

# Using FT-IR Spectroscopy to Characterize Gypsum Drywall Samples

Steve Lowry and Mick Dowd

Thermo Fisher Scientific

Madison Wisconsin, U.S.A.

## Abstract

A number of laboratories have reported that features in the infrared spectrum of a gypsum drywall sample can be attributed to the material being produced in China. In this study we acquired spectra from seven different samples of drywall, six of the samples were provided by a testing laboratory with no initial documentation. Several samples were also acquired locally in Madison WI. All samples were measured on a Thermo Scientific Nicolet iS10 FT-IR spectrometer with an ATR (attenuated total reflectance) accessory. A significant peak was observed near 1450 cm<sup>-1</sup> in the spectra from four of the samples. A small peak was seen in two and the peak was extremely weak or missing in three of the samples. A comparison of the acquired spectra to a series of reference spectra suggest that the peak at 1443 cm<sup>-1</sup> corresponds to the presence of Strontium Carbonate in the samples. When the documentation was provided, the samples with the largest peaks were from material produced in China. The origin of the fourth sample with a large peak was unknown, but was reported to have a high level of strontium by XRF. Two samples with a smaller but detectable peak were from a US produced material.

## Introduction

An extreme shortage of domestic drywall occurred during the recent housing boom in the southeast and repair of damage caused by several major hurricanes. This demand resulted in a drastic increase in the use of imported drywall, with millions of sheets of gypsum drywall being imported from China during the period from 2004-2007. Recently homeowners have reported physical ailments, odors and corrosion of copper hardware in their homes. These problems appear to correlate with the presence of drywall imported from China in the homes. Several contract Analytical Laboratories have reported that FT-IR spectroscopy can be used to identify drywall produced in China (2). The laboratories base their classification on the presence of a spectral feature near 1450cm<sup>-1</sup>. We have recently obtained several samples of drywall from known sources and reproduced the results described in these Analysis Reports from independent laboratories. We also performed a TGA-IR analysis of one sample to identify the volatile components evolving during the heating of the sample. In a final test we compare the intensity of the FT-IR peak at 1450cm<sup>-1</sup> to the strontium values obtained with a Thermo Scientific NITON handheld XRF analyzer.

## Materials & Methods

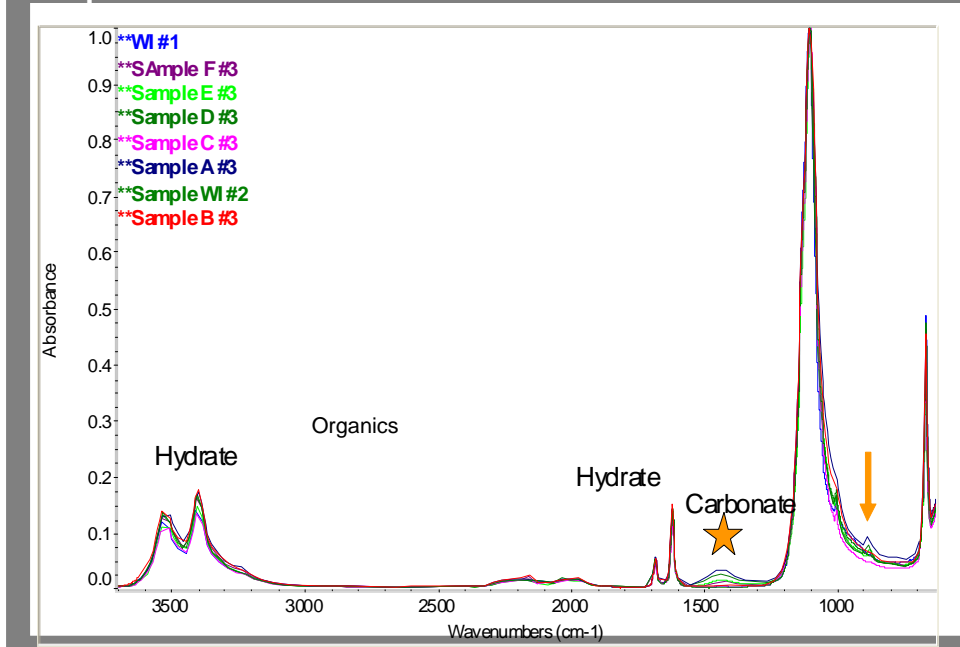
Six samples of gypsum drywall were provided by an independent testing lab. No information about the samples was provided before the analysis was completed. A Thermo Scientific Nicolet iS10 FT-IR spectrometer with the ITR accessory was used to obtain a spectrum from each sample in less than a minute. A small amount of powder or a piece from the drywall is placed on the ATR crystal and the Pressure Applicator precisely presses the powder against the crystal. Less than 100 milligrams of sample are required to obtain a high signal-to-noise spectrum. A comparison of spectra acquired at a 4 cm<sup>-1</sup> and 2 cm<sup>-1</sup> resolution confirmed that the spectral features could be correctly analyzed at the lower resolution with enhanced sensitivity. In the ATR experiment, the evanescent wave of the infrared energy interacts with a sample in contact with the ATR crystal. The light absorbed by the sample is directly related to the vibrations of the chemical bonds in the material. Different chemical bonds produce spectral features at specific locations in the infrared spectrum.

