MATERIALS ANALYSIS

QUANTITATIVE DETERMINATION OF COMMON TYPES OF ASBESTOS BY DIFFUSE REFLECTANCE FTIR

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Solution Note

Materials

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Abstract

The high infrared power provided by the Agilent Cary 660 FTIR allows the use of Diffuse Reflectance as sampling technique for qualitative and quantitative analysis of common types of asbestos like Chrysotile, Crocidolite and Amosite. Diffuse reflectance achieves good reproducibility with fast sample preparation, minimizing exposure to harmful asbestos during the analysis.

Introduction

Asbestos is the name for several fire-proof minerals that belong to the silicate class. There are two groups: serpentines and amphiboles.

Infrared spectrometry is one of the official methods for the quantitative determination of asbestos. Historically, the method used is the traditional transmission technique on KBr pellets. There are two main limitations of this method. The first limitation involves the potential uncertainty of the thickness of the pellet, which, as established by the Lambert-Beer law, is linearly related to concentration. The other limitation is the long preparation methodology, which requires grinding the sample down to 10 microns. Using this method, the average time taken to grind the sample down can be more than 10 minutes and, consequently, the operator can face a high risk of exposure.

The high power of the Agilent Cary 660 FTIR spectrometers allows the diffuse reflectance sampling technique to be used. Diffuse reflectance FTIR spectroscopy consists in analyzing the diffuse radiation from a sample mixed with KBr and placed on a flat surface in a sample holder with the appropriate reflectance accessory.

Diffuse reflectance offers two fundamental advantages in quantitative analysis compared to the pellet transmission technique. First and foremost, the technique provides greater accuracy as there is no intrinsic uncertainty of the pellet thickness. Secondly, the preparation times required are decidedly shorter given that the sample only needs to be ground down to about 40 microns thus reducing preparation time to about 2 minutes. In addition, there is no need to use pellet grinders and presses — the sample is simply placed inside the sample holder. Total preparation and analysis time is therefore reduced to a few minutes, and the risk of asbestos exposure for the operator is substantially reduced.

Experimental

Sample preparation

For the analysis to be successful, it is essential that a representative quantity of the sample is homogenized. This homogenization can be carried out by coarse grinding in a large agate mortar. The ground sample is then mixed with KBr for infrared spectrometry at a ratio of 1:4; normally, 40 mg of sample is weighed and mixed with 160 mg of KBr. The mixture is then further homogenized and ground in an agate mortar and placed in a 4.7 mm diameter sample holder of an Easydiff reflectance accessory (Pike Technologies, USA).

Parameter	Value
Instrument	Agilent Cary 660
Source	Ceramic Duraglow™
Beam Splitter	Extended range KBr
Detector	DLaTGS Peltier
Scans	16
Resolution	4 cm^{-1}
Unit	Kubelka Munk
Velocity	5 KHz
Apodization	Blackman-Harris
Symmetry	Asymmetrical

Table 1: Parameters used to obtain a blank KBr spectrum.

Typical spectra and peaks

Three common types of asbestos were analyzed. Chrysotile, or white asbestos, the name 'chrysotile' comes from the Greek for 'gold fiber'. It is a serpentine phyllosilicate $Mg_3(Si_2O_5)(OH)_4$ of the triclinic pedial class. The typical peak position for quantitative determination of chrysotile, in compliance with current regulations, is the SiOH stretching band at 3680 cm^{-1} (Figure 1).

