Active Materials, LLC
5755 Industrial Place, Suite A
Colorado Springs CO 80916
+1-719-966-429
www.active-materials.com

The chemistry you trust. The support you need.™

Tech Note	Getter Sorption Testing

Introduction

This tech note discusses two methods of getter sorption testing. The first method is used to measure the sorption capacity and rate of a hydrogen getter. Please note that while it is not applicable to all types of hydrogen getters, it is useable for many of the hydrogen getters in common use like metal oxides. The second method is used to measure the sorption capacity and rate of a water getter. These methods are designed to be simple to use and not require expensive equipment or dangerous test gases.

This tech note is presented as a simple introduction to these test methods and does not enter into the details of design, implementation, and interpretation. Please contact us if in depth discussion is desired.

The Delta P Test Method for Measuring Hydrogen Getter Performance

Hydrogen getter sorption rate and capacity data can be generated by the delta P, or change in pressure, method. This method is applicable to all hydrogen getter sorption mechanisms and most hydrogen getter systems in common use, except for hydride types that need thermal activation in a vacuum.

This method quantifies the performance of a getter by measuring the change in pressure in a known volume during the gettering reaction. If hydrogen is sorbed by the getter in a fixed volume, the Ideal Gas Law,

$$PV=nRT$$

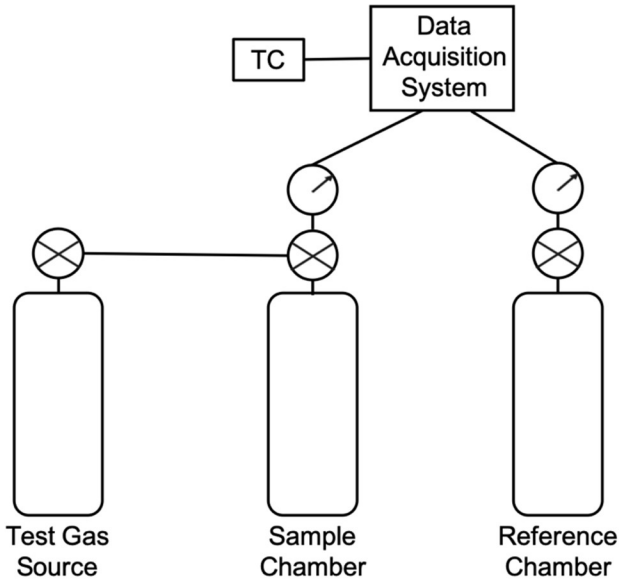
gives the amount of hydrogen sorbed by simple calculation:

$$n=PV/RT$$

The pressure change can be determined by comparing the difference in pressures in two known volumes or by using a sensitive capacitance manometer on a single known volume.

For this discussion, we will discuss the two known volumes method. The system is designed to simultaneously monitor the difference in pressure between the sample test chamber and the reference test chamber. The reference chamber is not exposed to the test gas or the atmosphere and serves as a constant pressure reference that only varies due to any changes in the external ambient temperature in the laboratory. Of course, these temperature changes also

impact the sample chamber equally. In the instance of a single test chamber system the reference chamber is typically replaced with a MKS Baratron or similar precision capacitance manometer. An example of such a system is offered by Oneida Research.*



Two Chamber Delta P System

Delta P systems also typically monitor the ambient temperature in order that the data can be normed to Standard Temperature and Pressure (STP) if desired. Ambient atmospheric pressure is not monitored as the system is isolated from it by the test chamber and the reference chamber if present. In the instance of a single chamber system, it is important to log the starting pressure as well as the completion pressure.

The change in pressure between the reference chamber and the sample chamber, the sample mass, the temperature of the system, and the concentration of the test gas used allow calculation of the getter capacity and speed using the Ideal Gas Law:

$$PV=nRT$$

in the form of

$$n=(P_1-P_0)V/RT$$

Where:

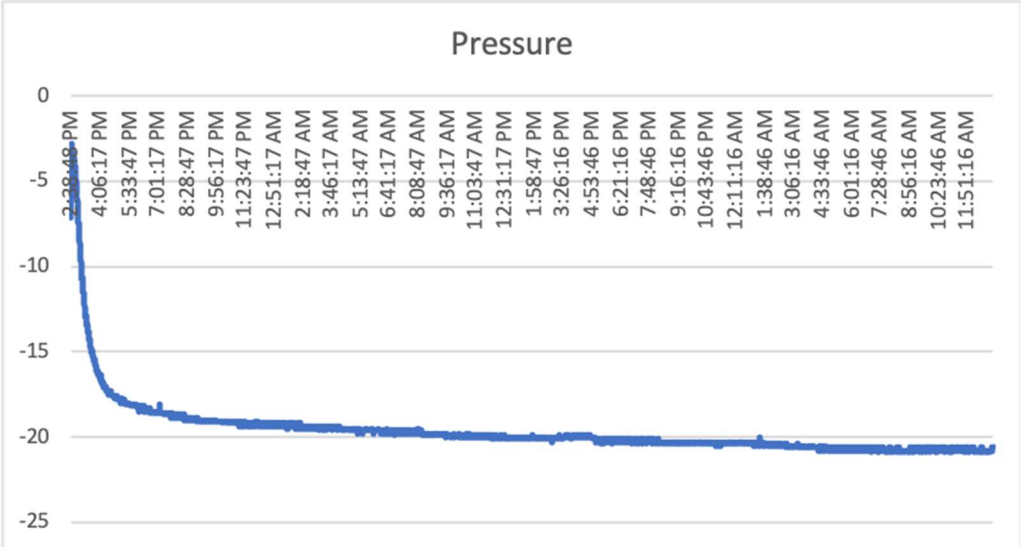
n is the number of moles of hydrogen sorbed
P_0 is the starting pressure differential between the two chambers
P_1 is the pressure differential between the two chambers at the end of the test
P_1-P_0 is the change, or delta, in pressure between the sample and reference chambers

* https://orslabs.com/test-equipment/delta-p-model-410-getter-sorption-analyzer/

V is the volume of the sample chamber
R is the gas law constant
T is the temperature at which the test is performed

Depending on the needs of the user, the results can be reported in various units to include moles and various versions of pressure-volume (PV) units including standard cubic centimeters (scc), torr-liters (t-l), torr-cubic centimeters (t-cc), or other variations.

The data acquisition system samples pressure and temperature at a designated sampling rate. The data points are used to generate a sorption curve, which shows the decrease in the pressure differential over time. This is a negative number indicative of the sorption of hydrogen from the sample chamber. The change in pressure over time can be used to calculate the sorption rate over any time period of interest during the test by calculating the slope between two data points. Depending on the getter's chemistry and bill of materials other information can be inferred from a sorption curve. However, such inference is beyond the scope of the current discussion.



Example Sorption Curve

As the getter sorbs hydrogen from the test gas the pressure in the sample chamber drops.

Safety Note Regarding Hydrogen Sorption Test Gases

There are different schools of thought regarding the hydrogen concentration to be used for the test. Before deciding on what concentration is used, it should be kept in mind that the flammability limits for hydrogen in air at 1 atm are 4 and 75%. Active Materials strongly urges working with hydrogen concentrations below the lower flammability limit in order to prevent accidents. Active Materials performs hydrogen sorption testing using a mixture of 3% hydrogen, balance argon.

Water Vapor Impacts on Measuring Hydrogen Sorption

Some types of hydrogen getters generate water vapor as part of the reaction. In these instances, the water vapor generated can mask the hydrogen sorption measurement. In the instance of a getter formulation inclusive of a desiccant, this masking does not occur, so long as the getter is properly formulated and activated.

There are getter applications where the water vapor that may be generated is not of concern. Getters for these applications can be formulated for hydrogen sorption only. It is convenient for testing purposes of the test to include a small amount of desiccant in such instances to simplify the analysis of the test results by removing water vapor from the system.

Water Sorption Quantity by Gravimetric Analysis

It may be desirable to easily check the capacity of a desiccant or determine the amount of water vapor generated during a sorption reaction. For example, water vapor sorption can be used as a cross check on the amount of hydrogen sorbed from a system in the case where the active hydrogen gettering agent used generates water vapor as part of the gettering reaction.

In order to determine the amount of water vapor generated, simple gravimetric analysis can be performed by measuring the mass of either the getter formulated with a desiccant or a separate desiccant introduced into the delta P test system before and after the test is performed using an analytical balance. The difference in mass is due to any water vapor generated and simple calculation will tell you how many moles of hydrogen were consumed to generate the amount of water sorbed by the desiccant. Water vapor sorption rates can be determined by taking mass measurements at intervals during the sorption process if necessary.

General discussion of gravimetric analysis can be seen at:

https://en.wikipedia.org/wiki/Gravimetric_analysis

Third party delta P testing of hydrogen getters can be performed by Oneida Research:

https://orslabs.com/services/rga-package-gas-analysis/getter-sorption-testing/

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