FIGURE 1. Nicolet iS10 FT-IR with ITR Smart Accessory for rapid analysis based on ATR (Attenuated Total Reflectance) with expansion of a sample and the Analyzer Crystal



## Results and Discussion

FIGURE 2. ATR spectra from seven Drywall samples acquired with the Nicolet iS10 system with 1 minute acquisition times



### Rapid Sample Analysis

The size of the peaks in the spectrum corresponds to the amount of a particular species that is in contact with the analyzer crystal. The spectra from the seven samples have been normalized to the sulfate peak at 1100cm<sup>-1</sup>. The gold star identifies the peak used by several laboratories to classify the sample as made in China. The gold arrow designates a second peak near 875cm<sup>-1</sup> also corresponding to a carbonate spectrum. The main feature at 1100cm<sup>-1</sup> is due to the sulfate of the gypsum. Small differences in the shape of this peak may be due to hydration or the presence of other atoms than Calcium. Slight variations in the hydrate peaks may be due to the environment of the material. With sufficient samples and proper statistical analysis these differences may provide information about the origin of the samples

The expanded spectra shown in figure 3, reveal the differences in the 1450cm<sup>-1</sup> and identifies the samples as well as the estimated Strontium concentrations from the XRF measurements. An examination of the spectra shows that the three samples with the largest FT-IR peaks are imported material. The origin of a fourth sample (Spectrum F) that contains a strong peak is unknown. One sample identified as US material does have a small peak and also has a higher level of reported strontium. This expansion also shows the small differences in the height of the Hydrate peak near 1650cm<sup>-1</sup> relative to the Sulfate peak at 1100cm<sup>-1</sup>. A more detailed examination of the relative peak heights and the shape of the sulfate peak might also reveal more information about the chemical makeup and possibly the origin of the different gypsum materials.

FIGURE 3. Expansion with sample identifications and strontium results from the XRF measurements

