Application Note
C-219

A Highly Flexible and Integrated TG/FTIR System

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As a one of the most commonly used physical property measurement apparatus, Thermogravimetry (TG) has widely been used to detect material composition. [1,2] When the sample's evolved gases are also of interest, an evolved gas analyzer (EGA) unit will be utilized. Among the options for EGA, Fourier Transform Infra-Red spectroscopy (FTIR) and Mass Spectroscopy (MS) are two techniques often used. Between TG/FTIR and TG/MS, TG/FTIR is the more common combination because of the relatively simple coupling technique involved and generally lower cost of the equipment.

In a TG system, the important measured parameters are weight, temperature and time. Weight is the signal, which is the most important of these, and is dependent on the capacity and sensitivity of the balance used in the TG system. In FTIR, besides the accuracy of the wavenumber, another important feature is the signal to noise ratio or sensitivity.

To measure small amounts of a component in a sample, the detection limit will ultimately decide if the measurement can be made. The detection limit is defined as the smallest concentration or percentage of a component, which can be detected. Such a definition has been widely used for various analytical instrumentation, and in general, the higher sensitivity, the better the detection limits. However, this term has not been applied to TG or TG/FTIR, because sample size will decide the amount of evolved gases and sample weight loss/gain, and eventually the detection limit for a sample. [3]

The Thermo Scientific Cahn TG systems have the largest sample size and volume to accommodate the widest range of sample geometries. Especially when the Thermo Scientific Cahn TG is integrated, both hardware and software, with the Thermo Scientific Nicolet FTIR, this makes the most flexible and sensitive integrated TG/FTIR system.

Experimental

Experiments were performed on a Cahn Synergy™ TG-FTIR system. The thermogravimetric analyzer is a Cahn TG-2131. The FTIR signal is collected on a Nicolet FTIR, with a DTGS detector and they are coupled by the Cahn TG/FTIR interface. [4] The equipment is controlled from a single computer and both the TG and FTIR data are saved into the same data file to insure data integrity. Data is displayed in real time during the course of the experiment. [5]

In order to show the flexibility and capacity of the integrated system, three different kinds of samples were analyzed. One sample is a small amount of calcium oxalate monohydrate, about 7 mg, another sample is a silicon wafer, with a thin Teflon coating, and the third, a polystyrene sample spiked with very small amount of hydrogen bromide.

All samples were heated under a nitrogen environment. The evolved gases passed through a heated gas cell where the FTIR spectra were collected. The FTIR spectra were collected continuously at a resolution of 4 cm^{-1}, and the sampling scan number for each spectrum was 16 (about 8.2 seconds). The transfer

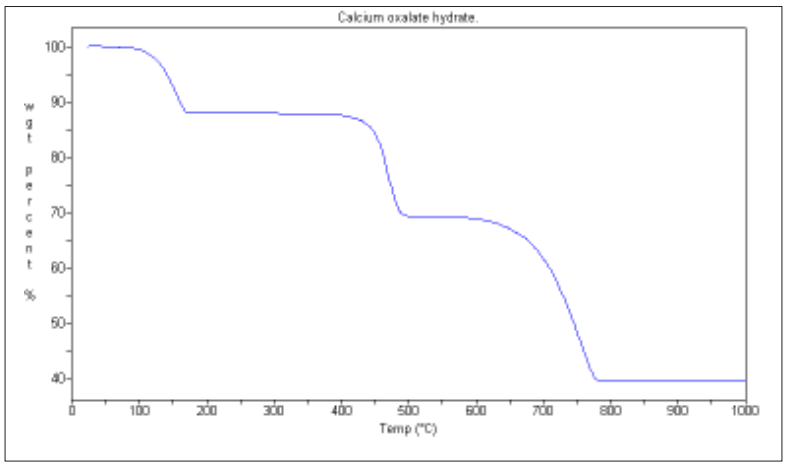

Figure 1: 7.0 Milligram Calcium Oxalate

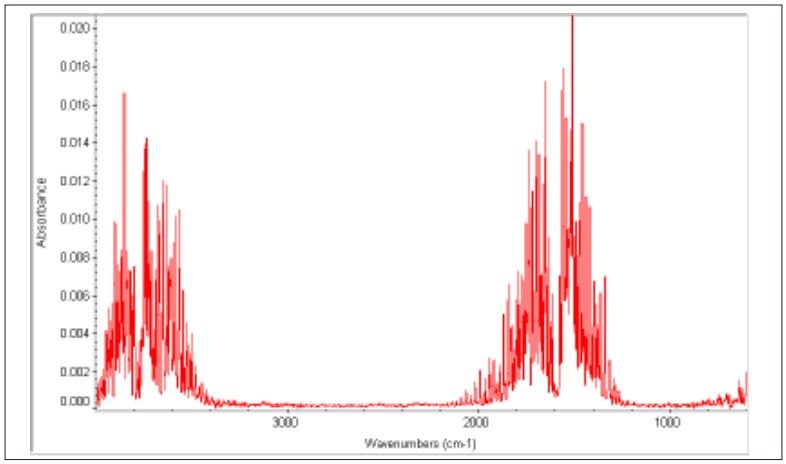

Figure 2: Evolved water at 150 °C.

