MATERIALS ANALYSIS

QUANTITATIVE DETERMINATION OF COMMON TYPES OF ASBESTOS BY DIFFUSE REFLECTANCE FTIR USING THE AGILENT CARY 630 SPECTROMETER

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Materials

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Abstract

The Agilent Cary 630 FTIR spectrometer allows the use of the Diffuse Reflectance sampling technique for qualitative and quantitative analysis of common type of asbestos such as Chrysotile and Crocidolite. Diffuse Reflectance offers the benefits of better reproducibility with faster sample preparation, whilst reducing operator exposure to harmful asbestos.

Introduction

Asbestos is the collective 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 types of asbestos. Historically, the method was based on the traditional transmission technique using potassium bromide (KBr) pellets, but there are two main limitations of this method. The first limitation relates to the potential uncertainty in the thickness of the pellet which, as established by the Beer-Lambert law, is linearly related to concentration. The other limitation is the time-consuming preparation methodology, which requires grinding the sample down to a particle size less than 10 microns. Using this approach, the average time taken to grind the sample down can be more than 10 minutes and consequently, there is a high risk of exposure to the operator.

The Agilent Cary 630 FTIR spectrometer allows the Diffuse Reflectance sampling technique to be used. Using this approach, the sample is ground and mixed with KBr powder and then the loose powder is transferred to a sample cup and inserted into the Diffuse Reflectance Accessory (DRA). IR light is then directed onto the sample and the diffusely scattered radiation emanating from the sample is collected and directed to the spectrometer detector (see Figure 1).

Diffuse reflectance offers two fundamental advantages for quantitative analysis compared to the pellet transmission technique. First and foremost, the technique provides greater accuracy as there is no intrinsic uncertainty related to the pellet thickness. Secondly, the sample preparation time is considerably reduced and simplified, because the sample only needs to be ground to around 40 micron particle size, and the pelletisation step is completely removed. Total preparation and analysis time is therefore reduced to only a few minutes, and the risk of asbestos exposure to the operator is substantially reduced.

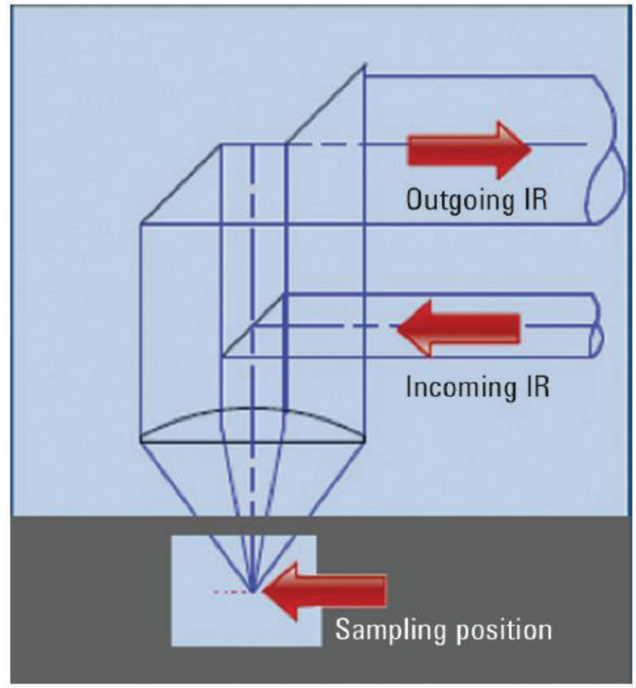

Figure 1. Optical diagram of the Agilent Cary 630 FTIR DRA.

Experimental

Sample preparation

For the analysis to be successful, it is essential that a representative quantity of the sample is homogenised. This homogenisation step is carried out by coarse grinding in a large agate mortar. In readiness for infrared spectrometry, this ground sample is then mixed with KBr in a 1:4 ratio (40mg of sample accurately weighed and mixed with 160mg KBr), and the mixture is further ground and homogenised in an agate mortar before being transferred to the DRA sample holder. Prior to analysis of the sample a background spectrum is recorded using the diffuse gold mirror supplied with the DRA.