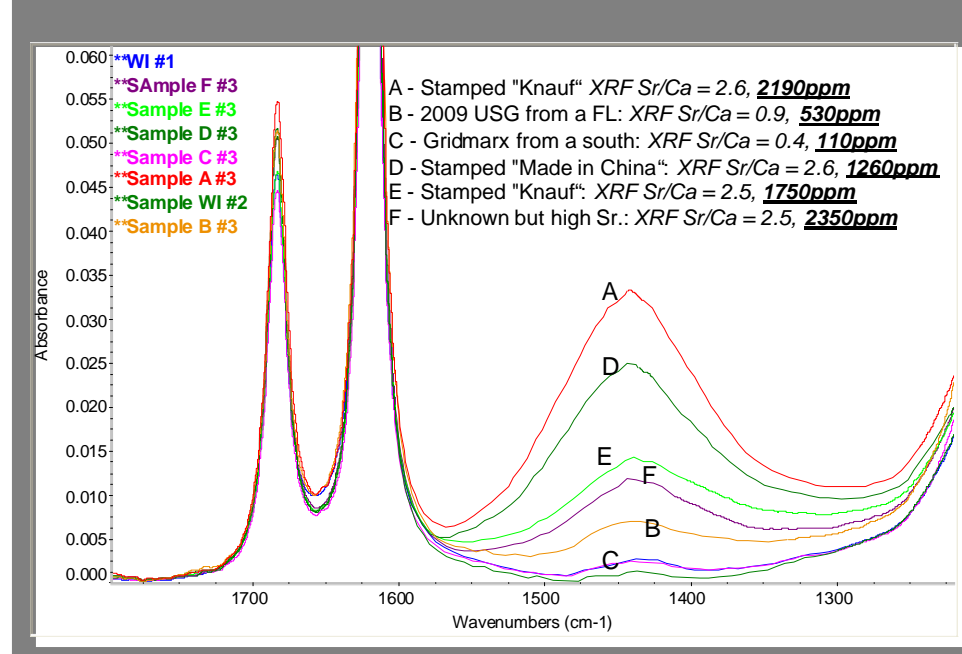
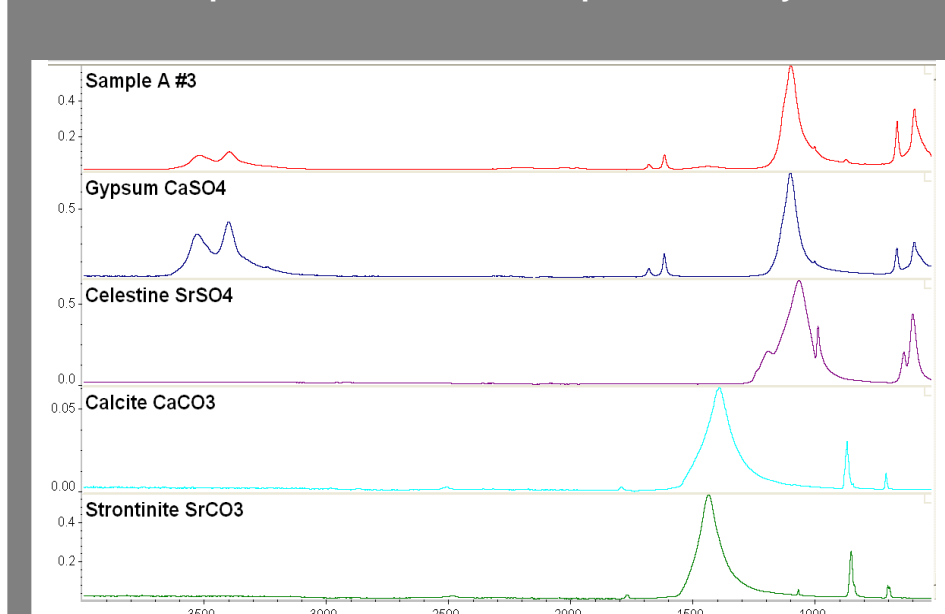
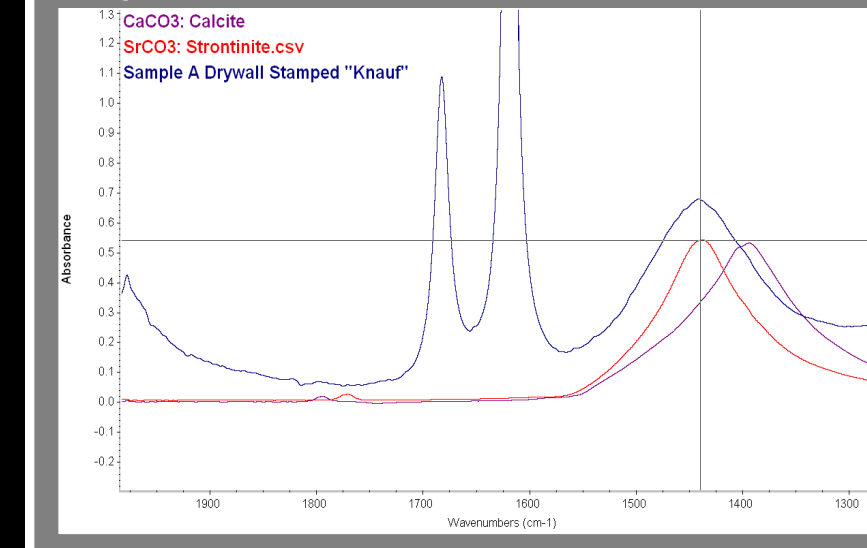


