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Potential of forensic analysis of multicomponent samples

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Introduction
Forensic laboratories analyse wide variety of samples, either man made or created by nature. Regarding inorganic materials, laboratories most often combine the SEM/EDS/WDS, EBSD, CL methods with optical microscopy in transmitted and reflected light and fluorescence, XRF/mXRF, Raman spectroscopy/FTIR, XRD/mXRD, and others. These methods are by large non-destructive and samples therefore retain their full potential for further evidencing of cases.

Methodology
The case below concerned identification of bismuth applied rather unconventionally in the red layer of polychrome under gilding and silvering layers on a Krivoklat Castle chapel altar (around 1480-1490), Central Bohemia (Czech Republic). This surprising discovery resulted in a complex research of the altar, focusing on the layered structure and the method of bismuth layering to metal-coated fields. The sample was screened with optical microscopy followed with SEM and elementary mapping, and then performed by XRD micro-diffraction phase analysis, which allows for an analysis of a surface of approximately 100 μm and enables for identification similar to microscopic methods. It can be also used to analyse individual grains of pigments in the colour layer. In the mono-capillary, there is a total reflection of the X-ray beam and the divergent beam becomes a quasi-parallel one. Apart from collimating the X-ray beams, the mono-capillary acts as an energy filter and suppressor of white radiation. The mono-capillary is optimized for Cu Kα radiation, but can also be used for other types of radiation where energy drops below 10 keV, such as Cr, Fe, and Co Kα. The XRD technique is irreplaceable especially for phase identification or their components which are indistinguishable by chemical composition analysis. For accurate determination of the analysed point, the image analysis is used. The image of primary beam spot is transferred to an overlay image and a live image is of the real sample. They can therefore adjust heterogeneous samples very precisely. For micro fragments the zero background silicon plate is used. Presence of exotic Bi phase was verified by another independent technique - orthogonal TOF-SIMS FIB. The system presents a new potential in electron microscopy, and is now available as attachments to dual SEM/FIB. The compact orthogonal extraction TOF instrument works with a 0.5m nominal drift length, including the reflectron. In this case, the selected part of the scanned area (10 x 10 μm) was analysed. The primary ion beam energy was at 500 pA and 3D volume element mapping was performed. In detailed TOF-SIMS mass spectrum the peak Bi (mass 209) limits of integration was clearly identified.

Conclusions
These methods are complementary to standard ones and enable verification of analytical findings by independent methods. This is especially important in forensic science since the law enforcement bodies often use the experts´ findings and conclusions in criminal proceedings.
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