**H-Sorb 2600** The Ultimate Equipment for High Pressure Gas Sorption Analysis

**Introduction**

Gas sorption analysis is important in many fields of materials science and consumer product development. Some examples of current hot areas of technology involving gas-solid (or gas liquid) interactions include the development of energy storage materials, improved catalysts for petrochemical processing, advanced pharmaceuticals and food industries. At present, there is tremendous interest in the development of innovative materials for the transition to renewable energy. The science of gas sorption has become particularly critical in the advancement of materials for storing fuel gases such as hydrogen or natural gas, as well as for sequestration of greenhouse gases. Some exciting prospects for the chemical storage of gases include high-surface area and nanomaterials (graphitic carbons, CNT, zeolites, Metal-Organic-Framework MOFs). In the field of hydrogen-storage, a host of novel materials are being developed including high-pressure metalhydrides, light-weight complex hydrides, destabilized multicomponent chemical hydrides and amides, and other crossover materials such as ammonia borane encapsulated into mesoporous silica scaffolding.

Of prime interest in the discovery of these and many other new materials is the characterization of gas absorption, adsorption and desorption kinetics, capacity, thermodynamic properties, as well as cycling performance of reversible materials. Also important is the ability to measure the tolerance of catalyst, chemicals, and consumer products to air, moisture and low-level contaminants. These wide-ranging requirements and the need to perform analysis at both a research and a production level (milligrams to kilograms) have driven the development of the **H-Sorb 2600**, the ultimate tool for gas sorption analysis.
Careful analysis of physical and chemical interactions between gases and solids (or liquids) require extremely precise measurements. Several techniques are employed for gas sorption measurements the most common of these being gravimetric and volumetric methods. In volumetric methods the amount of gas sorbed is typically determined by a change in pressure within a calibrated volume containing the sample. In gravimetric methods, the amount of sorbed gas is determined by measuring the apparent mass change of the sample. In volumetric methods, the amount of gas sorbed is typically determined by a change in pressure within a calibrated volume containing the sample. We believe the volumetric method offers several distinct advantages over the gravimetric method, and of the volumetric instruments available, the Gold APP Instruments H-Sorb 2600 offers the best state-of-the-art measurements.

The Volumetric Method

One of the most common and versatile types of volumetric instruments is the Sieverts Apparatus. Simply put, a Sieverts Apparatus is an instrument containing two reservoirs of known volume connected by an isolation valve, as shown in Figure 1. The sample for measurement is loaded into the sample volume and the initial pressure reading is taken. For absorption the reservoir is filled with the sorption gas to a predetermined pressure above that of the initial pressure in the sample volume. The isolation valve between the two volumes is opened and the gas is allowed to equilibrate between the reservoir and sample volume. By knowing the initial gas pressures and the volumes of the system, the quantities of absorbed or desorbed gas can be determined.

Figure 1. Schematic of a Sieverts Apparatus.

By applying the Sievert’s approach to a system using a wide range of calibrated volumes, advanced pressure control, pressure measurements, and temperature control, it is possible to make a full suite of analyses with one device. Such analyses include:
Capacity

The total amount of gas absorbed or desorbed by a sample is pressure and temperature dependent and therefore requires precise measurements of both. In the volumetric method, capacity is directly related to pressure change. Small amounts of impurities in the test gas may be strongly absorbed by the sample. This can be very problematic in gravimetric methods as this uptake can cause significant weight change that might be misinterpreted. In the volumetric method, low levels of impurities may impact the performance of the material, but will not produce significant pressure changes.

Kinetics

Kinetics consists of the dynamic measurement of the change in moles of sorbed gas versus time. An advanced sorption analyzer should consist of multiple reservoirs with a wide range of volumes to match sample size and a wide range of sorption conditions associated with each different type of material. Additionally, sorption reactions generally involve significant endothermic or exothermic reactions. Heat transfer from the sample is a critical aspect of proper kinetics measurements. In a gravimetric instrument the sample is suspended from a microbalance that does not allow direct contact of temperature measuring probes or provide good heat transfer, limiting its usefulness.

