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Dynamic Technique for Measuring Adsorption in a Gas Chromatograph

The problem:

The adsorption characteristics of a compound can be obtained with the help of a gas chromatograph. However, when studying the adsorption of materials at low concentrations (less than 1 ppm) the standard technique requires several hours.

The solution:

A dynamic gas-chromatographic procedure, together with a mathematical analysis of the adsorption isotherm, allows relative surface areas and adsorptive powers for trace concentrations to be determined in a few minutes.

How it's done:

The adsorbent (charcoal for instance) heated in vacuum until it is free of all adsorbed compounds, is packed in a gas-chromatograph column. The retention volume of the compound of interest is measured in a temperature-controllable chromatograph. An inert gas such as nitrogen is used as a carrier.

The solid curve in the figure outlines a typical peak observed on the chromatograph. The shape of the

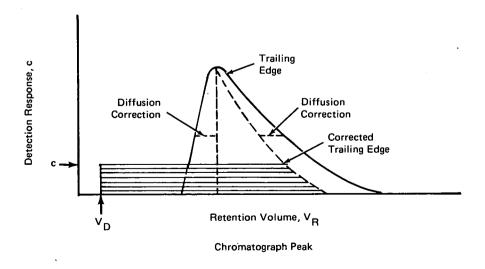
trailing edge can be corrected for diffusion, by assuming that the front edge deviates from a vertical line due to diffusion alone. The dotted curve shows the corrected edge, assuming diffusion broadened the trailing edge by the same amount that it broadened the leading edge. The detector response can be calibrated in terms of concentration by setting the total area under the curve equal to the amount of adsorbate used.

It can be shown that the time required for a point on the tail of the peak to pass through the column is proportional to the slope of the isotherm, at the point corresponding to the concentration in the tail, and that the adsorption isotherm can be found by integrating the tail of the chromatographic peak according to

$$f(c) = \int_0^c f'(c) dc = \int_0^c \frac{V_R - V_D}{m} dc$$

where:

 V_R (c) is the retention volume for the tail of a chromatographic peak corresponding to a gas-phase concentration of "c";



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m is the mass of the solid phase;

f (c) is the adsorption isotherm evaluated at "c" with f'(c) as the first derivative; and

 V_{D} is the dead space.

In the figure, then, the shaded area represents

$$mf(c) = \int_0^c V_R - V_D dc$$

and can be evaluated for all values of c from 0 to the peak height.

This technique may be used to evaluate the relative surface areas of different adsorbates, expressed as a volume of adsorbent/gram of adsorbate, and to evaluate their relative adsorptive power.

Note:

The following documentation may be obtained from: National Technical Information Service Springfield, Virginia 22151 Single document price \$3.00 (or microfiche \$0.95

> Reference: NASA CR-115202 (N71-37657), A Study of Physiochemical Factors Affecting Charcoal Adsorption of Contaminants in Manned Spacecraft Atmosphere.

Patent status:

NASA has decided not to apply for a patent.

Source: C. L. Deuel, N. W. Hultgren, and M. L. Mobert of Analytical Research Laboratories Inc. under contract to Johnson Space Center (MSC-14083)