Application note | AN41514

Analysis of chrysotile asbestos in air filters using ARL X'TRA Companion X-ray Diffractometer

Authors

Dr. Simon Welzmiller, Ju Weicai,
Application Specialists XRD

Introduction

X-ray diffraction (XRD) is a powerful analytical technique used to identify and quantify crystalline solid materials and therefore is well suited to analyze asbestos fibers in air filters. XRD works by directing X-rays at a sample and measuring the angles and intensities of the diffracted beams. Each type of asbestos mineral produces a unique diffraction pattern, allowing for precise identification and quantification. This method is highly sensitive and can distinguish asbestos from other fibrous materials, making it invaluable for environmental monitoring and ensuring compliance with safety regulations. XRD is especially useful in complex matrices where other methods might fail to differentiate asbestos fibers.

Regulatory control of asbestos concentrations is stringent due to its severe health risks, including lung cancer and mesothelioma. Occupational exposure limits are set by agencies such as OSHA (Occupational Safety and Health Administration – US), which mandates a permissible exposure limit of 0.1 fibers per cubic centimeter of air. For asbestos analysis in air filters, norms specify methods and limits of detection. Most used methods apply polarized light (PLM) and electron microscopy with LoDs ranging from 0.001 to 0.01 fibers per cubic centimeter of air (f/cc). Chinese norm GBT 23263 mandates the use of filter membranes for very low asbestos quantities to allow for concentrating of fibers followed by XRD analysis.

Instrument & software

The Thermo Scientific™ ARL™ X'TRA Companion X-ray Diffractometer (c.f. Figure 1) is a simple, easy-to-use benchtop XRD instrument for routine phase analysis as well as more advanced applications. The ARL X'TRA Companion XRD uses a θ/θ goniometer (160 mm radius) in Bragg-Brentano geometry coupled with a 600 W X-ray source (Cu or Co). The radial and axial collimation of the beam is controlled by divergence and soller slits, while air scattering is reduced by a variable beam knife. An integrated water chiller is available on demand. Thanks to the innovative solid state pixel detector (55 x 55 µm pitch), the ARL X'TRA Companion XRD provides very fast data collection and comes with one-click Rietveld quantification capabilities and automated result transmission to a LIMS (laboratory information management system).

Figure 1: ARL X'TRA Companion X-ray diffraction system.

Experimental

Quartz fiber air filters spiked with 10, 20, 30, 50, 100, 200, 300, 500 and 1000 µg Cchrysotile were measured for 15 minutes. The samples were loaded in special air filter sample cups and measurements from 11.0 to 13.5°2θ (Cchrysotile 002 reflection) were performed in reflection mode using Cu Kα (1.541874 Å) radiation with sample spinning. (c.f. Figure 2). A single peak fit in Profex software was carried out to build a linear regression (samples 10, 20, 30, 50, and 100 µg) using the integral intensities. The range till 100 µg was chosen because it reflects actual concentration ranges the best and allows calculating sound statistical values. To extend the calibration range, a second linear regression could be calculated for higher concentrations. The standard error of estimate (SEE or $S_y = \sqrt{\frac{\sum(y_i-y_i)^2}{n-2}}$ with y_i = observed values and $\hat{y}_i$=predicted values) and limit of detection ($LoD = \frac{3*Sy}{b_i}$) was calculated from the linear regression.

Results & Discussion

Performing a linear regression (Figure 3) of the 10 – 100 µg samples result in a linearity R^2 = 0.992 with a SEE = 3.9 µg and LoD = 11.9 µg. Assuming sampling flow rates of 1-2 m^3 through the filters with average fiber dimensions of 8 x 0.02 µm, will result in a required LoD between ~7 – 60 µg of chrysotile fibers in the air filter depending on the norm. Comparing these values with the LoD determined from the linear regression, it is apparent that XRD can be a highly sensitive complementary method to microscopy, or in case of the GBT 23263 norm satisfy the requirements.

Your Benefits

The ARL X'TRA Companion XRD yields data perfectly suited to quantify chrysotile asbestos in air filters. Utilizing a calibration curve in a one-click refinement yields results in minutes, enables ease-of-use for operators and reliefs training constraints. XRD allows quick screening of air filter samples to comply with regulatory requirements.

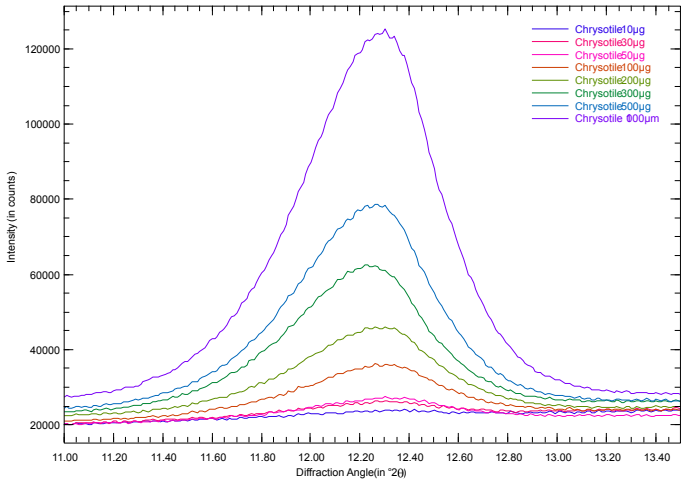

Figure 2: XRD patterns of 10, 20, 30, 50, 100, 200, 300, 500 and 1000 µg chrysotile samples (11.0 to 13.5°2θ; 15 minutes).

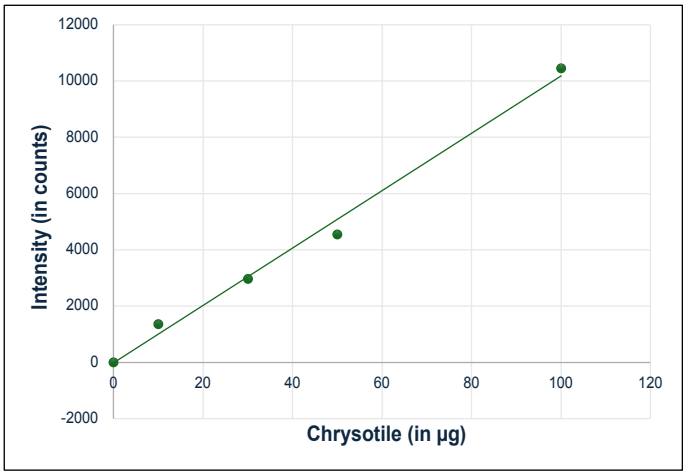

Figure 3: Linear correlation between the reference and values determined by XRD of 10, 20, 30, 50, 100 µg chrysotile samples.

Learn more at **www.thermofisher.com/xtra**

thermo scientific