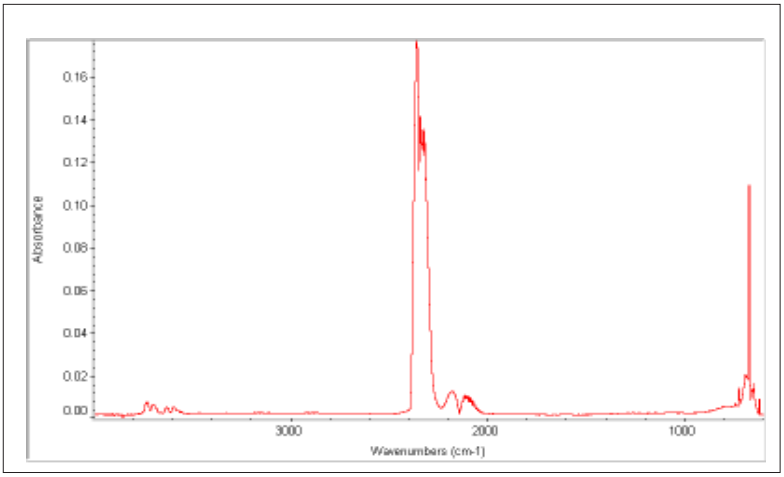

Figure 3: Evolved CO2 with CO at 470 °C.

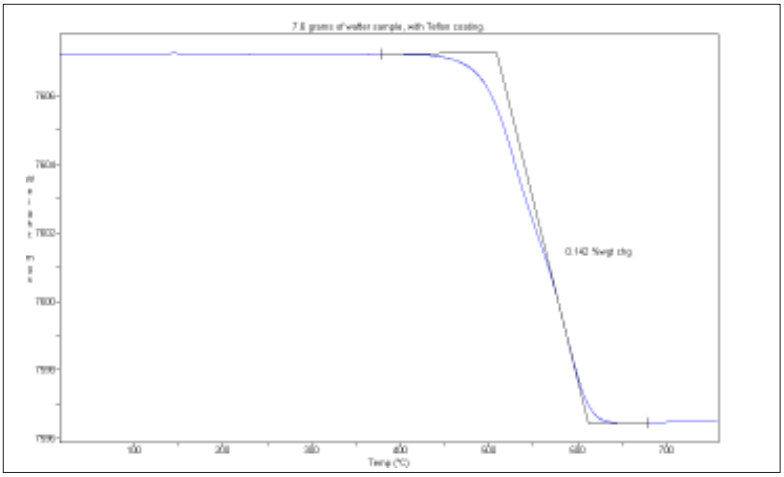

Figure 4: TG weight loss of the Wafer sample

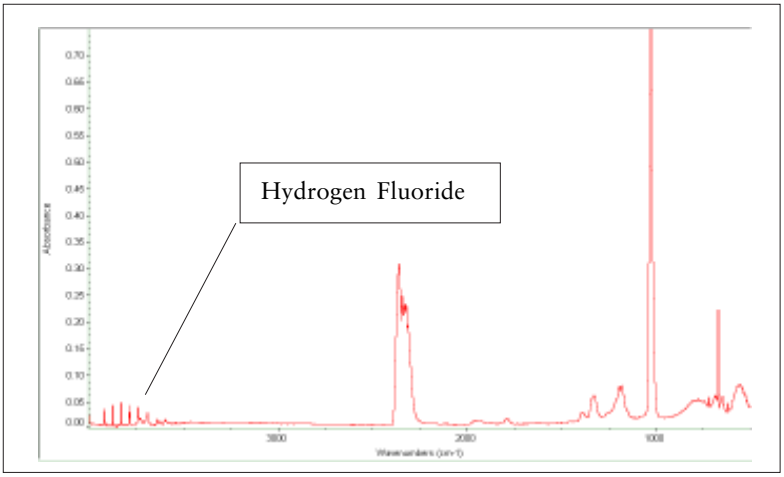

Figure 5: Evolved gas from Wafer at 25.8 minutes, 535 °C

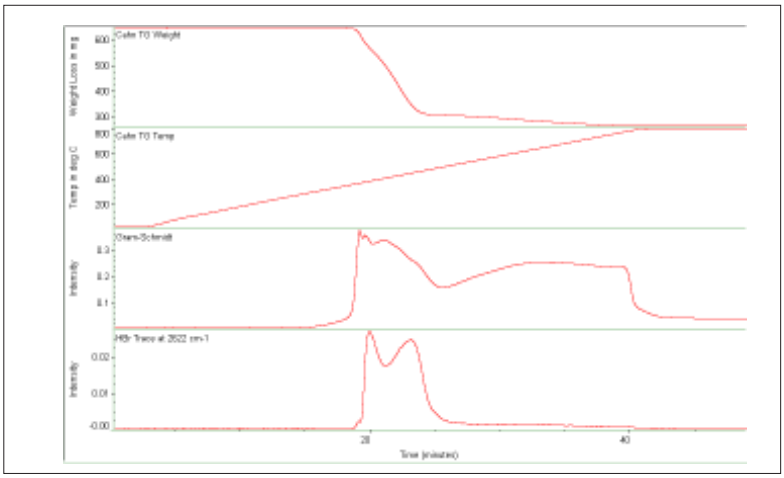

Figure 6: Time evolved traces for HBr.

lines and the FTIR cell of TG-FTIR interface were heated at 200 °C to prevent evolved gases from condensing.

