Volumetric Gas Adsorption Apparatus

for the Measurement of Physical Adsorption and Desorption Isotherms

Presented by Micromeritics Instrument Corporation

The illustration to the right represents a generic volumetric physical adsorption analyzer in its most elementary form. The critical components are:

1) Analysis manifold of accurately known volume and temperature
2) Vacuum system with valve to manifold
3) Source of adsorptive gas (typically, N_2) with valve to manifold
4) Pressure transducer and temperature sensor
5) Means for recording the signal from the transducer and temperature sensor
6) A sample tube of precisely known free or void-space
7) Sample tube connected to analysis manifold
8) Means to reduce the temperature of the sample when required, (typically to liquid nitrogen (LN_2) temperature).

Preparation

The adsorptive gas supply valve (3) is closed and the vacuum (2) and sample (7) valves are open allowing the manifold and sample tube to be evacuated. The sample tube is not in the cold bath, so the sample is at ambient temperature.

When the necessary vacuum is achieved, valves 2 and 7 close and the cold bath is raised, cooling the sample to the analysis temperature.

Charging the Manifold

Valve 3 is opened momentarily to charge the manifold to a pressure (P_m) slightly above vacuum, preparing the instrument to dispense a dose of adsorptive onto the sample. The quantity of gas (n_m) in the manifold can be determined from the universal gas law

$$n_m = \frac{P_m V_m}{RT}$$

Dosing

Valve 7 is opened allowing some of the gas to enter the sample tube.

Equilibration

Some quantity of gas (n_{ads}) will be adsorbed by the sample and removed from the gas phase. Pressure is monitored until it stabilizes, indicating adsorption has equilibrated. The equilibration pressure (P_e)is recorded.

Quantity Adsorbed

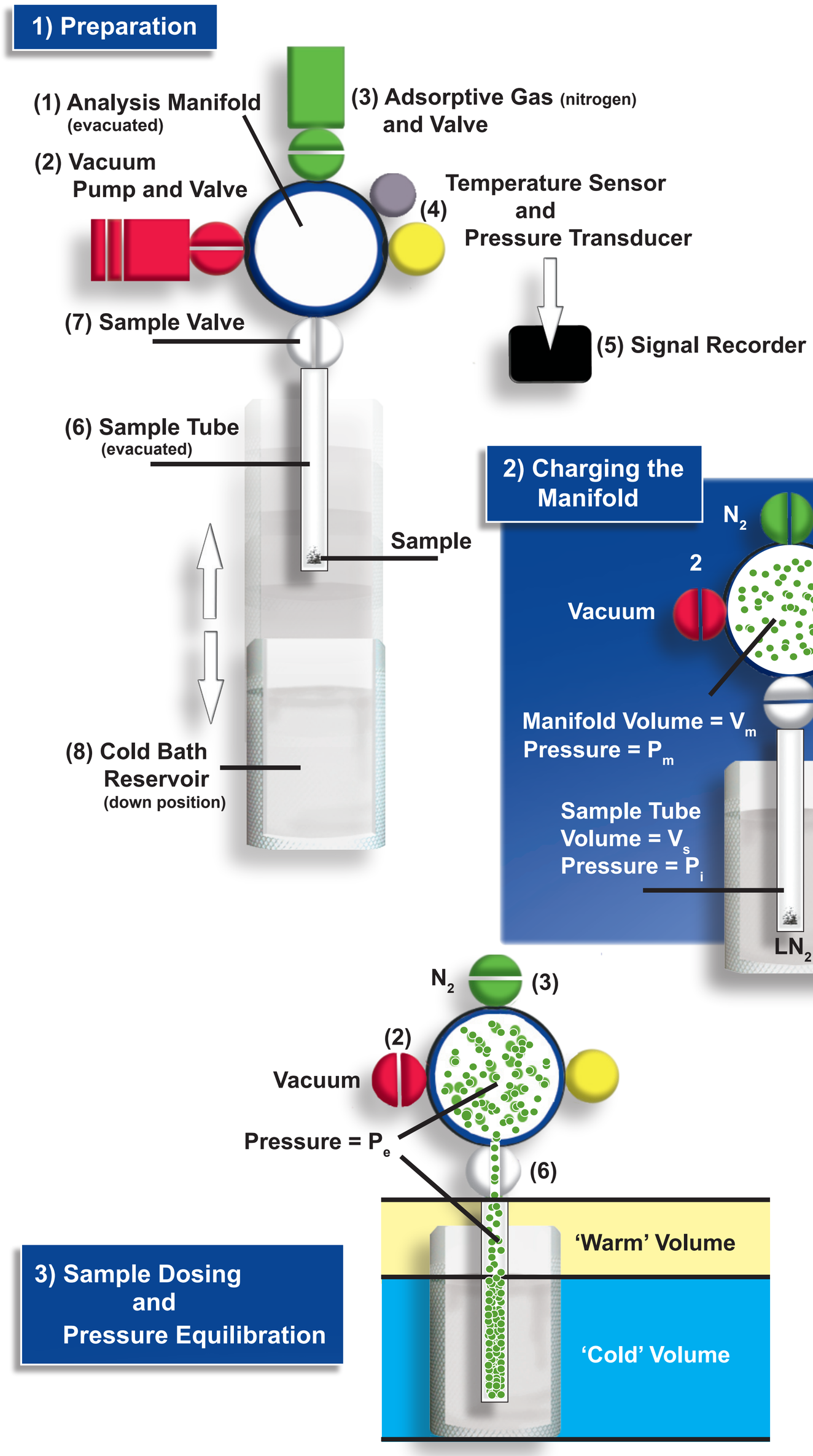
The quantity of gas (n_e) remaining in the combined manifold and sample tube volume ($V_m + V_s$) can be calculated from the universal gas law. This is complicated by the vertical temperature profile in the sample tube, one portion essentially being at ambient temperature and another portion being at the temperature of the cold bath typically LN_2.

