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Blunders in Automatic Qualitative Analysis (Peak Identification) for Energy Dispersive X-ray Spectrometry: the Low Voltage Microanalysis Situation

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Comprehensive analytical systems for energy dispersive spectrometry (EDS) x-ray microanalysis include an automatic qualitative analysis (peak identification) procedure. The results of these automatic peak identifications are routinely accepted by some analysts, especially those novices with little experience as well as analysts who make use of the "one button spectrum-to-final report" operation that is now possible with some systems. Previous work has shown that at least some automatic peak identification systems of recent installation are subject to occasional egregious mistakes when identifying the prominent peaks of major constituents, i.e., elements present at concentrations above 0.1 mass fraction (10 weight percent) [1]. A misidentification of a major component of a material, such as a member of a binary, stoichiometric compound like KBr, constitutes such a severe mistake that it qualifies as a "blunder", described in statistics literature as an "illegitimate error" [2]. Blunders are mistakes that damage the credibility of the analyst. Such blunders occur for approximately 3% to 5% of peak identifications for major elements selected across the Periodic Table. In general, automatic peak identification appears to be quite robust for elements identified with K-shell x-rays. When blunders are encountered, they are usually associated with peak identifications that involve L-shell and M-shell photons below 4 keV. Automatic peak identification is also more robust when the procedure incorporates redundant information available when K-L or L-M x-ray combinations occur for an element, e.g., ZnK-ZnL or TaL-TaM. Many systems provide manual software tools to "ground truth" the automatic solution, but such tools often rely on a good understanding of x-ray spectrometry and may be ignored under full automation.

Low voltage microanalysis involves incident beam energies of 5 keV or lower, a value selected because it represents the lowest beam energy for which a measurable characteristic x-ray peak exists at the major constituent level for all elements except H, He, and Li [3]. In the low-voltage beam, low-energy photon regime, K-L and L-M redundancy is lost, and the energy separation of peak family members is reduced, decreasing the information available to make an elemental identification and making blunders more likely. Figure 1 shows the spectrum of a zircon excited with E_0 = 5 keV where a blunder occurs when PtM is identified instead of ZrL. Figure 2 shows a spectrum of KBr with E_0 = 5 keV where BrL is misidentified as AlK. These blunders likely occur because the peak channel of the α–β composite peak from the EDS convolution is sufficiently displaced from the expected channel for the α-peak to give a false look-up table solution. The expedient solution to recognize the blunder would be to increase the beam energy and seek additional x-ray families. When the beam energy must be maintained in the low voltage range, spectrum fitting against known peak references can provide the needed information to determine which element is actually present.

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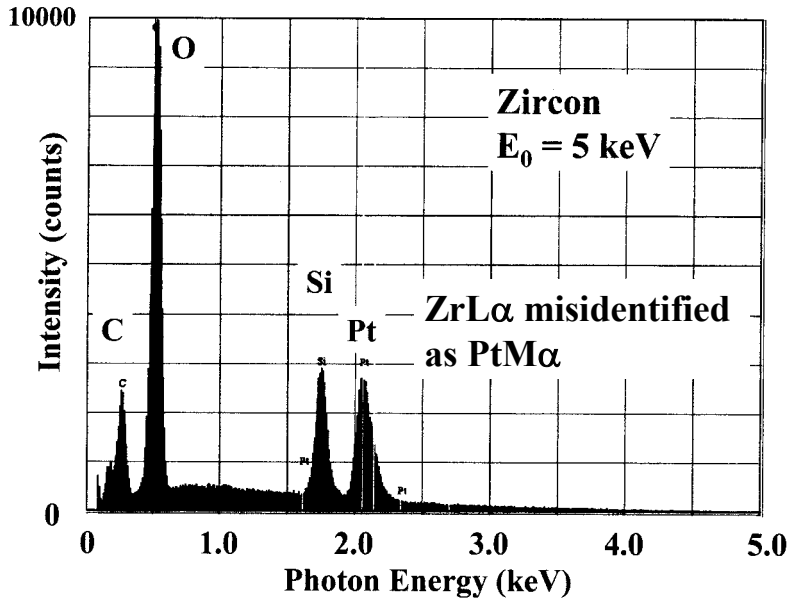

Figure 1. EDS spectrum of zircon with automatic peak identification; E_0 = 5 keV

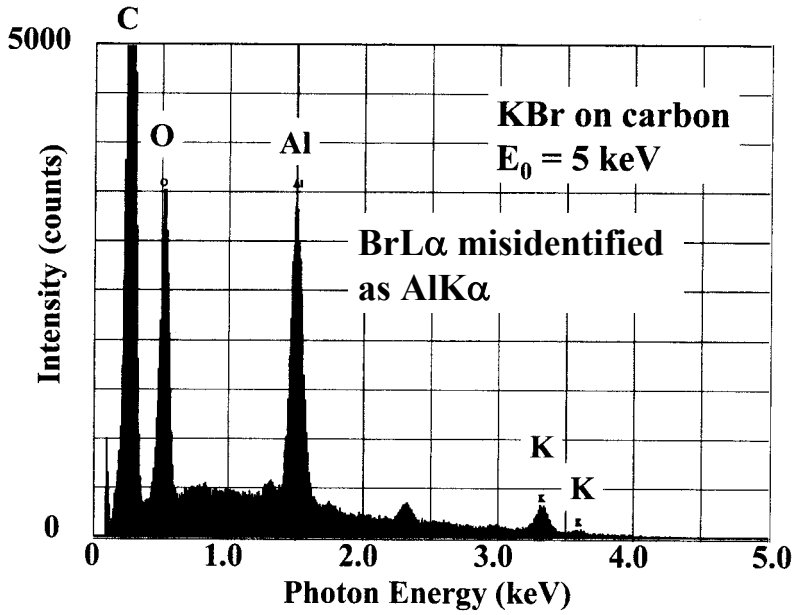

Figure 2. EDS spectrum of KBr particle on C tape with automatic peak identification.

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