B11 Discrimination of Glass by Cathodoluminescence, Color Analysis, and Chemometrics

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After attending this presentation, the attendees will be familiar with the application of CIE L*a*b* color coordinates to cathodoluminescence spectra of glass. The presentation will cover cathodoluminescence spectroscopy as well as associated uncertainties with color determination. Chemometric analysis of data will also be discussed.

This presentation will impact the forensic community by demonstrating a new technique, color of glass derived from cathodoluminescence spectra, that may be employed to link suspect glass to known glass.

Forensic scientists are well versed in dealing with colored physical evidence, such as fibers and paints. Glass is a common piece of physical evidence that is generally visually colorless. However, under cathodoluminescence (CL) conditions, glass emits light in the visible range and in effect can be treated as colored. CL is a phenomenon that occurs when visible light is emitted from a material upon electron bombardment. This study utilized CL spectroscopy. Analysis of glass by CL spectroscopy detects luminescence in the visible range. This range of detection can assist in assigning a color to a colorless glass. In this study, a scanning electron microscope (SEM) equipped with a CL detector was used. The electrons generated in the SEM provide sufficient conditions for CL detection. The combination of SEM and CL results in a non-destructive technique for analysis, thus allowing the integrity of the sample to be maintained. Sample preparation for analysis of materials in the SEM is relatively simple. For glass samples, no surface preparation was necessary; the surface did not require carbon coating. Currently employed methods of glass analysis include scanning electron microscopy equipped with energy dispersive spectroscopy (SEM-EDS), laser ablation inductively coupled plasma mass spectrometry (ICP-MS), refractive index measurements, and x-ray fluorescence spectrometry (XRF).

Soda-lime float glass (NIST1830) and multicomponent glass (NIST1412) were analyzed via CL spectroscopy. For each sample, spectra from five different areas were collected as well as five replicate spectra of the same area. Data analysis and chemometrics was conducted using Excel and MatLab. ASTM Standard Methods (D2244, 2616, 6290 and E284, 308, 313, 1164, 1345, and 2022) were used as references and starting points for color determination and calculations. For extraction of tristimulus values, L*a*b coordinates, and color differences, spectra were standardized to a D65 illuminant, 10°observer, 1964 weighting table factors from ASTM. Intra-sample variation was addressed by collection of replicate emission spectra from the same location on a standard glass sample. Although intensity decreased with each collection event (likely due to a surface charging effect), the spectral pattern was consistent. Accordingly, all spectra were normalized to the highest emission for analysis. A similar procedure was used for single samples collected from different locations on the same glass sample. Together, these measurements established the range of instrumental, inter-, and intra-sample variation. The quantitative characterization of this uncertainty was essential in identifying statistically significant color differences between different glass samples.

Results showed that the L*a*b* values for each glass had a %RSD of between 4% and 15% for the five spectra collected in different areas of the sample. Even with these ranges, the two NIST glasses were easily distinguished and the difference was statistically significant. Once uncertainties were determined, it was found that there some intra-sample variation exists within each glass. Although variations do exist, the two types of glass were easily distinguished from one another. This work demonstrates the need for replicates to be collected from each sample. Future work includes CL analysis as well as offline data analysis of non- NIST standard glasses.

The impact of color of glass derived from cathodoluminescence spectra will provide the forensic community with a new technique that may be employed to link suspect glass to known glass.

Cathodoluminescence, Glass, CIE L*a*b* Coordinates