The calculation of n_e is made traceable by a free-space measurement typically performed prior to the analysis and which characterizes the sample tube volume in regard to 'warm' and 'cold' volumes. Once n_e is determined, the quantity of gas adsorbed by the sample at P_e is

$$n_{ads} = n_m - n_e$$

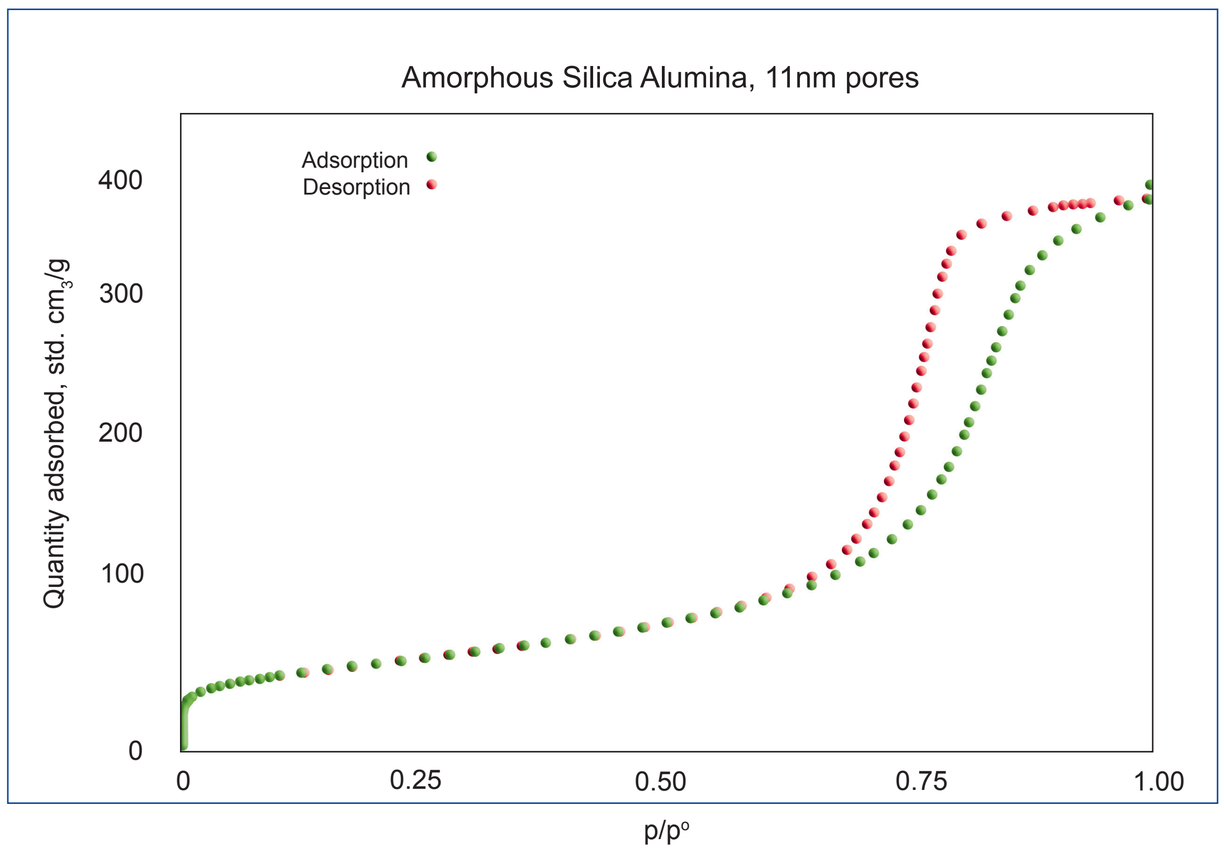
This establishes the point on the isotherm (P_e, n_{ads}). Valve 7 closes and valve 3 opens, and the manifold is charged to a pressure slightly higher than P_e after which the dosing and quilibration processes are repeated.

This cycle continues until the analysis pressure is near saturation pressure at which time the complete adsorption isotherm has been developed. The desorption isotherm is measured by a step-wise reduction in pressure until the a low pressure over the sample is achieved. At that point, most of the physically adsorbed molecules will have been desorbed from the surface.



Type IV isotherm:

Nitrogen adsorption and desorption on amorphous silica alumina.



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Micromeritics Instrument Corporation
One Micromeritics Dr., Norcross, GA 30093-1877, USA
US Sales: 770.662.3633, International Sales: 770.662.3660
Customer Orders: 770.662.3636, Fax: 770.662.3696

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