

## Application of Fourier Transform Infra-Red Spectroscopy (FTIR) for Mineral Quantification

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*John Woods, SGS Australia Pty Ltd, 28 Reid Road, Perth Airport, WA, 6105, Australia, Email: [john.woods@sgs.com](mailto:john.woods@sgs.com)*

### Introduction

Fourier-transform infrared spectroscopy (FTIR) (Griffiths & de Haseth 2007) is a technique used to obtain an infrared spectrum of absorption or emission of a solid, liquid, or gas. An FTIR spectrometer simultaneously collects high-spectral-resolution data over a wide spectral range. In this study we utilised the range 500 – 4000  $\text{cm}^{-1}$  with a resolution of 4  $\text{cm}^{-1}$ . It is an established experimental technique for determining qualitative mineral identification. In this paper we examine the use of the Bruker Alpha II unit (Fig. 1) in the quantitative determination of common rock forming minerals.

For this paper, we used an attenuated total reflection (ATR) sample analysis unit. ATR is a sampling technique used in conjunction with infrared spectroscopy which enables samples to be examined directly in the solid state without further preparation (Perkin Elmer Life and Analytical Sciences 2005). ATR uses a property of total internal reflection resulting in an evanescent wave. A beam of infrared light is passed through the ATR crystal in such a way that it reflects at least once off the internal surface in contact with the sample. The ATR system used in this study was based on a diamond system which made it ideal for minerals which have a high hardness index.



*Figure 1. Bruker Alpha FTIR fitted with ATR unit inset showing sample plate (image courtesy of Bruker) used in the quantitative determination of common rock forming minerals.*

### Experimental

Rock samples of between 200-300 g with a wide range of mineralogy were dried and pulverised until 100% passed a 75  $\mu\text{m}$  sieve. A split was then taken for analysis by high resolution X-ray diffraction (XRD) to act as calibration standards. The remaining portion was then analysed with the FTIR using the ATR attachment.

Method optimisation was determined using artificial intelligence (AI). The frequency region and preprocessing method as well as the resulting RMSECV value were displayed in order of relationship to the analyte calibration values. The selection frequency parameters were then checked against the known spectral response to the pure minerals to ensure that the parameters selected were valid. Further to this, samples which gave a result more than 20% from the calibration line were removed as outliers. The method was then recalibrated with the outliers removed.

The samples selected for validation encompassed the full concentration range for each analyte with the same variability in sample characteristics as contained in the calibration set. The validation set contained about 50% as many samples as the calibration set and 5 samples were selected for independent testing.

### Results and Conclusions

R-squared is a statistical measure of how close the data are to the fitted regression line. It is also known as the coefficient of determination, or the coefficient of multiple determination for multiple regression. 0.00% indicates that the model explains none of the variability, a result of 100% would mean all the data fits the model. So, R-squared can take a value between 0 and 1 where values closer to 0 represent a poor fit while values closer to 1 represent an excellent fit. If  $R\text{-squared}=0.93$ , then it infers that 93% of the variations in dependent variable Y are explained by the independent variables present in the model. The R-squared results for some of the mineral methods are displayed in Table 1. It is noted that

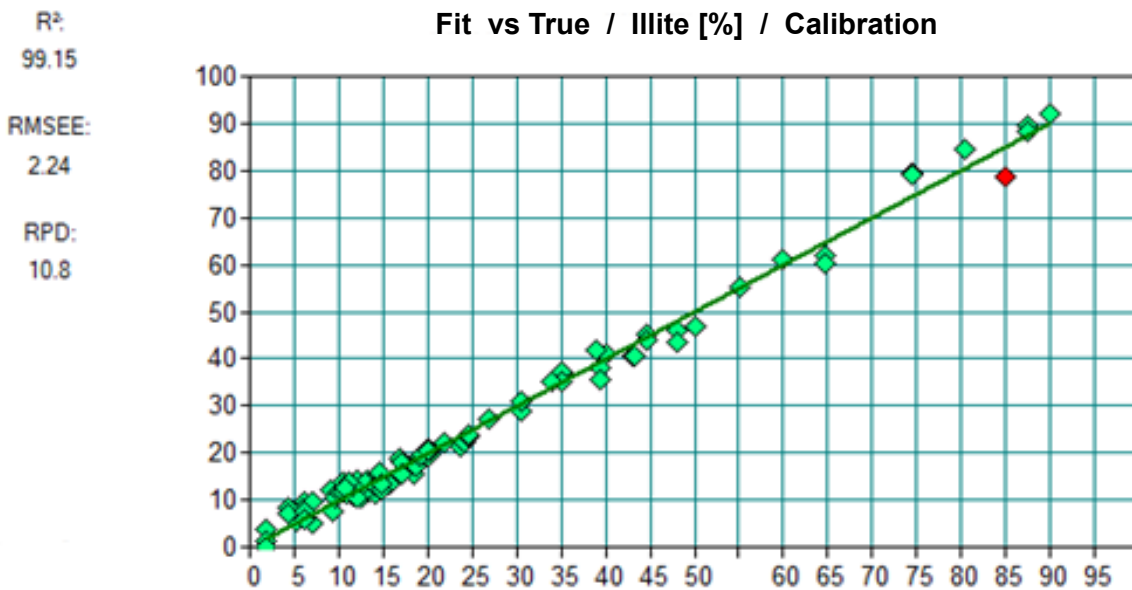
**Application of Fourier Transform Infra-Red Spectroscopy (FTIR)...** *continued from page 1*

only one of the methods has an R-squared value of less than 0.90, therefore for nearly all the methods covered in this report have a 90% fit. The presence of outliers is determined based on the Mahalanobis distance (Mahalanobis 1936) and displayed in Table 1.

