

Application News

No. X257

X-ray Analysis

High-Speed X-Ray Diffraction Analysis of Asbestos (Chrysotile) Using Wide-Range High-Speed Detector "OneSight"

Introduction

Asbestos is a material that has long been widely used as a building material due to its excellent heat and acid resistance. However, its use has gradually been banned because of widespread recognition of its adverse health effects. Due to revisions of Japan's Industrial Safety and Health Act enforcement ordinance and Ordinance on Prevention of Asbestos Hazards in 2006, the regulatory limit value for asbestos content became more stringent. Accordingly, the current content by weight of asbestos in any product is specified to be less than 0.1 %. As X-ray diffraction has been adopted as the official method for measurement of asbestos content, the two official methods specified for its analysis in Japan are JIS A 1481-2: Determination of asbestos in building material products – Part 2: Sampling and qualitative analysis for judgment of existence of containing asbestos, and JIS A 1481-3: Determination of asbestos in building material products – Part 3: Quantitative analysis of containing asbestos by X-ray diffraction method. Here, using the new wide-range, high-speed detector OneSight, we introduce an example of high-speed quantitative analysis of the chrysotile, one kind of asbestos.

Wide-Range High-Speed Detector "OneSight"

The wide-range, high-speed detector OneSight consists of a semiconductor sensor array with more than 1000 channels. It achieves sensitivity greater than twenty times that possible with existing scintillation detectors, while providing high-speed measurement. Quantitative analysis can be conducted very quickly using the "One Shot" mode, in which the detector is focused on one peak using a wide acquisition angle.



Fig. 1 XRD-7000 Equipped with OneSight

Analysis of Asbestos

Quantitative analysis of asbestos is conducted using the base standard absorption correction method. Here, asbestos that was subjected to formic acid treatment was collected onto a filter and placed on a base standard Zn plate, and then measured. The measured diffraction intensity was corrected using the transmitted Zn diffraction intensity, and a calibration curve was generated. The lower limit of quantitation was then determined.

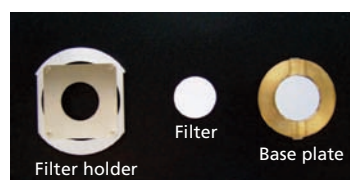


Fig. 2 Shimadzu Proprietary Holder for Environmental Quantitative Analysis

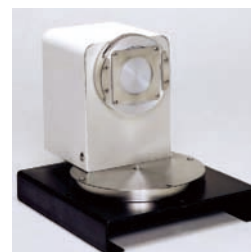


Fig. 3 Rotation Sample Attachment (for Environmental Quantitation)

Table 1 Asbestos Calibration Curve Generation Flow

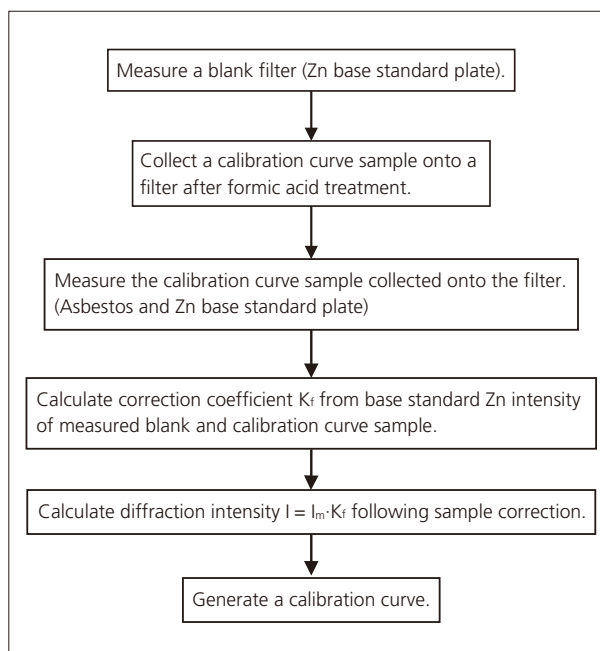


Table 2 Analytical Conditions

Instrument	XRD-6100
X-Ray Tube	Cu target
Tube Voltage	40 kV
Tube Current	30 mA
Monochromation	Ni filter
Integration Time	120 seconds
Rotation Speed (rpm)	60 rpm
Detector	OneSight wide-range, high-speed detector
Measurement Mode	"One Shot" mode

Standard sample: JAWE 111 chrysotile
 Sample concentrations: Blank, 0.05 mg, 0.1 mg, 0.3 mg, 0.5 mg, 1 mg, 3 mg, and 5 mg

Calibration Curve

Measurement of the sample used for generating a calibration curve was conducted using the One Shot mode of the OneSight wide-range, high-speed detector. This mode eliminates the need for scanning with a goniometer, and permits high-speed measurement by fixing on the target peak position to obtain adequate intensity. In this measurement, the measurement time was 120 sec for the asbestos peak, and 120 sec for the Zn peak.