Pressure-Composition Isotherms

Pressure-Composition Isotherms (PCT or PCI) is one of the most informative sorption measurements. The result is a plot of the equilibrium absorbed gas concentration in the material as a function of pressure and temperature.

The volumetric approach to PCI measurements consists of adding (or removing) gas to the sample volume in a small dose from one of the calibrated reservoirs and waiting for the resulting gas/solid equilibrium. The PCI curve is produced from the concentration of gas sorbed and the final pressure of each dose in a series of many doses that either increase (absorption) or decrease (desorption) in pressure. This is essentially a gas titration process.

The PCI plot will typically show an equilibrium plateau associated with the co-existence of the remaining unreacted material and the gas-reacted material, thus providing a complete phase diagram. For example, metal hydride materials that reversibly react with hydrogen show a well-defined plateau between the solid solution a-phase, where hydrogen is randomly dissolved within the solid matrix, and the b-phase, where hydrogen is in distinct structural sites and has bonded to the host solid material. In addition, precise dosing PCI measurements can provide detailed information on the presence of crystal structure changes, new phases and sorption kinetics as a function of gas concentration.
**H-Sorb 2600 high pressure gas adsorption parameters**

**Analysis Method:** static volumetric, high pressure gas sorption

**Versatility:** adsorption/desorption isotherms measurement (cryogenic to 500°C), Gibbs supercritical adsorption measurement

**Data Reduction:** Langmuir model regression isotherm, PCT(Pressure-Composition-Temperature) diagram/curve/isotherm measurement, Langmuir maximum adsorption constant L and adsorption pressure constant B measurement; Langmuir correction model loading-ratio correlation regression (LCR) isotherm; Ono-Kondo (OK) lattice model isotherm regression; adsorptive gas density and vapor-liquid equilibrium accuracy calculation under different pressures and temperatures

**Measuring Ranges:** continuous adsorption and desorption measurement from normal to 200Bar pressure

**Accuracy:** repeatability errors <3%

**Temperature Range:** cryogenic to 500°C, accuracy±0.1°C

**Sample Ports:** two samples analyzing and two degassing, stainless-steel micro welding sample cells, software integrated temperature PID adjustment

**Transducer Accuracy:** imported high precision transmitter, accuracy can reach 0.05% of FS. (full-scale), long-duration usage stability is 0.025% of FS.

**Ultimate Vacuum:** 4x10^-2 Pa (3x10^-4 Torr), molecular pump system (1x10^-6 Pa) is optional

**Adsorbate Gas:** high purity nitrogen, CO2 or others (Ar, Kr, H2, CH4 etc.)

**Data Acquisition:** high-precision and high integration data acquisition modules, minimal error, strong anti-interference ability

**Specifications:** Height 22.05 inches (56cm)* Width 20.87 inches (53cm)*Depth 23.62 inches (60cm); 132 pound (60kg); 220V/5A

**H-Sorb 2600 high pressure gas adsorption features:**

**Control System:** imported VCR interface high pressure pneumatic valve can realize auto on-off within 200Bar, sealing performance is top to 1x10^-10 Pa.m3/s, can be used more than 500 million times; programmable logic controller (PLC) system, high integration and sound anti-interference, improve stability and prolong life

**Analysis Management:** parameters be set by software, fully automated, unattended operation at night; H-Sorb mode is available for accurate control gas charging pressure points, gain ideal data points

**Manifold System:** imported 316L stainless steel thick-walled manifolds, micro welding sealing can reduce dead space largely; metal VCR connection is convenient for installation and uninstallation

**Safety Measures:** H-Sorb asymptotic technology realizes auto gas-charging and degassing, prevents high pressure dangers caused by artificial misoperation, and also minimizes large differential pressure's impact to pressure transducer