Parameter	Value
Instrument	Agilent Cary 630
Spectral Range	4000 - 600cm-1
Optics	ZnSe
Detector	DTGS
Scans	128
Resolution	4 cm^{-1}

Table1. Instrument operating parameters.

Two common types of asbestos were analysed, Chrysotile and Crocidolite.

Chrysotile (white asbestos) gets it's name from the Greek for "gold fibre". It is a serpentine phyllosilicate $Mg_3(Si_2O_5)(OH)_4$ of the monoclinic or orthorhombic classes.

Current regulations stipulate that quantitative IR determination of Chrysotile is based on measurement of the SiOH stretching band around 3680 cm-1 (see Figure 2).

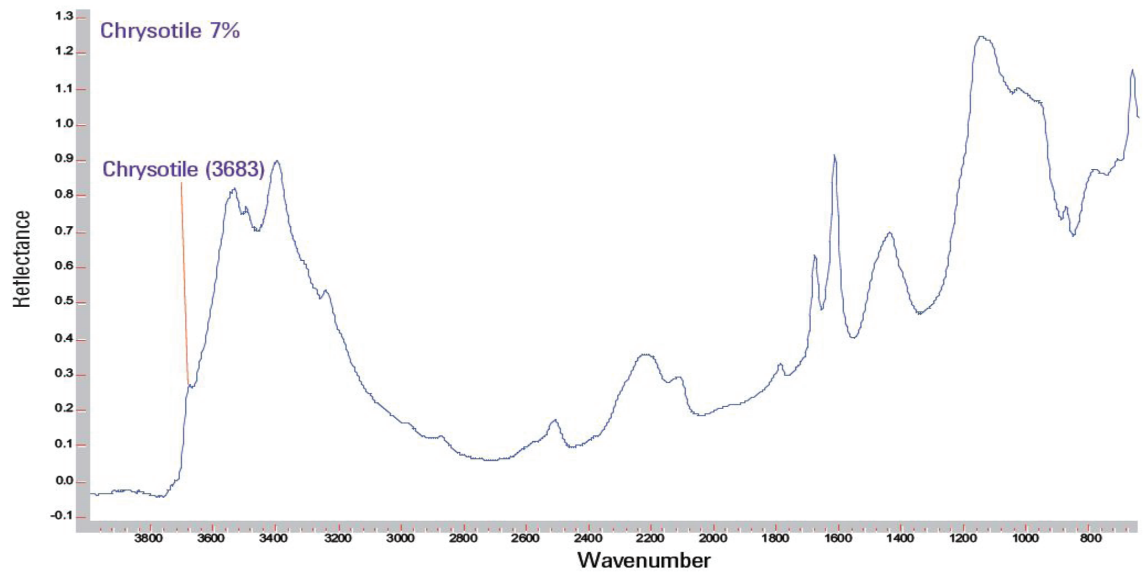

Figure 2: Spectrum of Chrysotile in Gypsum matrix

Crocidolite (blue asbestos) derives it's name from the Greek for "wool flake". It is an inosilicate amphibole; a fibrous variety of the mineral Riebeckite $Na_2Fe_3^{2+}Fe_2^{3+}Si_8O_{22}(OH)_2$ 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 around 770 cm^{-1} (Figure 3).