FIGURE 4. Comparison of the spectrum of Sample A with reference spectra from the RRUFF Spectral Library



A comparison to reference spectra from various minerals reinforces the conclusion that the reported peaks are due to the presence of carbonate. However the peak observed in the drywall samples does not exactly align with the calcite reference spectrum but does match very well with the peak in the strontinite reference spectrum. One of the web sites stated "Strontium naturally occurs in carbonate mineral deposits in China of the type that go into gypsum based drywall Sulfur also naturally occurs. The U.S. drywall tested against the Chinese sample contained smaller amounts of Strontium and no measurable sulfur" These observations are summarized in the expanded figure where the reference spectra from Strontinite and Calcite are shown with a spectrum from a sample of drywall imported from China.

FIGURE 5. Overlay of spectra from Strontium Carbonate and Calcium Carbonate on spectrum from sample A

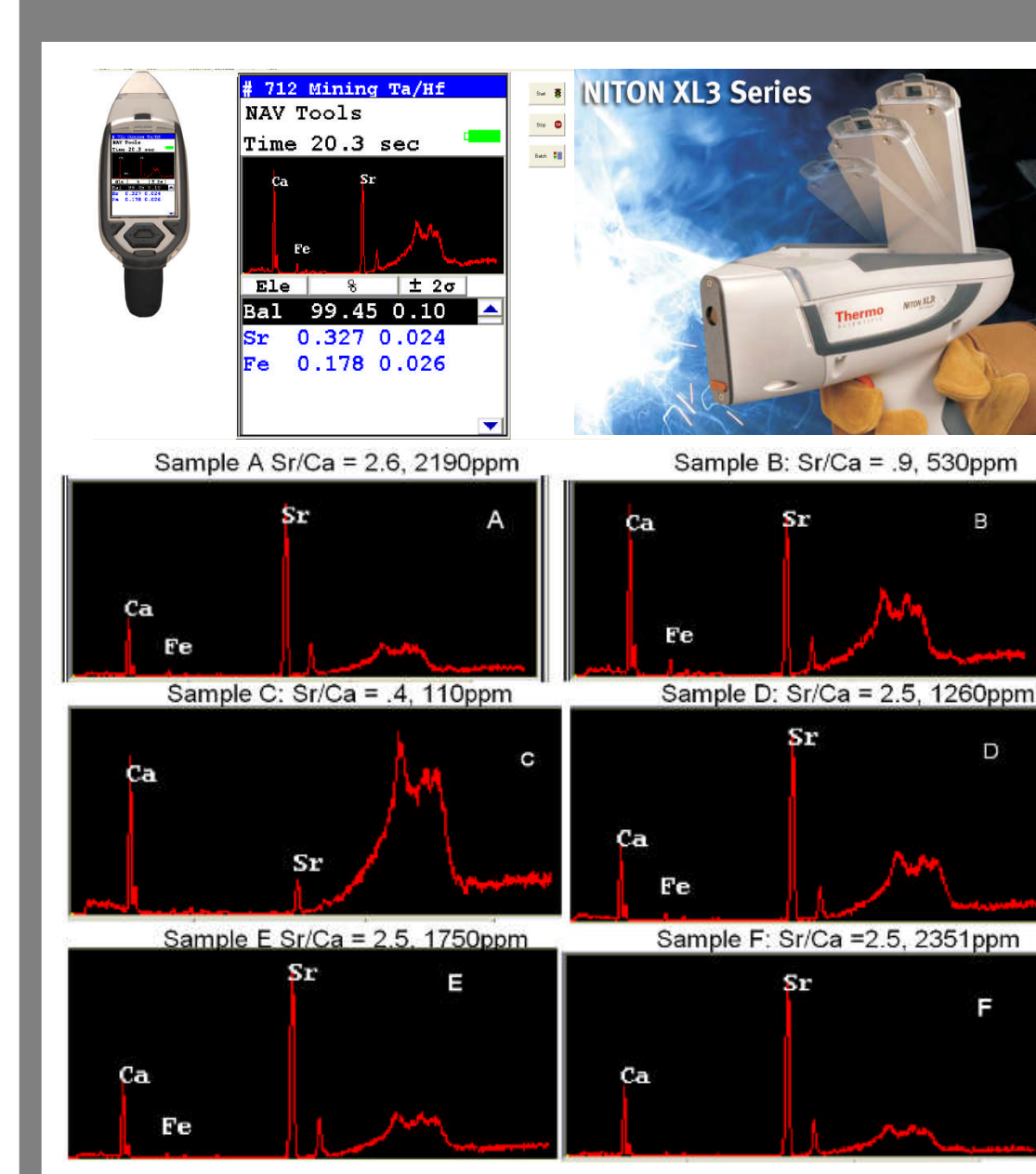


This expanded spectrum compares the reference spectra from Calcium Carbonate and Strontium Carbonate to the spectrum from Sample A. The main peak in the CaCO3 spectrum does not align with the peak in the sample. However, the SrCO3 peak aligns quite well with the peak in Sample A. This is strongly indicative that the samples from China contain higher levels of Strontium Carbonate. The peak in the sample spectrum is wider than the reference spectrum, which may be due to interactions with the CaSO4 major component.

