A COMBINED TG-GC-MS SYSTEM FOR MATERIALS CHARACTERIZATION

Jen Chiu
Polymer Products Department, Experimental Station
E. I. du Pont de Nemours & Company, Inc.
Wilmington, Delaware 19898

ABSTRACT

Thermogravimetry is a powerful technique for studying stability and determining composition of a variety of materials. However, it derives information based on monitoring weight losses as a function of temperature and does not identify the volatilized components. Its capability for materials characterization is greatly enhanced if other analytical techniques are combined with it to analyze the off-gases at various weight loss steps. This presentation describes a coupled thermogravimetry-gas chromatography-mass spectrometry (TG-GC-MS) system based on the Du Pont 951 thermogravimetric analyzer and a Hewlett-Packard computerized GC-MS instrument. Examples will be given to illustrate the features of such a combination.

INTRODUCTION

Thermogravimetry (TG) is a powerful technique for materials characterization based on continuous measurement of weight changes upon controlled heating in a controlled environment. Its application on stability studies and compositional analyses are well known. However, this technique provides mainly weight change information and does not identify the nature of volatiles lost or the residues remaining. It also does not detect any structural transformations if there is no weight change. Attempts have been made to couple other analytical techniques to TG in order to enhance its capability, most notably infrared spectroscopy (IR) (1,2), mass spectrometry (MS) (3-6) and titrimetry (7-9). These coupled systems have shown great promise in analysis of volatile
substances evolved from TG, resulting in a wide variety of applications. Unfortunately, in many practical problems, the volatiles are complex mixtures not easily handled by these analytical methods. One logical solution is to couple a separations tool such as gas chromatography (GC) to TG (10-11). To go one step further, an ideal system would include a TG apparatus as a sample conditioner or reactor, a GC unit to perform separations and then an identification instrument. Such a combination has been demonstrated by combined TG-GC-IR (12) and TG-GC-MS (13), and is expected to gain wider use since coupled GC-IR and GC-MS are now commercially available. This presentation describes our combined TG-GC-MS system which can be alternately used for TG-MS, and illustrates its features with several examples.

EXPERIMENTAL

A schematic diagram for the instrumentation is shown in Figure 1. A Du Pont 990 or 1090 thermal analyzer equipped with a 951 thermogravimetric analysis module (Analytical Instruments Division, Du Pont Company, Wilmington, DE) is coupled with a Hewlett-Packard 5710A gas chromatograph and a 5982A mass spectrometer (Hewlett-Packard, Palo Alto, CA). A dedicated Hewlett-Packard 21MX computer system combined with a Tektronix 4012 CRT and a Tektronix 4610 copier is used for GC-MS data acquisition and computation.

The GC-MS interface is a standard jet separator provided by Hewlett-Packard. The TG-GC interface is a U-shaped condensation trap made of 1/8-inch O.D. stainless steel tubing and connected to a 6-port microvalve (Cat. No. 5521, Carle Instruments, Fullerton, CA) previously used for coupling TG to MS (3) and shown in Figure 2. This interface is connected to the GC injection port through a 1/16-inch stainless steel tubing silver-soldered to a syringe needle. In the TG-GC-MS operation mode, the injection port is connected through a GC column and the jet separator to the MS inlet A. The volatiles from TG are condensed in the trap by liquid nitrogen during sampling along the weight loss curve. The collected sample is injected onto the GC column upon rotating the microvalve by a compressed air actuator (11), and heating the trap with a hot flame. This method does not provide an injection as fast as that previously reported (11), thus reducing the column efficiency, but is simpler to operate, and adequate for most applications. A 6 ft x 1/8-inch diameter stainless steel column packed with 80/100-mesh Porapak Q was used for the brown speck work, and a similar column packed with 80/120-mesh Polypak #1 (Hewlett-Packard) for the cyclohexanol desorption work. Helium was used as carrier gas at a flow rate of 20 ml/minute. The injection port temperature was 250°C and the column was programmed from 50° to 250°C at 8°C/minute for the brown speck problem. For