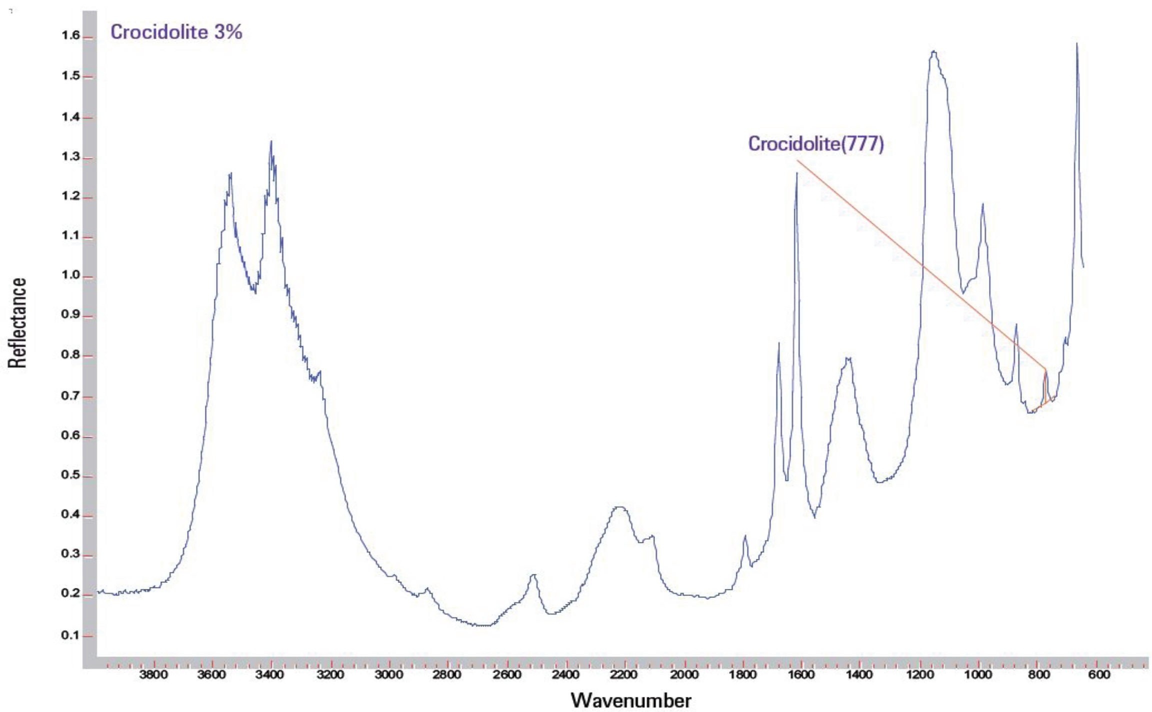

Figure 3: Spectrum of Crocidolite in Gypsum matrix

Calibrations

Calibrations for these two common asbestos types were prepared using the sample preparation regime described earlier.

Chrysotile Calibration

40 mg of Standard RTI Int. 7% Chrysotile in Gypsum, previously ground in a ceramic mortar, was mixed with 160 mg of KBr and homogenised in an agate mortar. This mixture represents the standard mixture at 7%. A series of further dilutions were carried out at a ratio of 1:1 in KBr in order to create standards at 3.5% and 1.75%. The calibration curve is derived from measurement of the peak area between 3747cm-1 and 3674cm-1 using a single baseline position averaged over the range from 3975 - 3790cm-1. The calibration curve obtained is shown in Figure 4.

