MATERIALS ANALYSIS

RAPID ANALYSIS OF MINERALS USING THE AGILENT CARY 630 FTIR WITH DIAMOND ATR

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Solution Note

Geochemistry, Minerals and Mining

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Abstract

This report details how the Agilent Cary 630 FTIR spectrometer equipped with monolithic design diamond Attenuated Total Reflectance (ATR) accessory can be used for simple, rapid measurement of mineral samples. The resulting spectra can be used to qualitatively identify the mineral type by cross-reference against a library of spectra.

Introduction

Fourier-Transform Infra-Red (FTIR) spectroscopy is widely used to characterize minerals. It is a complimentary technique to X-Ray Diffraction (XRD) because it has the advantage of being able to identify poorly crystalline "amorphous" materials which is a limitation of XRD. Historically, minerals analysis by FTIR has been achieved using "classical" approaches such as preparation of a potassium bromide (KBr) disk or Diffuse Reflectance (DRIFTS). These techniques are well suited to the analysis of such inorganic materials because many of the important absorbance bands found in minerals such as Si-O-Si and Si-O-Al appear below 600 cm^{-1} so it's important to use an approach that makes full use of the complete Mid-IR spectral range extending down to 400 cm^{-1}. However, the main problem with these techniques is that the sample preparation stage is both tedious and time-consuming, resulting in low measurement throughput. A much faster and more convenient approach is to use a "monolithic" diamond Attenuated Total Reflectance (ATR) accessory [1]. This type of ATR offers full spectral range analysis, and enables the direct measurement of samples without any lengthy preparation step. In this application note, we will show how the high sensitivity and wide wavelength range of the Agilent Cary 630 FTIR with diamond ATR enables high quality data to be collected from a range of mineral samples with a measurement time of only 15 seconds per sample.

Experimental

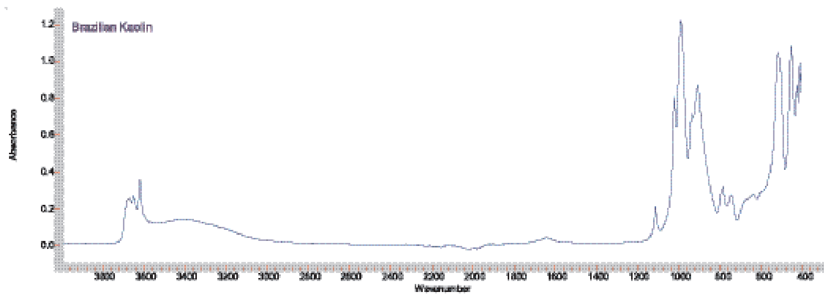
All samples were received as coarsely ground powders and all were measured without any further preparation using an Agilent Cary 630 FTIR spectrometer equipped with KBr optics and complimentary diamond ATR accessory. Measurement conditions are shown in the table below.

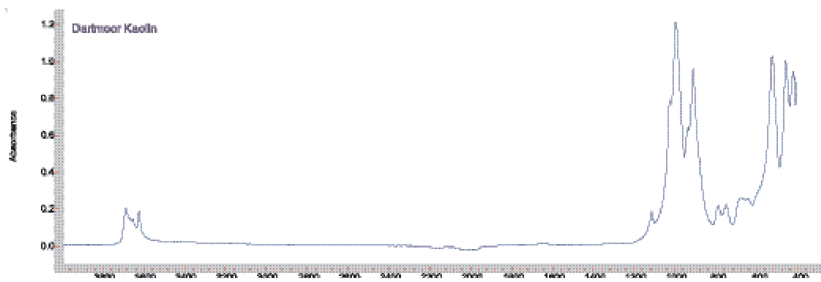
Spectral Range	4000 – 400 cm^{-1}
Number of Scans	32 (approx. 15 secs measurement time)
Spectral Resolution	4 cm^{-1}
Apodisation	Happ-Genzel

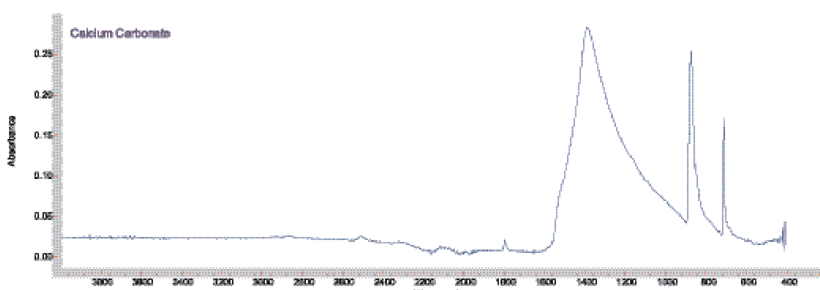
Results and Discussion

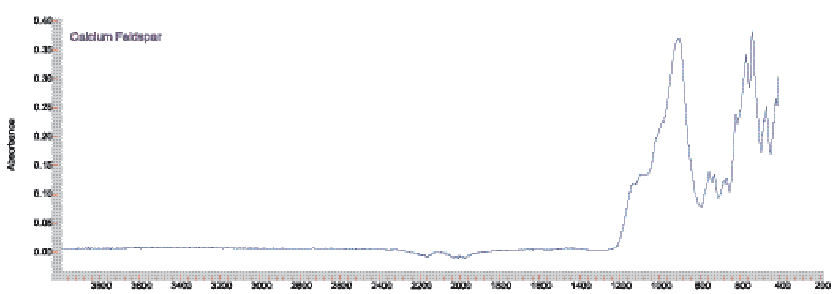
Spectra for each of the common minerals analysed in this study are shown below. All of the spectra show well resolved peaks, with excellent signal-to-noise ratio, and with several of the spectra showing an abundance of peaks below 600 cm^{-1}, the importance of using a monolithic ATR is clear.