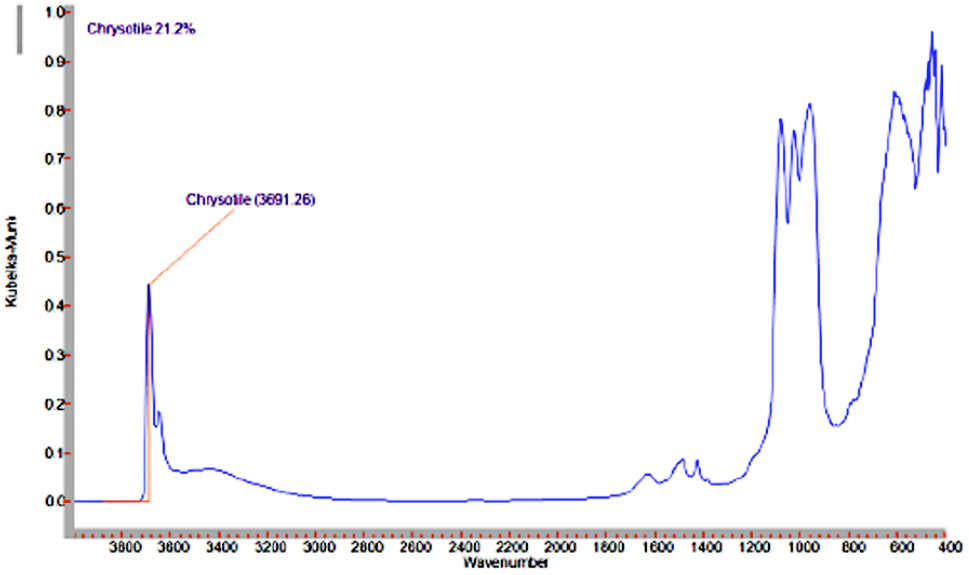

Figure 1: Chrysotile spectrum.

Crocidolite, or blue asbestos. The name 'crocidolite' comes from the Greek for 'wool flake'. It is an inosilicate amphibole, a fibrous variety of the mineral riebeckite $Na_2Fe_3Fe_3+2(Si_8O_{22})(OH,F)_2$ of the monoclinic prismatic class. The typical peak position for the quantitative determination of crocidolite, in compliance with current regulations, is the SiO stretching band at 770 cm^{-1} (Figure 2).

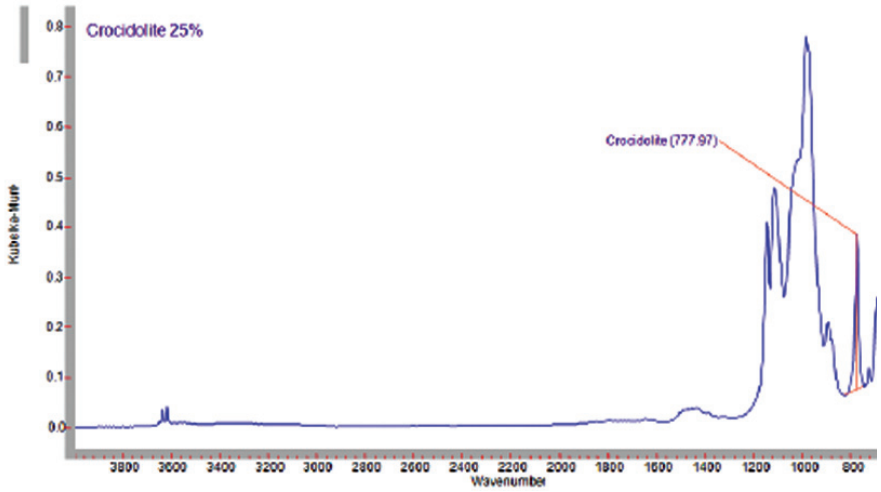

Figure 2: Crocidolite spectrum.

Amosite, or brown asbestos. The name 'amosite' derives from the acronym for 'asbestos mines of South Africa'. It is the commercial name for grunerite and cummingtonite. It is an inosilicate amphibole $(Mg,Fe)_7(Si_8O_{22})(OH)_2$ of the monoclinic prismatic class. The typical peak position for the quantitative determination of amosite, in compliance with current regulations, is the SiO stretching band at 1080 cm^{-1} (Figure 3).

