefore is recommended as the last analytical step in tape examinations.

The elemental composition of adhesive tapes has been previously analyzed by SEM-EDS and Micro-X-ray Fluorescence ( $\mu$ -XRF) [1,2,4,6]. As reported by Mehlretter et al. [2], the elemental characterization by SEM-EDS allowed for 87% discrimination by pairwise comparison; this technique showed to be the most discriminating tool for electrical tape backings. Although SEM-EDS and  $\mu$ -XRF proved useful for the inorganic characterization of tapes, these techniques present some limitations such as low sensitivity and selectivity. In addition, the penetration depth of the  $\mu$ -XRF beam into the sample may cause contamination issues between the backing and adhesive layers, requiring additional sample preparation steps.

LA-ICP-MS is proposed as a valuable complementary tool in tape examinations because of its superior sensitivity and selectivity, minimal sample destruction, short analysis time, and little to no sample preparation. Moreover, this study investigated if the chemical signature of tape backings could be used beyond comparative purposes to provide useful intelligence information about sources of origin.

## 2. Material and methods

### 2.1. Instrumentation and measurement parameters

The analysis by LA-ICP-MS was performed using a quadrupole ELAN DRC II (Perkin Elmer LAS, Shelton CT USA) ICP-MS coupled to a ns-Nd:YAG laser (NW UP213, New Wave, California). Data was acquired in mass scanning mode from  $m/z$  <sup>7</sup>Li to  $m/z$  <sup>238</sup>U. Performance checks were conducted daily and before each analysis to ensure the correct operation of the instrument. Standard reference material NIST 612 was used to monitor oxides (ThO/Th) and doubly-charged (Ca<sup>++</sup>/Ca) ratios we monitored, as well as the intensity counts for the background (mass 220), and for light, medium, and heavier isotopes (Li, Ce, La, U). The final element list was reduced to 29 elements that were found to be relevant for characterization of the backing components. Table 1 shows the optimum instrumental parameters for LA-ICP-MS of electrical tape backings. Spectral regions that were anticipated to have large contribution from Ar isotopes and other polyatomic interferences were excluded from the scanning method.

Microwave digestion was performed using the SK-15 Ethos UP microwave digestion system (Milestone, Shelton CT USA).

SEM-EDS analysis was also conducted using a Philips XL 30 scanning electron microscope (Philips, The Netherlands) coupled with an energy dispersive spectrometer detector (EDAX, USA) using a method previously reported [2]. The SEM-EDS was operated at 50X magnification, 15 mm working distance, 25 kV accelerating voltage and 200 s of acquisition time.

### 2.2. Sample collection and sample preparation

#### 2.2.1. Tape samples

Six electrical tapes rolls (Table 2) were locally purchased at retail stores. Each tape roll was divided into six sections and each section was further split into three (11 inches) subsections to constitute each analytical sample. The tape samples were placed on transparency films (Apollo, Acco Brands) and stored in plastic protectors. The local tapes were used to assess the intra-roll and inter-

**Table 1**

Optimized parameters for the analysis of tape backings by LA-ICP-MS.

LA-ICP-MS Optimized Parameters	
Laser	ns-Nd:YAG (213 nm)
Energy	100% (2.6 mJ)
Stage Speed	40 $\mu$ m/s
Spot Size	190 $\mu$ m
Frequency	10 Hz
Ablation Mode	Line
Line Length	4 mm
Scanned Spectra	$m/z$ 7 to $m/z$ 238
Sweeps/Reading	40
Readings/Replicate	1
Carrier Gas	Helium
Gas Flow	0.9 L/min

**Table 2**

Locally purchased black electrical tapes.

Sample Roll	Brand Name	UL
T02	Scotch (Super 88+)	539 H
T03	Scotch (Super 33+)	539 H
T04	Scotch	539 H
T05	Commercial Electric	E 305030
T06	General Electric	362 K
T07	General Electric	362 K

rolls variations in electrical tapes. Four out of the six tapes were also used as intra- and inter-day duplicate controls.

A selection of 90 black electrical tapes previously analyzed by Py–GC–MS, SEM-EDS, FTIR, and microscopical examination by Mehlretter et al. [1,2] was shared with our research group to assess the capabilities of SEM-EDS and LA-ICP-MS analyses. The samples were received as tape segments placed on plastic transparency films. The samples were stored in plastic protectors prior to and after analysis.

Prior to LA-ICP-MS analysis, a piece of  $\sim$ 1 cm by 2 cm of tape was cut and placed directly inside the ablation chamber.

#### 2.2.2. Certified standard sample preparation

A standard for tape analysis was prepared utilizing a certified reference material in pellet form (Polyethylene ERM-EC681m) that was purchased from the Institute of Reference Materials and Measurements (IRMM, Geel, Belgium). The certified polyethylene pellets were melted on a glass slide and cut into strips of  $\sim$ 1 cm by 2 cm. The thickness of the films was  $\sim$ 2 mm. Microwave digestion was performed for both the films and the pellets to determine any loss of analytes during the melting process and to assess the performance of the digestion. Fig. 1 represents the comparison between the recovery for the pellets and the recovery of the prepared film for the ERM-EC681m standard. The recovered concentrations for the pellets and the films show no significant loss after the melting process. The certified film can therefore be used for the semi-quantitative analysis of tapes and as calibration standards to account for instrument differences between laboratories.

## 3. Data reduction and statistical analysis

Data pre-processing included the removal of non-relevant mass-to-charge peaks originating from polyatomic and isobaric interferences [7] and normalization to the sum of the intensity peaks to account for any shot-to-shot variation and/or inter-day variations and as a mean to compensate for mass removal differences between replicates. In the absence of an internal standard, this normalization strategy accounts for small differences in the

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