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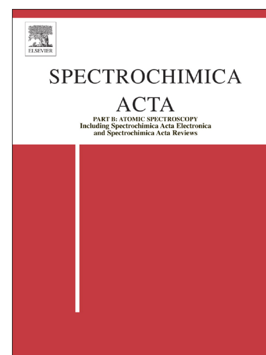

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PII: S0584-8547(18)30059-4
DOI: doi:10.1016/j.sab.2018.07.008
Reference: SAB 5483

To appear in: *Spectrochimica Acta Part B: Atomic Spectroscopy*

Received date: 30 January 2018
Revised date: 5 July 2018
Accepted date: 5 July 2018

Please cite this article as: Hikari Takahara, Atsushi Ohbuchi, Kensuke Murai , Method development for the analysis of poorly soluble solids by total reflection X-ray fluorescence spectrometry. Sab (2018), doi:10.1016/j.sab.2018.07.008

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Method Development for the Analysis of Poorly Soluble Solids by Total Reflection X-ray Fluorescence Spectrometry

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Keyword: trace element analysis; suspension; matrix effect; sample shape effect; scattered radiation internal standard method

Highlights:
Silicon nitride powders suspended in polymer solution was spin-coated on substrate.
Correlation of net intensities to certified values for impurity elements is not good.
The background intensity ratio effectively improved the correlation coefficient.
Scattered radiation internal standard method correct matrix effect and sample shape effect.

Abstract

Non-dissolution preparation method was developed for powder sample analysis by total reflection X-ray fluoresce spectrometry (TXRF). The certified reference materials (CRMs) of silicon nitride powder were suspended in polymer solution and the suspension was spin-coated on substrate. The fine silicon nitride particles dispersed on a few tens of nm thin polymer film were observed by surface profiler and scanning electron microscope (SEM) observation. For spin-coated specimens, TXRF results show not good correlation between the X-ray intensities and standard values for impurity components Cr, Mn, and Fe in silicon nitride powder CRMs. However, taking background ratio can effectively improve the correlation factor by correcting matrix effect and physical characteristics such as surface condition, grain size, and irregular sample shape.

1. Introduction

The incident X-ray is totally reflected on a flat polished surface of a substrate at glancing angle below the critical angle. The X-rays penetrate the substrate only for a small portion and fluorescence X-ray from trace amount on the surface can be detected. The advantage of the low penetration depth under TXRF condition is widely applied for trace analysis of liquids and suspensions. Micro-droplet of liquid sample is pipetted on a reflector

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