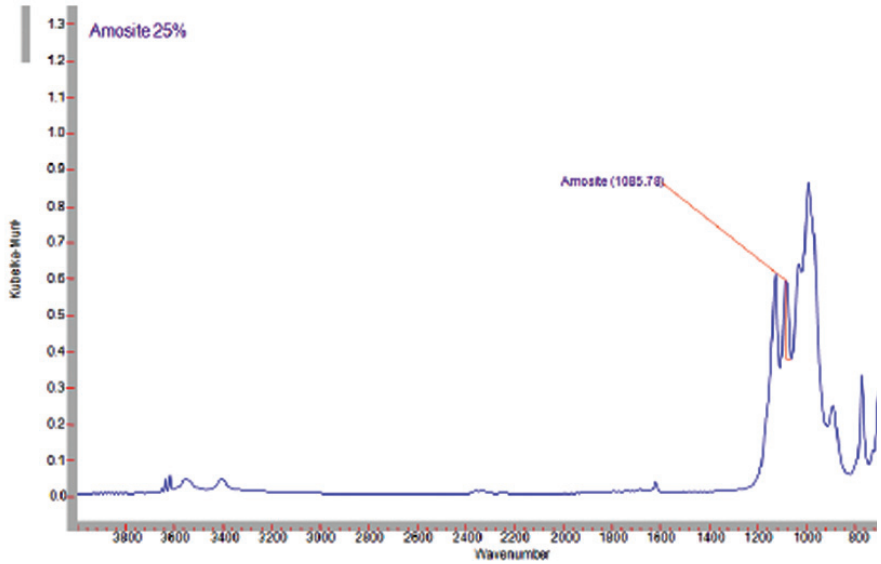

Figure 3: Amosite spectrum.

Calibration

Calibration for the three types of asbestos is carried out as previously described. The concentrations of the standard mixtures have been expressed taking into account that the dilution of the sample in KBr is maintained constant at 20 %.

Chrysotile. 10 mg of standard NIST SRM 1866a chrysotile previously ground in a ceramic mortar are quantitatively mixed with 190 mg of KBr and homogenized in an agate mortar. This mixture represents the standard mixture at 25 %.

Then a series of further dilutions are carried out at a ratio of 1:1 in KBr in order to successively create the following standards respectively at 25 % 12.5 %, 6.25 % 3.125 %, 1.56 %, 0.78 %, 0.39 %.

Calibration is carried out on the standards underlined with a peak position at 3680 cm^{-1} at peak height with an average baseline of between 3975 cm^{-1} and 3790 cm^{-1}. The calibration curve obtained is shown in Figure 4.

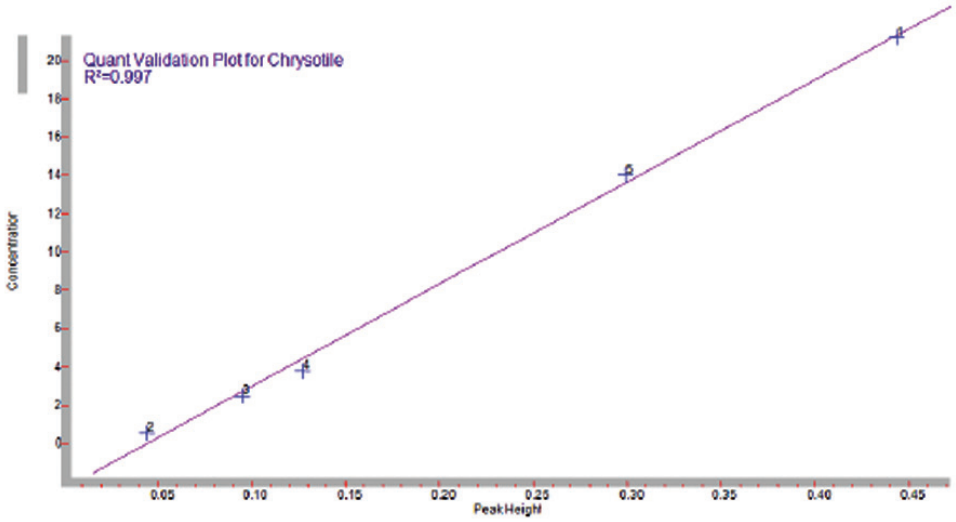

Figure 4: Chrysotile calibration line.

