



The PMI Advanced ChemMaster Chemisorption and BET Sorptometer

Not just products...solutions!

DESCRIPTION

ChemMaster has been designed has versatile tool to investigate and extract information about surface chemistry and surface structure, which are important for design production, and application of a catalytic material. The PMI's ChemMaster measures the quantity of adsorbed or desorbed molecules allowing determination of these characteristics, all measurements are done quickly with high repeatability. Further BET Sorptometer is also provided to extend the surface area measurement capability for variety of materials.

PMI's BET Sorptometer is fully automated, volumetric gas sorption analyzer to measure accurately adsorption and desorption isotherms for the characterization of surface area, pore size distribution, pore volume and pore structure of microporous and mesoporous materials, as well as the kinetics of adsorption.

CHEMISORPTION

Any of a number of reactive gases such as anhydrous NH_3 , CO_2 , CO_1 , H_2 , N_2O_1 , O_2 and H₂S can be used to react with the active surface. A series of injections of a known quantity of reactive gas is injected into an inert gas stream that passes through the bed of catalysts. On the downstream from the reactor is a detector, which determines the quantity of reactive gas that is removed from each injection. Chemisorption tests ideally are made with the sample at a temperature such that only chemisorptions occurs. The active surface of the sample is saturated when the detector indicates that the total quantity of subsequent injections passes through the sample bed without any loss. The sum of the injected quantity minus the quantity of gas that passed without adsorption equals the quantity adsorbed. Unlike physical adsorption, the injected gas chemically adsorbs only on the active surface and not on the support. Thus, the number of gas molecules required to cover the active surface area, once determined, leads directly to the active surface area. Applying the stoichiometry factor for the gas/metal reaction yields the number of accessible atoms of active metal. Furthermore, using the total quantity of active metal per gram of catalyst material (determined from the manufacturing formula) leads to the determination of the percent dispersion of active metal. Using the information gathered plus the density of the metal, the size of the metal crystallite can be estimated if it is assumed that these particles have uniform geometry of known volume-to-area ratio.

PHYSICAL ADSORPTION

Weak van der Waal's type interaction of molecules with a pore surface leads to physical adsorption. The Brunauer, Emmett and Teller (BET) theory of physical adsorption is normally used for analysis of adsorption data to compute surface area.

$$\frac{P}{W(P_{o}-P)} = \frac{1}{CW} \frac{C-1}{mCW} \frac{P}{P_{o}}$$

Where:

W = amount of adsorbed gas

Wm = amount of gas adsorbed in a monolayer

P = gas pressure

P₀= equilibrium (saturation) vapor pressure at the test temperature

C = dimensionless constant that depends on the temperature and the gas/solid system

When vapor pressure, P is low compared with P_0 (0.05 < P/P₀ < 0.3), the plot of [P/W (P₀ – P)] verses [P/P₀] is linear and the plot yields the magnitudes of C and W. The surface area S per unit mass, m, of the sample is computed using the cross-sectional area of the adsorbed gas molecule:

 $S = \frac{W_m N_o a}{m}$

Where: N₀ = Avogadro's number a = cross – sectional area of the adsorbed gas molecule Wm = amount of gas adsorbed in moles

Pore Volume & Pore Diameter

Pore volume, pore diameter and pore volume distribution can be determined accurately by the PMI BET Sorptometer. The distribution function is such that area under the function in any pore diameter range is the volume of pore in that range.



Figure 1 Pore Volume Distribution

NSTRUMENT

The ChemMaster consists of :

- Dual ports, one for analysis and one for sample preparation.
- Built-in sample cooling fan, four carrier gas inlets, and one preparation gas inlet.
- ChemMaster can measure percent dispersion, active metal area, crystallite size, and quantify acid and base sites using pulse chemisorption. Physisorption tests include BET and Langmuir surface area, and total pore volume.
- Optional additional access port for mass spectrometer or other external detector for identification of desorbed species or reaction products.

The Measurement Technique

The ChemMaster utilize the dynamic (flowing gas) technique of analysis. The quantity of gas adsorbed from the gas stream by the sample is recorded by a downstream thermal conductivity detector (TCD). Generally the temperature and pressure at which adsorption/desorption occurs is either known or recorded. The instruments can be used to study physical or chemical adsorption. Preparation usually is accomplished by flowing either an inert or chemically active gas over the sample. After preparation, another gas is selected for analysis. Preparation and carrier gases typically used to allow both physical and chemical adsorption experiments are He, Ar, N, He/N₂ mixtures, H₂, and O₂, few gases work as both as both preparation and carrier.

ChemMaster Features:

- Analysis Port:1
- Preparation Port :1
- Sample Chamber: Quartz
- Gas Inlet for Carrier : 4
- Gas Inlet for Preparation : 1
- Temperature Control: Upto 400 Deg C
- Fan assisted cooling

Data Analysis Features:

- % Metal Dispersion
- Metal Surface Area
- Average Crystallite Size
- First Order Kinetics
- Single-Point Surface Area
- BET Multipoint Surface Area
- Langmuir Surface Area
- Total Pore Volume

Specification

Sample Characteristics:

- Pore Size Range: 0.35 to 500 nm or more
- Surface Area Range (in m²/g): 0.0005 and up for all the sample station
- Micropore Volume: Detectable within 0.0001 cc/g
- Pressure Transducer: 1 Sample Chamber 1000 torr
- Ultimate Vacuum: Up to 5×10⁻¹⁰ mbar
- Resolution: 1 part on 60,000
- Accuracy: 0.15% reading
- Analysis Gases: N₂, O₂, Ar, Kr, CO₂, CO, H₂, methane or any other non-corrosive gases
- Dead-end & Through-pores
- Adsorption Temperature: -195.60°C (Nitrogen)
- Regeneration System (Pretreatment system): Temperature Range: Ambient to 400°C
- Power Requirements: 220 VAC, 50 Hz

FEATURES

PMI's BET Sorptometer uses the same sample chamber for micropore & mesopore analysis. The same sample chamber is used for Outgassing & testing.

- In situ outgassing: (No need for extra outgassing stations)
- Automated Control
- Display of full adsorption and desorption isotherms
- Graph overlays
- BJH pore size distribution
- DFT pore size method
- as Plot
- f-Ratio plot
- Freundlich & Temkin isotherms
- Langmuir Surface Area
- Horvath-Kawazoe
- Dubinin-Radushkevich
- Dubinin-Astakhov
- Micropore size distribution (by Horvath-Kawazoe method)
- deBoer t-plot method (for measurement of micropore volume in cc/g & micropore area in m²/g)

FEATURES

Pore Structure Characteristics:

- Mean Pore Size
- Pore Size Distribution
- Total Pore Volume
- Single Point Surface Area
- Multi-Point Surface Area
- Adsorption & Desorption Isotherms
- Micropore
- Mesopore

SOFTWARE

Suitable Microsoft software and other interfacing & integration for carrying out the measurement and analysis with high levels of reliability and accuracy. Software has been capable of performing the following tasks:

- Generate both 'Single- and Multi- point BET specific surface area'
- Generate all the measured 'Adsorption and Desorption Isotherms'
- Langmuir surface area with slope, intercept, constant and correlation co-efficient
- Mesopore volume and Mesopore area distribution by BJH, HK and other models
- T-plot for micro pore area & volume
- Density functional theory to generate various parameters
- Micro pore and mesopore distributions

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