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A Simple Micro BET System

It recently became necessary in this laboratory to determine the nitrogen BET surface areas of some well-sintered metal catalysts whose surface areas were known to be smaller than those which could be conveniently measured with the standard apparatus.

Accordingly, the system shown in Fig. 1 was devised: The adsorption cell and doser are built as a single unit comprising three Hoke 4616-M4N valves and a thermistor, installed as previously described (*1*). This particular Hoke valve was selected because of its low internal volume and excellent high vacuum properties. The portion of the system between the first two valves including the thermistor manometer comprise the doser and are enclosed in an air thermostat. The complete adsorption unit was attached to a standard high vacuum apparatus. The thermistor was calibrated with helium and nitrogen in the pressure range 10^{-1}–10^{-3} Torr using a McLeod gauge, after which the McLeod gauge was isolated from the adsorption system.

Some typical results are shown in Fig.

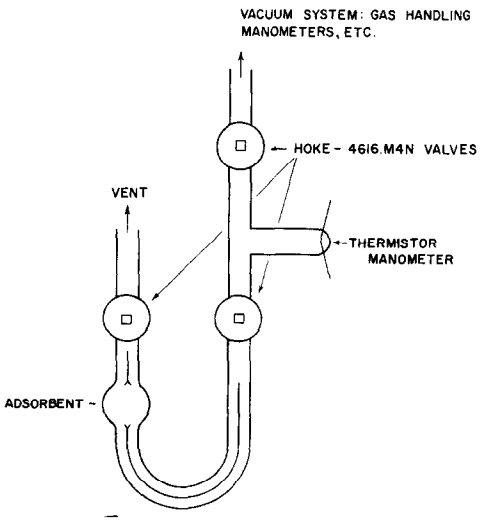

FIG. 1. The adsorption cell and doser.

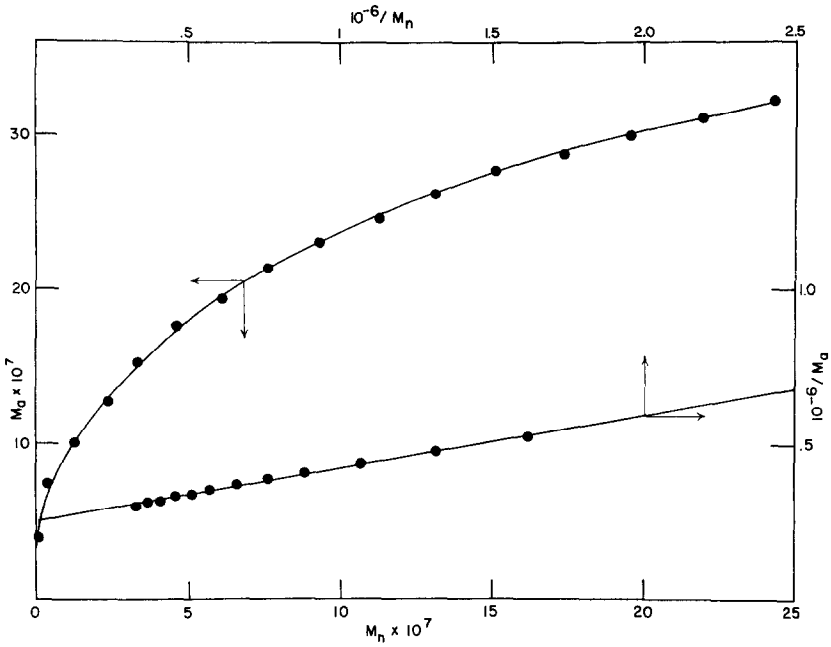

FIG. 2. Typical results for a gold catalyst.

2. The catalyst, a 2.98-g sample of Johnson Matthey "specpure" gold sponge was installed in the adsorbent cell and pretreated in flowing hydrogen at 500°C for 1 hr, after which it was pumped to less than 1×10^{-6} Torr. The dead volume was found using helium and the surface area with nitrogen, with the gold at 77°K; typical initial pressures in the doser prior to expansion into the adsorption cell were approximately 4×10^{-3} Torr (corresponding to an off balance of the thermistor bridge of about 0.5 mV).

The results are displayed as a plot of moles of nitrogen adsorbed (M_a) vs equilibrium moles of nitrogen in the gas phase (M_n) and since the pressures involved are so much lower than P_0, the surface area was estimated from a simplified form of the BET (*2*) equation, viz.,

$$\frac{M_a}{M_m} = \frac{KM_n}{1 + KM_n},$$

where M_m is the number of moles of nitrogen in a monolayer. The required inverse plot of $1/M_a$ vs. $1/M_n$ is shown in Fig. 2 from which M_m was obtained by the method of least squares and the surface area calculated as 0.15 m^2/g.

Although this method was devised to determine small surface areas, this has not proved to be a limitation, and the method is now being used for rapid routine adsorption studies in the author's laboratories, however, for adsorbents with much larger surface areas, the Langmuir plot used in this investigation is not applicable, and the complete BET equation must be used.

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