Crocidolite. 10 mg of standard NIST SRM 1866a crocidolite previously ground in a ceramic mortar are quantitatively mixed with 190 mg of KBr and homogenized in an agate mortar. This mixture represents the standard mixture at 25 %.

Then, after analysis, a series of further dilutions are carried out at a ratio of 1:1 in KBr in order to successively create the following standards respectively at 25 % 12.5 %, 6.25 % 3.125 %, 1.56 %, 0.78 %, 0.39 %.

Calibration is carried out on the standards underlined with a peak position at 775 cm^{-1} at peak height with a baseline of between 785 cm^{-1} and 825 cm^{-1}. The calibration curve obtained is shown in Figure 5.

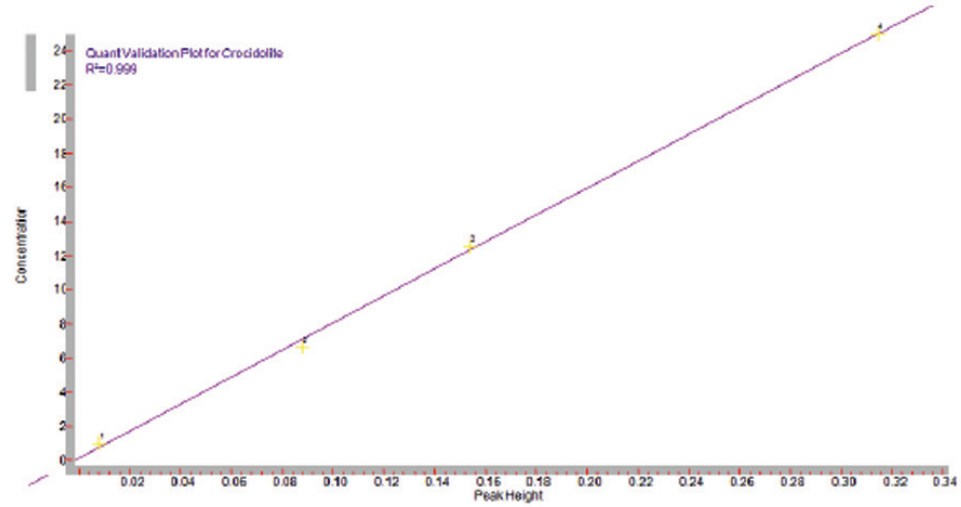

Figure 5: Crocidolite calibration line.

Amosite. 10 mg of standard NIST SRM 1866a amosite previously ground in a ceramic mortar are quantitatively mixed with 190 mg of KBr and homogenized in an agate mortar. This mixture represents the standard mixture at 25 %. Then, after analysis, a series of further dilutions are carried out at a ratio of 1:1 in KBr in order to successively create the following standards respectively at 25 %, 12.5 %, 6.25 %, 3.125 %, 1.56 %, 0.78 %, 0.39 %.

Calibration is carried out on the standards underlined with a peak position at 1080 cm^{-1} at peak height with a baseline at 1062 cm^{-1}. The calibration curve obtained is shown in Figure 6.