### Comparing FT-IR with XRF Analysis for Strontium

A number of groups have reported high levels of Strontium in samples of imported drywall. In this study we used a NITON XL3 handheld XRF analyzer to measure the amount of Strontium in the six samples of drywall. The samples were measured in the polyethylene bags with the powder collected in a corner to provide a thicker sample. Because of the sampling difficulties and observed variance in repeat sampling we used the ratio of the Strontium peak to the Calcium peak as a good estimate of the relative amount of strontium in the samples. Measurements between ten and twenty seconds were used for the samples. For measurements of samples with lower levels of Strontium the reported uncertainty was less than +/- 20ppm.

FIGURE 6. Measuring Strontium levels using a handheld XRF analyzer on powder samples in plastic bags



### TGA-IR: Combined Infrared and Thermo Gravimetric Analysis

The combination of Thermo Gravimetric Analysis and FT-IR provides an excellent tool to identify the gases evolved from a sample when it is heated. In this exploratory study 45 milligrams of Sample A were placed in the TGA and the temperature ramped from 30C to 900C over 45 minutes while the sample was purged with nitrogen gas. The heated gas stream from the TGA flows through a gas cell in the FT-IR and spectra are acquired every few seconds. The result file from the TGA is linked to the FT-IR series file. The derivative weight loss curve from the TGA correlates very well with the IR spectral response from the sample in the gas cell. The first weight loss corresponds to water and the second to CO2. A small sulfur dioxide peak is the only other evolved gas observed in this experiment.