Results and Discussion

While the Thermo Scientific Cahn's TG has a sample capacity of up to 100 grams and a sample volume of 35 ml, it doesn't mean that the system can't be used for small sizes. The run on 7 mg calcium oxalate hydrate shows that both the TG and FTIR can obtain the necessary information about sample's weight and effluents as shown in Figures 1, 2, and 3. The evolved water (figure 2) and carbon dioxide (figure 3) are easily identified in the FTIR spectra. This is due to the fact that TGA has 1-microgram sensitivity, which is very high to ensure the detection of small weight losses and the patented synergy setup, which is sensitive enough to allow the FTIR to observe small amount of effluents.

There are lots of samples come with very small amount of components that are of interest. One case is that the main substrate is inert, and another case is that the main component is not inert. In the later case, the worst case is that the interested minor component is embedded in the main components and has very similar thermal property.

In the first case, it is necessary to increase the amount of sample to allow the TG system to be able to detect the weight changes and FTIR system to detect the effluents. Semiconductor Wafer sample fits into this category perfectly, and Figures 4 and 5 are experimental results from this type of analysis. From the TG curve, Figure 4, it can be seen that the amount of weight loss is about 0.142 % weight. However, the FTIR spectrum, Figure 5, is able to detect the evolved gases, including hydrogen fluoride, very easily.

In an example of the later case, the sample is polystyrene sample spiked with Hydrogen Bromide, (HBr). Figures 6 and 7 show detection of HBr and the time-evolved traces for HBr. Figure 6 shows the fully integrated Thermo Scientific Cahn – Thermo Scientific Nicolet software storing all of the TG and FTIR information in the same data file,

allowing viewing in the real-time mode and analysis following the experiment. The software allows the user to create time-evolved traces for the interested effluents.

Figure 7 shows the TG/FTIR system can easily be used to obtain FTIR information of the major and minor components of samples.

Conclusions

Due to the sample capacity and sample volume, the TG/FTIR system proved to be able to handle a wide variety of samples. Furthermore, the Synergy TG/FTIR coupling makes the detection of minute amount of additives given off from the sample possible by FTIR. The fully integrated hardware and software between Cahn TG and Nicolet FTIR made the data collection and analysis very easy and powerful.

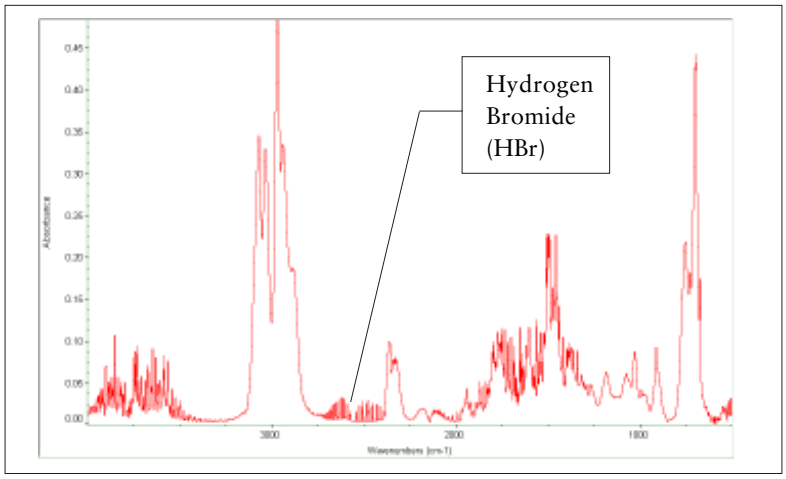

Figure 7: FTIR spectrum for polystyrene at 385 °C

Reference

[1] *W. W. Wendlandt and P. K. Gallagher, Thermal Characterization of Polymeric Materials, Ed. E. A. Turi, Academic Press, New York,* ***1982****.*
[2] *D. Dollimore, Analytical Chemistry,* ***1994****, 66, 17R-25R.*
[3] *D. Chen, and D. McMorran, Thermal Trends, Vol 3, No 4, Fall* ***1996****.*
[4] *Thermo Scientific Cahn Synergy Series, Thermo Fisher Scientific, Inc., Product Note,* ***1997****.*
[5] *Thermo Scientific Cahn TG/FTIR series, Thermo Fisher Scientific, Inc., Product Note,* ***2001****.*

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