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Guidelines for the preparation of standard routine methods with wavelength-dispersive X-ray fluorescence spectrometry

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Guidelines for the preparation of standard routine methods with wavelength-dispersive X-ray flourescence spectrometry

Table of contents

1 Purpose of the guideline

X-ray Fluorescence Spectrometry (XRF) has been used for several decades as an important analytical tool for production analysis. XRF is characterised by its speed and high precision over a wide concentration range and since the technique in most cases is used as an relative method the limitations are often connected to the quality of the calibration samples. The technique is well established and most of its physical properties are well known.

This guideline is primarily intended to be used for analysis of metal alloys but it is also applicable to other materials although the sample preparation techniques differs. The purpose of this document is to describe general concepts and procedures for calibration and analysis with XRF.

2 Principals of the method

The sample is finished to a clean uniform surface and then irradiated by an X-ray beam of high energy. The secondary X-rays produced are dispersed by means of crystals and the intensities are measured by detectors at selected characteristic wavelengths. The measuring time is set to reach below a specified statistical counting error. Concentrations of the elements are calculated by relating the measured intensities of unknown samples to analytical curves prepared from reference materials of known concentrations

3. Terminology - Definitions

3.1 Calibration

Calculation of the best fit of net intensities and concentrations from a number of calibration samples to an analytical curve.

3.2 Recalibration

Calculation of new calibration constants with a few number of samples, selected from the calibration samples and using the apparent concentrations calculated in paragraph 3.1.

3.3 Standardisation

To compensate for the instruments day to day variation a set of standardisation samples are measured, either one with a low and one with a high concentration for each element (two-point standardisation) or one with a high concentration only for each element (one-point standardisation). The intensities are compared to the initial intensities stored during the calibration procedure and standardisation coefficients are calculated. Calibration constants are not changed.

3.4 Reference measurements

Instead of using net intensities to calculate the concentrations it is sometimes convenient to use the ratios between the intensities for the unknown sample and a reference sample.

Initial intensities for the reference samples are stored during the calibration procedure and the intensities are updated to compensate for day to day variations. The procedure is equivalent to the one-point standardisation procedure in paragraph 3.3.

3.5 Background Equivalent Concentration, BEC

The quantity of analyte which, when subjected to excitation, provides a net intensity equal to the spectral background, see Annex B.

3.6 Limit of Detection, LOD

The minimum concentration at which the signal generated by a given element can be positively recognised above any background signals with a specified degree of certainty, see Annex B.

3.7 Lower Limit of Detection, LLD

The minimum concentration at which the signal generated by a given element can be positively recognised above any background signals with a specified degree of certainty. Calculations based only on the counting statistical error, see Annex B.

3.8 Limit of Quantification, LOQ

The smallest concentration that can be determined with a specified degree of certainty, see Annex B.