FIGURE 7. Experimental apparatus used in the TGA-IR analysis of volatile components.

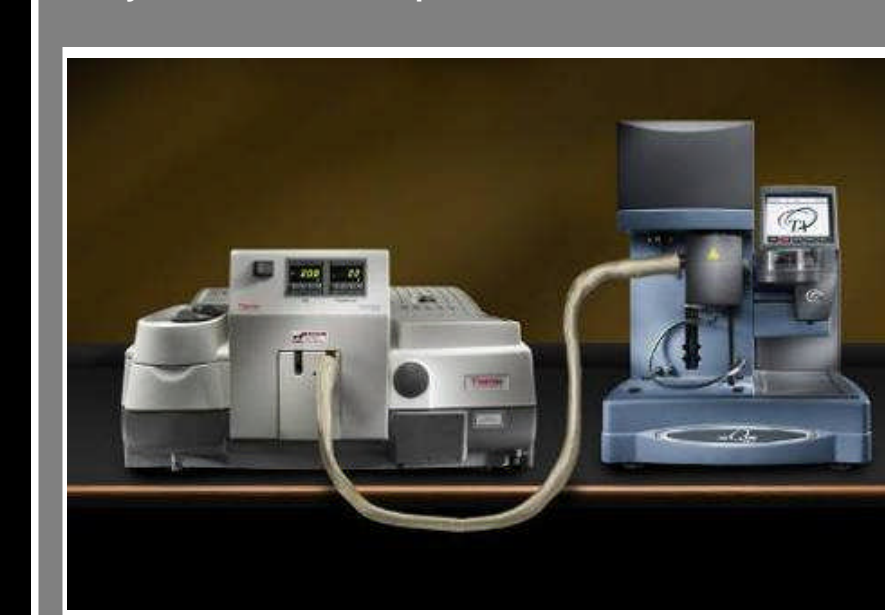


FIGURE 8. TGA results for 45mg of Sample A sample A

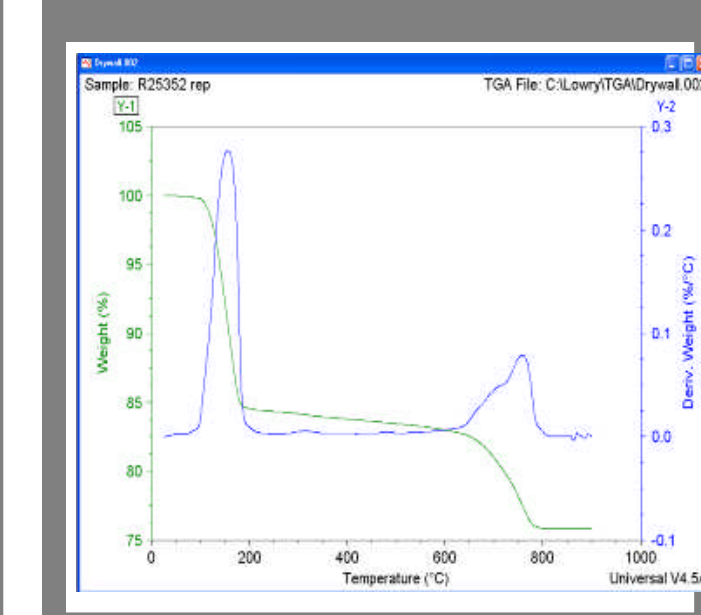
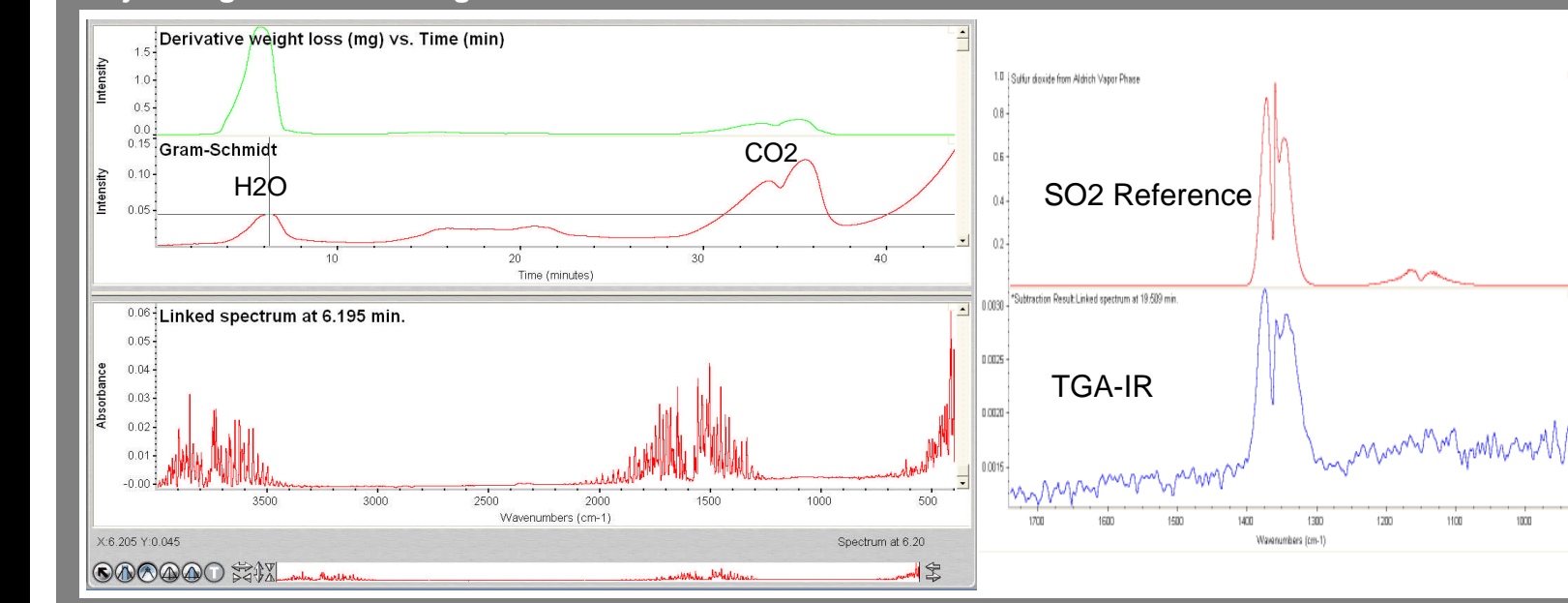


FIGURE 9. TGA-IR results for 45mg of Sample A showing derivative weight loss and IR response with major weight loss resulting in H2O and CO2. A small amount of SO2 was also detected.



## Conclusions

FT-IR spectroscopy provides a rapid and reliable way to obtain valuable information about gypsum drywall. The peak near 1450cm<sup>-1</sup> is most likely due to the presence of Strontium Carbonate and is observed at high concentrations in samples of imported drywall. However, it is also observed at a significantly lower level in two samples of drywall produced in the US. The intensity of the 1450cm<sup>-1</sup> peak correlates reasonably well with strontium levels measured by XRF. TGA-IR detected a small amount of SO2 but did not find any other sulfur species even at high temperatures. Large amounts of CO2 are observed at high temperature most likely due to the decomposition of the carbonate. There is no obvious cause and effect between the presence of strontium in the drywall and observed corrosion in homes.

## References

1. Downs, R. T., (2006) *The RRUFF Project: an integrated study of the chemistry, crystallography, Raman and infrared spectroscopy of minerals*. Program and Abstracts of the 19th General Meeting of the International Mineralogical Association, Kobe, Japan.
2. "Suspect Chinese Drywall Analysis Fourier Transform Infrared Spectroscopy", [www.assuredbio.com](http://www.assuredbio.com)