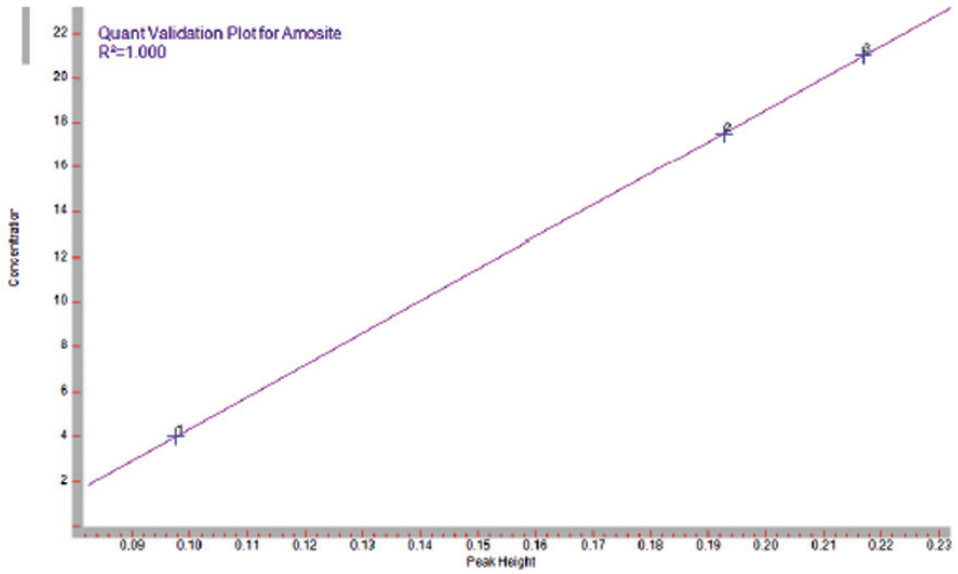

Figure 6: Amosite calibration line.

Analysis of a real sample

A sample of building sediments made up of cement and potentially of types of asbestos was prepared using the method previously described. The spectrum obtained is shown in Figure 7.

The presence of the chrysotile peak at 3680 cm^{-1} can be immediately identified, but no other peaks can be seen for crocidolite and amosite. Quantitative analysis was carried out on the curve previously obtained for chrysotile.
The quantitative result was 1.7 % of chrysotile.

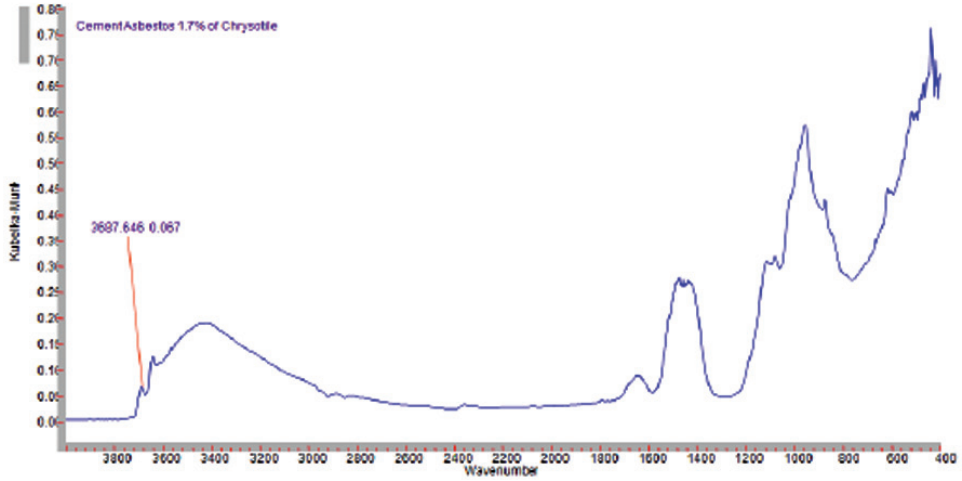

Figure 7: Spectrum of building sediment sample.

Instrumentation
Agilent Cary 660 FT-IR

Conclusion

Quantitative analysis of types of asbestos using the FTIR diffuse reflectance method offers these advantages:

- Fast sample preparation.
- Reduced risk of exposure for the operator.
- Good accuracy of measurements.
- Detection limits in compliance with current regulations.

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The Measure of Confidence

Agilent Technologies