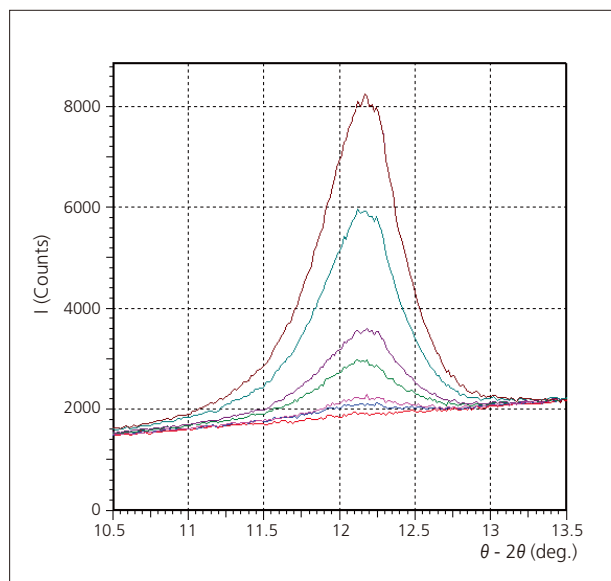


Fig. 4 Peak Profiles of the Calibration Curve Samples (Chrysotile 0.05 mg, 0.1 mg, 0.3 mg, 0.5 mg, 1 mg, 3 mg, and 5 mg)

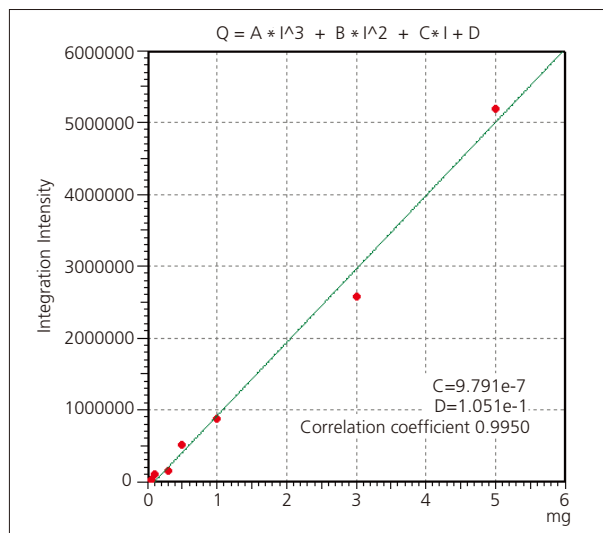


Fig. 5 Calibration Curve of Chrysotile over 120 sec Integration Time

Table 3 Comparison of Lower Limit of Detection Using OneSight and Scintillation Detector

Measurement Mode	One Shot Mode Measurement Using OneSight (120 sec Integration Time)	Measurement Using Scintillation Detector* (1000 sec Integration Time)
Lower Limit of Detection	0.0034	0.0045
Lower Limit of Quantitation	0.0102	0.0135

(Unit: mg)

Accuracy comparable to that obtained using the quantitative analysis measurement conditions (1/8 ° – 1/16 °/min: 1000 second integration using the Bragg-Brentano optics) when conducted as prescribed by JIS A 1481-3 was obtained by the 120-second measurement time that was used with the wide-range, high-speed OneSight detector permits measurement to be completed in an extremely short period of time.

* The analytical conditions using the scintillation detector were as follows.

- Instrument : XRD-7000
- Monochromation : Curved graphite monochromator
- Current and Voltage : 40 kV and 30 mA
- Measurement Time : 1000 sec measurement time (Asbestos and Zn, respectively)
- Slit : DS 1 degree, SS 1 degree, RS 0.3 mm
- Rotation Speed : 60 rpm