XRF Analysis by FP Calibration Using Standards with Unknown Components

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The fundamental parameter (FP) method allows theoretical calculation of X-ray intensities of a sample based on physical and instrument parameters. Since absorption and enhancement effects are considered in the equation, the method has been adopted to perform semi-quantitative analysis over a wide concentration range and many sample types. The FP method is also commonly applied to various fabricated materials such as high alloys and materials for electrical components to achieve high precision analysis.

Theoretical X-ray intensities of bulk samples are calculated based on Sherman's equation. Both nominator and denominator of this equation have terms corresponding to mass fractions, which means that theoretical intensities of elements with fixed mass fraction ratio are identical regardless of mass fraction sum. This is problematic in that theoretical intensity calculation of a sample with unknown components (total known content is less than 100%) yields the same result as the same sample but the known components normalized to 100%. Consequently, when a calibration standard with 98% known total composition is used to set up FP calibrations and the calibration sample is then analyzed as an unknown, the analysis result sum erroneously totals 100%. In contrast, this problem does not occur for empirical calibration method with influence coefficients.

In practice, it can be difficult to find standards with 100% known composition. Typical standards that differ from 100% are for example powders that contains iron as Fe_3O_4 but standard value is given as $T.Fe_2O_3$. Standards may also contain unknown amount of ignition loss, or chemical analysis values for certain components are simply not available. Such samples have been considered less than ideal for use as standards for FP calibration.

To overcome this problem, the theoretical intensity equation has been rearranged taking difference from 100% into account. By using this newly developed method, analysis result sum erroneously yielding 100% is avoided improving accuracy and therefore broadening possible applications of FP method. The method and experimental results for validation will be presented.

References

J. Sherman. Spectrochim. Acta 1,283-306 (1955)