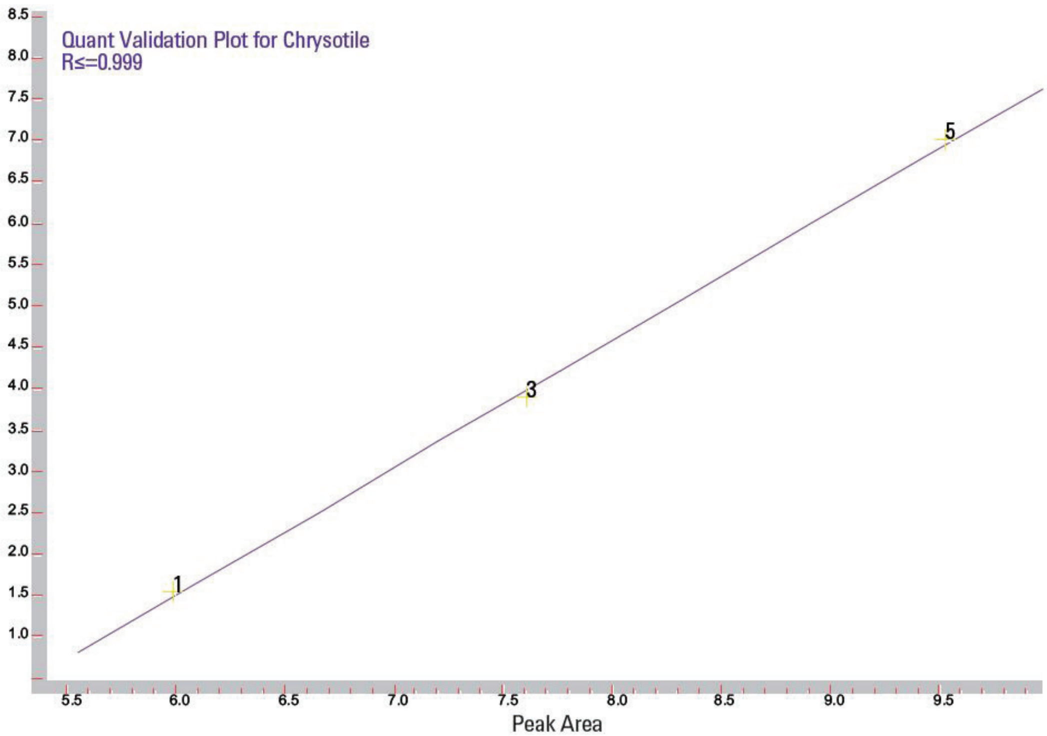

Figure 4. Chrysotile calibration curve.

Crocidolite Calibration

40 mg of Standard RTI Int. 3% Crocidolite in Gypsum, previously ground in a ceramic mortar, was mixed with 160 mg of KBr and homogenised in an agate mortar. This mixture represents the standard mixture at 3%. Then, after analysis, a series of further dilutions were carried out at a ratio of 1:1 in KBr in order to create standards at 1.5% and 0.75%. The calibration curve was derived using measurement of peak height at 775cm-1 with a single baseline averaged over the range from 785 - 825cm-1. The calibration curve obtained is shown in Figure 5.

