

Identification and Remedy of Spectral Interference in Crystalline Silica Analysis by X-ray Diffraction Method (Modified NIOSH 7500)

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Introduction

Crystalline silica analysis by XRD (X-ray Diffraction) method utilizes the relative intensity of crystalline silica-specific peaks in the angular-dispersive XRD spectrum. However, when other crystalline compounds are present in the sample, it is possible that some of the XRD peaks from these compounds overlap with those of crystalline silica and contribute towards the measured peak intensities. When left unchecked, this type of spectral interference can cause an overestimate of the crystalline silica contained in the sample or even a false-positive result. Some examples of crystalline mineral/compounds that pose interferences to crystalline silica include barite, micas (muscovite, biotite), potash, feldspars (microcline, plagioclase), montmorillonite, sillimanite, zircon, graphite, iron carbide, clinoferrosilite, wollastonite, sanidine, leucite, orthoclase, and lead sulfide.

Identification and Remedy of Spectral Interferences

When XRD peaks are detected at the angular positions for crystalline silica, the sample should be subjected to further verification scans, such as a wide range XRD scan. The spectrum from this wide range scan will be used to verify the presence of crystalline silica and identify the known interferences. When the interfering compounds are identified, the crystalline silica peaks that do not overlap with those of the interferences should be selected further analysis. In order to achieve the highest analytical sensitivity, the next available strongest peak should always be used. For example, the primary peak should be used if no interference on it is found, or the secondary peak should be used if the interference is determined or suspected on the primary peak but not the secondary peak.

It is not always possible to identify the interfering compounds. If the interferences can not be identified from a wide range scan, the intensity ratios among the crystalline silica peaks should be used to either verify the non-existence of interferences or determine the existence of suspected interferences. In case that the wide range scan is not sufficiently sensitive for the measurement of lower-intensity silica peaks, narrow range long time scans should be carried out for those peaks. The peak intensity ratios so obtained will be compared to the ratios established theoretically or experimentally, for example, the peak intensity ratios published in the ICDD-PDF database. A substantially different peak ratio indicates the presence of interferences, and the crystalline silica peaks with suspected interferences should be avoided. For example, if the measured ratio between the primary and secondary peak intensity is much higher than the established ratio, then interferences on the primary peak are suspected, and the secondary peak intensity should be used for quantification.

In rare circumstances where interferences are found or suspected on all the available crystalline silica peaks, a best-effort-based measurement should be performed using a peak deconvolution approach, and the report should note the potential adverse effect on the accuracy of the analysis caused by such an approach.

References

NIOSH Manual of Analytical Methods (NMAM), Fourth Edition