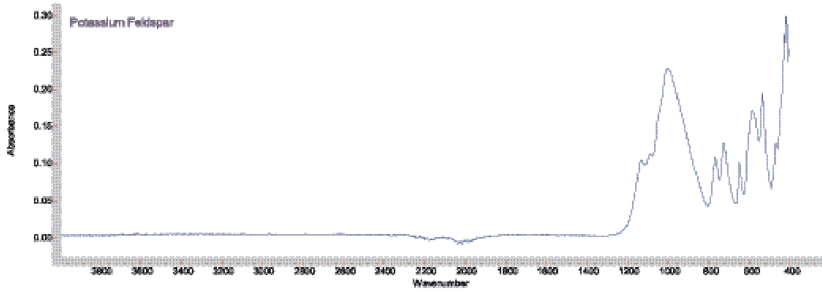
For the purposes of identifying each mineral type, the Cary 630 MicroLab software allows the creation of libraries from the user's own measured spectra against which unknown samples can be cross-referenced, or spectra can be searched directly against commercial libraries (including libraries that have been created in transmission using the KBr disk approach).

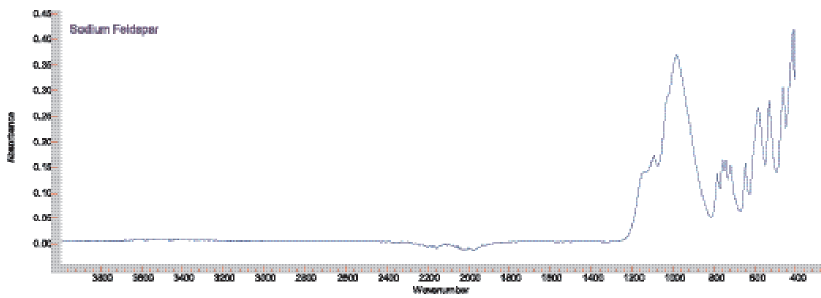


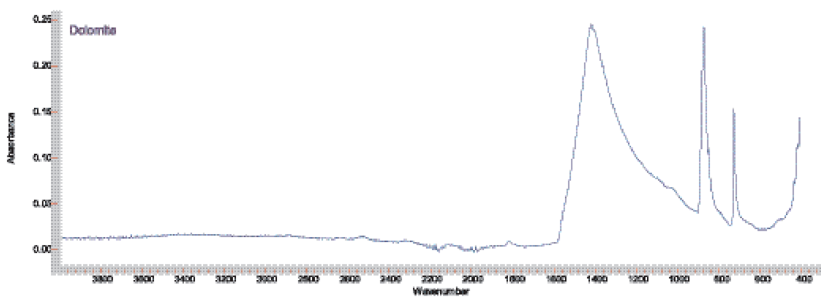


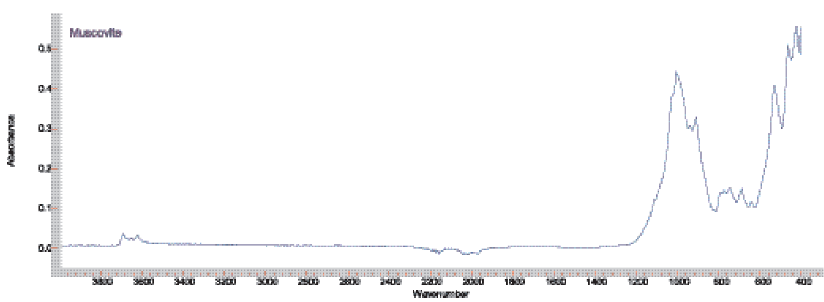


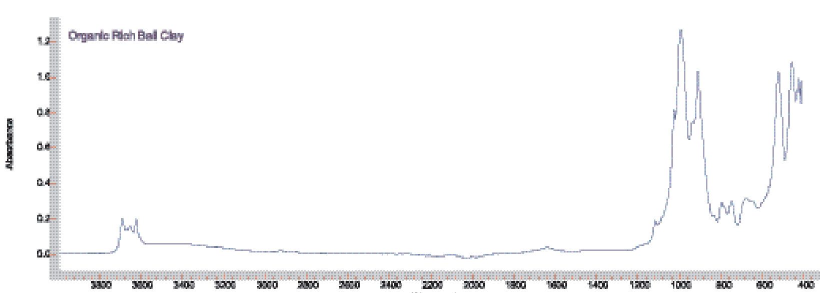



Potassium Feldspar
Absorbance
Wavenumber


Sodium Feldspar
Absorbance
Wavenumber


Dolomite
Absorbance
Wavenumber


Muscovite
Absorbance
Wavenumber


Organic Rich Ball Clay
Absorbance
Wavenumber

Conclusion

The results clearly demonstrate that the Agilent Cary 630 FTIR spectrometer is suitable for the analysis of minerals. The high energy throughput monolithic diamond ATR enables measurement down to 400 cm^{-1} with excellent signal-to-noise. The resulting spectra can be easily identified using a library search.

References

1. Rob Wills, “Advantages of the Agilent Cary 630 Diamond ATR”, Agilent Solution Note Reference 5991-3361EN, published **2013**.

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