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NASA TECH BRIEF

Langley Research Center

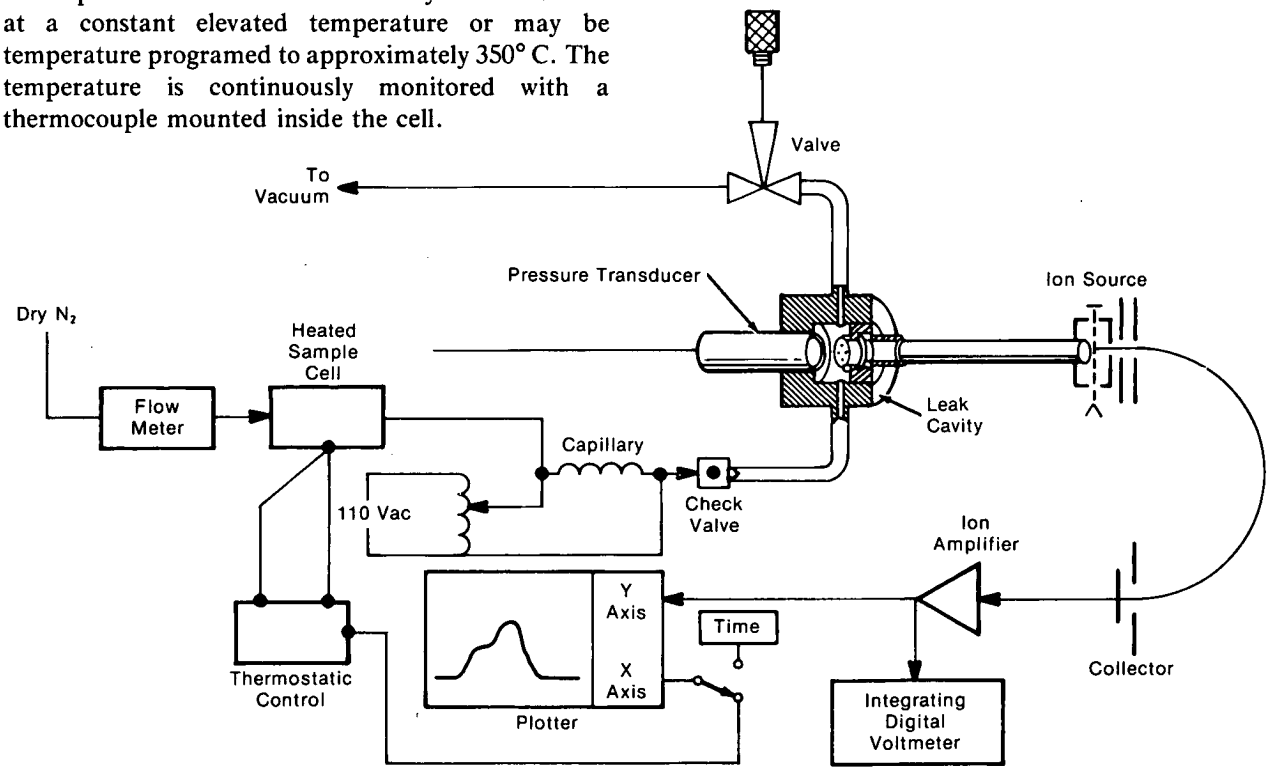
Determination of Water Content Using Mass Spectrometry

A new system using a mass spectrometer has been developed to measure small quantities of water present in different materials. It has been applied in measuring water and gases desorbed from less-than-1-g samples of Teflon/FEP (fluorinated ethylene-propylene copolymer) insulation. The insulation is used in microcircuitry. The system can also be used with foods, polymeric materials, and organic solvents.

As shown in the illustration, the system includes a Pyrex sample cell. The cell is connected in-line with a dry nitrogen gas source and with an electrically-heated, 0.010-in. (0.3-mm) stainless-steel capillary, approximately 25 in. (63.5 cm) long, leading to the mass spectrometer inlet. The cell may be maintained at a constant elevated temperature or may be temperature programed to approximately 350° C. The temperature is continuously monitored with a thermocouple mounted inside the cell.

A weighed sample is introduced into the cell, and the evolved gas is swept into the mass spectrometer leak cavity by the dry nitrogen gas at approximately 25 cm^3/min. The data are obtained by recording ion current on an X-Y plotter as a function of time or temperature, with the mass spectrometer tuned to monitor a single mass. The ion current is simultaneously measured with an integrating digital voltmeter to obtain a quantitative measure of the total water or gas evolved. This value is determined by dividing the total number of counts by the instrument response.

The calibration procedure to obtain the instrument response is as follows: Samples of reagent-grade



Water Analysis System

(continued overleaf)

disodium tartrate dihydrate weighing 10 to 20 mg are introduced into the cell. The cell is subsequently programed to 180° C or is operated isothermally at 150° C, and the data are obtained as described above. Since the sodium tartrate contains 15.66 percent water by weight, these measurements yield instrument response in counts/mg H_2O.

The new method can measure water content over a range of milligrams to micrograms with an apparent detection limit of approximately 0.3 μg. Measurement precision, obtained from the determination of total water evolved from 10-mg to 20-mg samples of barium chloride dihydrate (14.75 percent water by weight), is of the order of 1 percent of the determined value. For the part-per-million concentrations measured from Teflon, the relative precision is better than 15 percent (standard deviation). The time required to obtain the data is approximately 2 to 4 minutes.

Note:

Requests for further information may be directed to:

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Reference: B75-10157

Patent status:

NASA has decided not to apply for a patent.

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