Application Notes

仪器的设计特征对应用的影响

摘要

化学吸附是一种高度专业的分析方法，因此，需要高度专业的设备。与表面积和孔隙率分析仪不同，化学吸附仪器涉及到化学反应。由于这些反应可能使用具有腐蚀性的气体，因此对仪器本身就存在着严格的要求。本文针对一些可能影响化学吸附仪使用的仪器的设计特征进行了一系列的讨论。便于您能更加了解您的设备，能够更大程度的利用仪器性能。

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Design Characteristics Impact Applications

Instrument Type: 2900,2000,2010

Technique: Chemisorption

Chemisorption is a highly specialized branch of materials analysis, and thus, requires highly specialized equipment. Unlike surface area analyzers and porosimeters, chemisorption analyzers must examine chemical reactions. Since these reactions may involve corrosive gases, harsh demands are placed on the analyzer itself. Perhaps the following discussion will help clarify these and other considerations that impact the utility of chemisorption analyzers.

Stainless Steel Tubing

The type of tubing used in chemisorption analyzers has a direct impact on the type of analysis that can be performed. Stainless steel tubing, for example, is impervious to the corrosive gases, (namely H_2S, NH_3, SO_2, and NO). This allows the scientist to carry out many types of analyses including determination of active site density, acid sites by ammonia titration, catalyst lifetimes, and catalyst regeneration.

Copper tubing has a different impact on the scientist's work. Copper will corrode when using the corrosive gases. Ultimately, the corrosion process interferes with the analysis in that the data will incorporate some unspecified amount of chemisorbed species from the copper tubing. This shows up in the graphs as delayed and distorted peaks. Under these circumstances, the types of analysis that can be performed are severely limited.

Degassing *In Situ*

Most chemisorption studies are especially sensitive to air contamination. Even a small amount of oxygen, like a few parts per million, will reoxidize a reduced (degassed) sample and cause false data. This occurs if the preparation and analysis ports are separated. Simply moving the sample tube from the preparation port to the analysis port introduces some amount of air and reoxidizes the sample just prior to analysis.

Some analyzers use a one-port design allowing sample preparation to occur *in situ* . By turning a valve, the preparation port is converted into an analysis port, thus preventing air contamination. This means critical chemisorption studies requiring thoroughly degassed samples can be accurately preformed.

Calibration

Chemisorption analyzers can be calibrated by either a manual syringe or by a built-in calibration loop. The significance of these methods lies in the degree to which they contribute convenience and accuracy.

For instance, calibrating by syringe injection is simple enough to perform, but hazardous when using noxious gases like CO and NO. The operator either siphons the gas from an air-borne stream or uses a septum<197>both methods are potentially dangerous. Taking precautions against exposure to the gas add inconvenience to the technique. Also, this method is difficult to reproduce precisely in that an accurate volume is dependent upon the hand-to-eye coordination of the operator.

The built-in calibration loop solves the problems associated with syringe calibration. Use of noxious gases is not restrictive since the calibration involves a closed loop. Gas cannot escape into the air. Furthermore, this method supports chemisorption studies that require exact calibrations each and every time as is necessary for most R&D applications.

Thermostatically Controlled

Thermal Conductivity Detectors (TCDs) are designed to respond to slight changes in the concentration of a given gas mixture. The degree of change will reflect the amount of gas adsorbed by the sample. But, the TCDs are so sensitive to temperature changes that they will also respond to changes in ambient or room temperature. Consequently, these ambient temperature fluctuations can interfere with the analysis.

A change in room temperature during a working day results in a temperature-induced drift; that is, the baseline shifts downward or upward. Data generated from an offset baseline is not useful.
-Chemisorption analyzers designed with thermostatically controlled TCDs maintain a leveled baseline so that the scientist can focus on the analysis instead of the baseline.