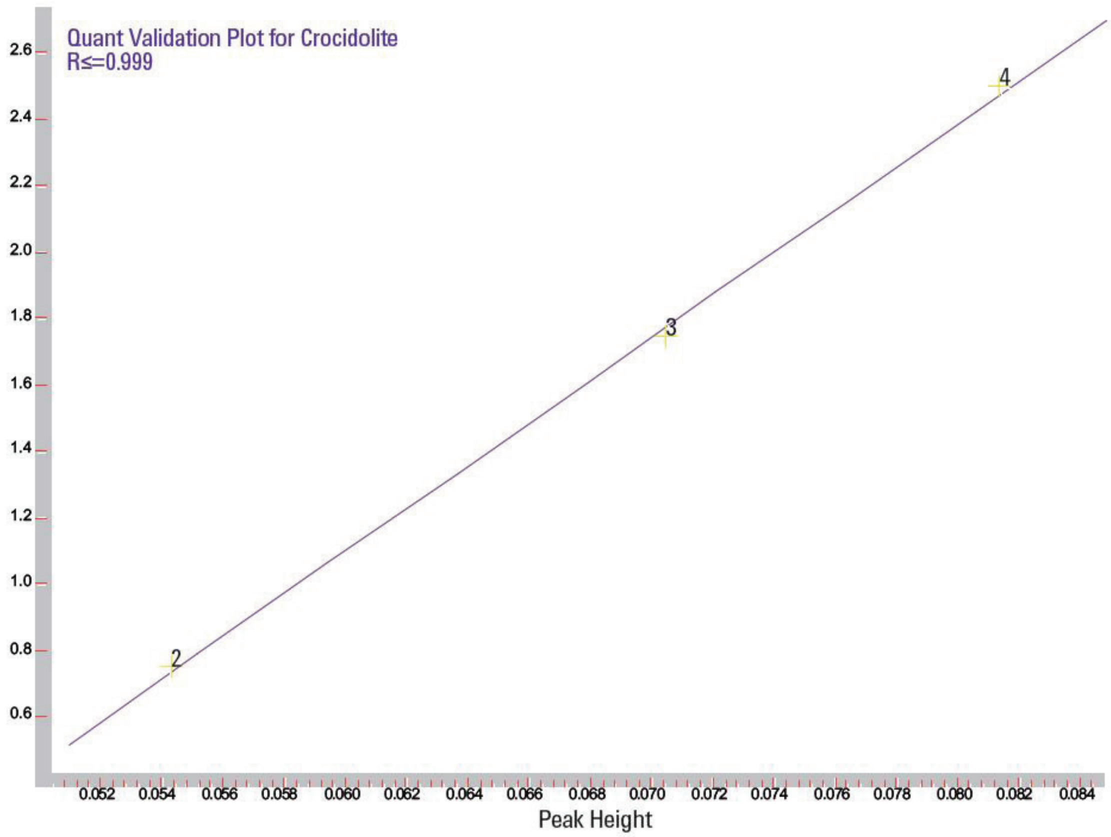

Figure 5. Crocidolite calibration curve.

Analysis of a real sample

A sample of building sediment made up of cement and potentially different types of asbestos was prepared using the sample preparation technique. The spectrum obtained is shown in Figure 6.

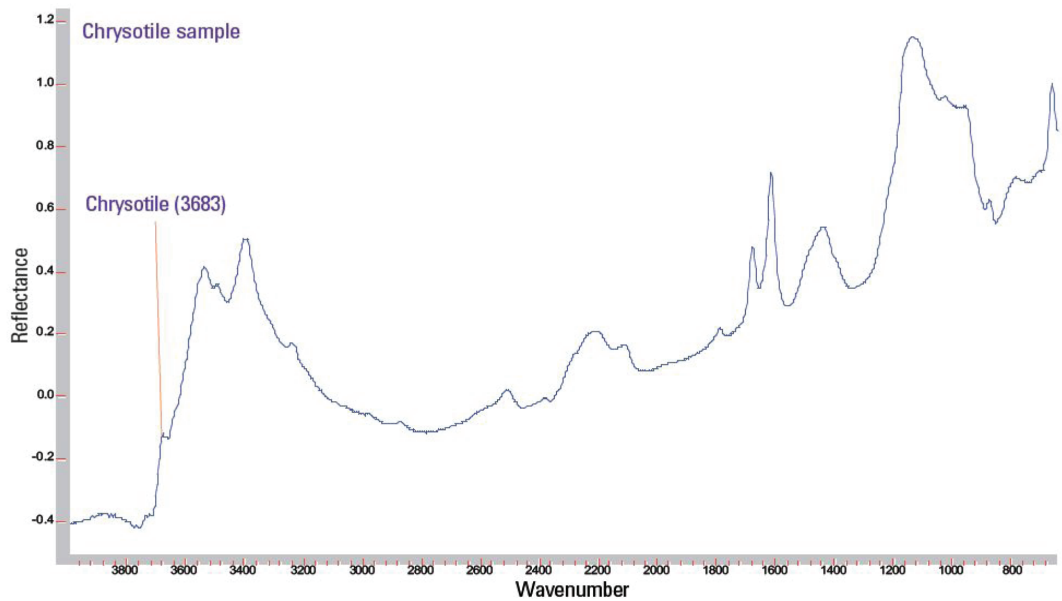

Figure 6. Spectrum of building sediment sample.

The presence of the Chrysotile peak at 3680 cm^{-1} can be immediately identified, but the spectrum shows no evidence of the characteristic peak associated with the presence of Crocidolite. Using the previously derived calibration, the predicted concentration of Chrysotile in this sample was 3.9%.

Conclusion

Quantitative analysis of different types of asbestos using the Cary 630 Diffuse Reflectance method offers the following advantages:

- Fast sample preparation
- Reduced risk of exposure to the operator
- Good measurement accuracy
- Detection limits in compliance with current regulations
- Low instrumentation costs

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