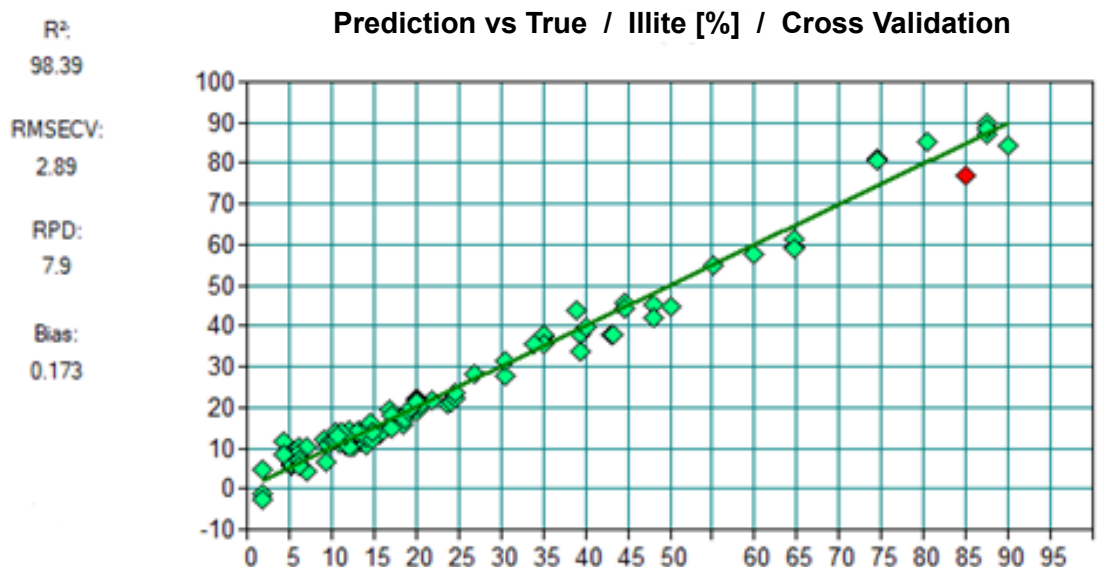
Mineral/ Analyte	R squared	Mahalanobis distance
Apatite	99.59	0.22
Calcite	98.66	0.24
Chlorite	99.67	0.44
Dolomite	99.05	0.21
illite	98.39	0.55
Kaolinite	99.86	0.52
K-Feldspar	96.07	0.21
Muscovite	95.36	0.17
Petalite	89.18	0.8
Plagioclase	95.29	0.31
Quartz	98.63	0.17
Siderite	98.23	0.26
Smectite	98.79	0.18
Spodumene	97.65	1.2

*Table 1. Mineral Quantification Methods – Performance quality of mineral methods with statistical assessment of error.*

The calibration graph for illite is displayed in Figure 2 and the validation graph for illite is displayed in Figure 3. Outliers are displayed in red. RMSEE is the root mean square error of estimation RPD is the residual prediction deviation, RMSECV is the root mean square error of cross validation and bias is the value for the rank displayed in the graph.



*Figure 2. Calibration graph for illite.*



*Figure 3. Validation graph for illite.*

## Application of Fourier Transform Infra-Red Spectroscopy (FTIR)... *continued from page 5*

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From these data FTIR can provide a highly accurate method for the determination of a wide range of minerals. The degree of accuracy is in general better than that achieved by X-ray diffraction (XRD), namely that it has errors of less than 10% from true rather than being in the order of 20%. The degree of accuracy is, in general, better than that achieved by X-ray diffraction (XRD) in that it has error of less than 10% from true, rather than being in the order of 20%. This is especially notable for clay minerals that present considerable difficulties for XRD analysis where preparation is a limiting factor in accuracy.

As the FTIR only requires that the samples are dry and ground to less than 75 microns this offers the opportunity for low cost, rapid mineralogical analysis without the need for complex preparation. This type of instrumentation is portable and robust and as the range of minerals that can be quantified expands may supplant more traditional technologies for a wide range of industrial applications.

There are, however, limitations to what the technology can provide. Firstly, the technique relies on there being bonds present within a mineral that have a vibrational mode. Halite and galena do not possess this type of bonding and so will always remain beyond the capability of this type of instrumentation to identify and quantify. Secondly, although there have been significant improvements in detector technology limits of quantification for most minerals in a complex rock matrix remain at about 5% w/w.

### Further Work

As mentioned in the previous section, the list of minerals that can be analysed and quantified by FTIR is ever expanding. New methods are currently under development that will provide accurate determinations of minerals such as biotite mica, potash, ferroan dolomite, haematite, goethite, topaz and sellaite. Other minerals will soon follow as commercial requirements dictate.

### Acknowledgments

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