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COUPLING OF A THERMOGRAVIMETRIC ANALYZER WITH MASS SPECTROMETER.

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ABSTRACT

A simple experimental set-up was designed to interface a thermogravimetric analyzer(TGA) and a mass spectrometer. With appropriate calibration the system is suitable not only for qualitative, but also the quantitative, determination of the pyrolytic products evolving from pyrolyzing carbohydrate samples in the TGA.

INTRODUCTION

Thermogravimetry has been used intensively for studying the reaction kinetics of various pyrolytic processes. Theoretically, it is also an excellent method to follow and understand the multiple reactions occurring when such complex molecules as carbohydrates are pyrolyzed. However, this application requires the continuous quantitative analysis of the products from a highly dilute gaseous stream. To analyze these dilute pyrolytic products a mass spectrometer can be used, but very little is known about interfacing TGA and MS.

RESULTS AND DISCUSSION

In our experiments, a TGA, a GC and a MS were interfaced successfully in such a way that the flow from the TGA could be introduced either directly to the MS or first to the GC and then into the MS. In case of the direct introduction, the gas flow was sampled and regularly evaluated using a computer program specifically written for this purpose. After determining the maximum amount of each possible compound in relation to water, the data was plotted versus time. Since the starting materials were carbohydrates, the computer search was limited to carbon, hydrogen and oxygen containing compounds. But even in this case, the statistically possible compounds are many.

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The mass spectrometric analysis of a complex mixture is difficult, because the composite mass spectrum of two compounds together with the background noise can easily contain the major characteristics of a third compound that might not be present. The direct sampling of the gas flow by MS provided an excessive number of possiblities, even after accounting for background noise level. Therefore, the flow was directed at room temperature through a GC using a Tenax column that adsorbed all organic molecules. Only the water passed through. This was confirmed by monitoring the flow leaving the GC column by the mass spectrometer. The computer evaluation of the MS data showed only water above the noise level. At the end of the pyrolysis, the GC was programmed to separate the adsorbed materials while continuously monitoring by MS the effluent gases. The analysis of the MS data compared with the GC spectrum allowed better identification of the components. Further identification was also made by comparing retention times. Knowing the compounds actually present, the evaluation of the data obtained by the direct introduction to MS was improved through another computer program written for this purpose. This also allowed an approximation of the quantitative composition of the mixture as a function of time and temperature.

This method is still not perfect since it only determines that a compound forms at some time during the pyrolysis, and it does not guarantee that the given compound is forming all the time. The possibility that two compounds will provide the mass spectra of a third ine which is not present at a given time is still possible, but the probability decreases. This method must be combined with additional spot checking through adsorbing in smaller temperature intervals the pyrolytic products on the GC column and comparing the results with analytical data obtained from the computer evaluation. The paper will describe the instrumental set-up and the interface. It will also discuss in details the problems and their solutions. The method to quantitize the MS data through calibration will be illustrated through examples. A description of the computer programs will be given.