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Surface chemical analysis — Total reflection X-ray fluorescence — Principles and general requirements

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Foreword

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This document was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 10, *X-ray Reflectometry (XRR) and X-ray Fluorescence (XRF) Analysis.*

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Through general technical developments, total reflection X-ray fluorescence analysis (TXRF) has been developed into a robust independent physical method of instrumental analytical chemistry. TXRF is a distinct multi-element micro-method and a method of surface analysis. It is used for the simultaneous qualitative detection of the elements and quantification of the element masses. Notably, only small quantities of material are used for the analysis to benefit from all the advantages inherent to the procedure and thus to achieve correct analysis values. TXRF is used alongside other atomic spectroscopic methods, such as atomic absorption spectrometry (AAS) or inductively coupled plasma-based techniques (ICP-AES and ICP-MS) that are also established in the field of element analysis. In addition, TXRF is used in surface analysis and complements techniques such as Auger electron spectroscopy (AES), Rutherford backscattering spectrometry (RBS) or secondary ion mass spectrometry (SIMS). The rapid and in part non-destructive measurement process of a TXRF configuration allows screening or qualitative analysis of unknown flat, film-like and particulate samples and is therefore a valuable addition to other analytical techniques.

The main difference between TXRF and conventional X-ray fluorescence spectrometry (XRF) is the efficient way to excite the material to be examined to fluoresce which is achieved by a special geometrical arrangement in the spectrometer. The spectral background is drastically reduced, the fluorescence intensity increased resulting in a significant enhancement of the signal to noise ratio. Special sample treatment and sample presentation are required for this purpose. If these boundary conditions are met, TXRF achieves a high degree of accuracy and repeatability of the analyses, although a simplified calibration and quantification is used. The detection limits currently achievable with TXRF are approximately 10^{-12} g or 10^{10} atoms/cm^2 and are thus orders of magnitude lower than for XRF. The relative element concentrations are, depending on the matrix or the main components of the sample and for the elements to be examined, between 0,1 % and up to 10^{-9} for the low concentrations.

This document describes the physical principles and instrumentation required for TXRF analysis assuming that the conditions for total reflection are met, without referring to the samples' specificity. As a demonstration of the good reproducibility of the TXRF instruments, data from a round-robin test performed using centrally prepared and optimized samples are presented (see Annex B).

Other standards focussing on specific case studies and procedures are available for silicon wafers (see ISO 14706 and ISO 17331), environmental and biological samples (see ISO/TS 18507), and water (ISO 20289).