B214 A Survey of Elements Detected in Automotive Paint Layers by Scanning Electron Microscope/Energy Dispersive X-Ray Spectrometry (SEM/EDS)

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Learning Overview: After attending this presentation, attendees will develop an understanding of the major, minor, and trace elements commonly detectable in automotive paint samples by EDS in an SEM.

Impact on the Forensic Science Community: This presentation will impact the forensic science community and, in particular, trace evidence analysts by providing an increased context by which to assess the significance of elemental data collected from automotive paint samples.

The elemental analysis of paint layers by SEM/EDS is a distinct step in the forensic comparison of automotive paint as defined by the American Society for Testing and Materials (ASTM) Guide E2809. While the elemental analysis of paint by SEM/EDS is utilized by many laboratories conducting paint analysis, case reviews and a review of several crime laboratory Standard Operating Procedures (SOPs) show that various stages of this process, from sample preparation through interpretation, could be better defined. A set of 300 automotive paint samples (i.e., street samples), comprising 1,203 individual layers, was employed to investigate each step of this process. Here, data on the elements that were detected in each layer of this automotive paint sample set are presented. While this is nominally a trivial process for major elements, the impact of closely spaced layers, the choice of analytical variables, and the challenges of objectively processing spectra demonstrate that this is not a trivial process for the determination of minor and trace elements.

Layers in each paint sample were delineated and identified as a clear coat, tinted clear coat, base coat, or primer by a combination of light microscopy and backscatter electron imaging. Using a thick section, each of the 1,203 layers (356 clear coats, 79 tinted clear coats, 347 color coats, and 421 primer layers) was then analyzed by EDS twice, first using livetimes of 20s and 200s. In both cases, analyses were performed from a manually drawn raster area within each layer. The resulting spectra were evaluated to determine which elements were detected in each layer. To avoid the subjectivity of manual peak assignments, this work evaluated several objective criteria for peak detection. Various combinations of the following approaches were compared: (1) a fixed element list, (2) an automated peak identification algorithm, (3) a 3-sigma detection limit threshold (based on the EDS counting error), and (4) fixed weight percent detection limit thresholds. Through iterative processing of the data, a fixed list of 24 elements was developed, which included every element detected in at least one of the 1,200 layers. Spectra from both the 20s and 200s livetime runs were then processed using three detection limits: 3-sigma (representing the lowest detection limit), 0.25wt-% fixed, and 0.50wt-% fixed (representing the highest detection limit). Using the highest detection limit (0.5wt-%) and a 20s analysis time, four elements (carbon [C], nitrogen [N], oxygen [O], and silicon [Si]) were detected in at least 40% of the clearcoats. Seventeen elements were detected in at least 1% of the color coats (C, N, O, magnesium [Mg], aluminum [Al], Si, phosphorus [P], sulphur [S], chlorine [Cl], potassium [K], calcium [Ca], titanium [Ti], chromium [Cr,] iron [Fe], copper [Cu], zinc [Zn], and barium [Ba]). Seventeen elements were detected in at least 1% of the primer layers (C, N, O, Mg, Al, Si, P, S, Cl, Ca, Ti, Fe, Zn, tin [Sn], Ba, bismuth [Bi], and strontium [Sr]). The average number of elements detected in a given layer increased from the clear coat (four) to the color coat (seven) to the primer (nine). The 200s analyses increased the number of elements detected; however, this impacted only elements detected at low concentrations (i.e., near the detection limit).

These results have numerous practical applications: (1) each of the detected elements can be associated with an expected component in the paint (e.g., a binder, filler, pigment, or additive); (2) this data provides information about the frequency of particular elements in each layer of paint, as well as their anticipated concentration range in the paint; and (3) this dataset provides new insight into the significance of the suite of elements detected in a questioned automotive paint layer and sample, which may ultimately improve the value of elemental data obtained from paint samples.

Paint, SEM/EDS, Elements

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