HORIBA Scientific

Application Note

BET Surface Area Determination

ADS129

A Comparison of Single-Point Surface Area Measurement to Multi-Point Determination

Introduction

The Flowing Gas Technique for determining BET Surface Area has been in use for over 70 years. Many facets of the technology make it a very attractive alternative to the Static-Volumetric approach.

First and foremost is the fact that the detection is done by measuring a gas concentration difference instead of an absolute pressure. Difference measurements are typically more accurate than many absolute measurements.

The speed of analysis and the resulting high sample throughput is also quite attractive. As a result, for routine QA/QC analysis, there has been a renewal of interest in this technique.

Analytical Test Method

The analytical technique is very easy to understand as it has great similarity to chromatographic determinations. The change in the ratio of nitrogen to helium gas flowing over the sample is measured by a thermal conductivity detector. The trace of the output from the detector is shown below in Figure 1 for the measurement of a silica sample. This shows the automatic calibration injection followed by an adsorption peak which is then followed by desorption.

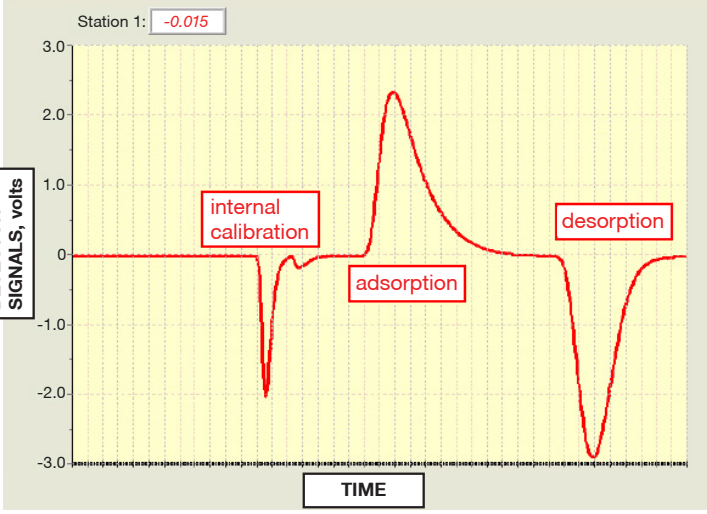

Figure 1.

In the SA-9600, the measurement typically involves the automatic integration of the area under the desorption peak. The lack of long waiting times following dosing, as happens in the Static-Volumetric case which also requires many doses even for a single-point analysis, makes this a very popular tool in a production environment.

One of the key questions is, what is the best way to implement this technology? For speed, a single-point measurement is typically completed in less than five minutes. For greater accuracy, a multi-point determination can be completed in less than 15 minutes. The chart below (Figure 2) compares these two types of analyses for several real-world samples.

	Single-Points	Multi-Points	Difference
Silica	4.30	4.23	-1.6%
Silica	22.99	23.43	1.9%
Carbon Black	97.85	101.59	3.8%
Carbon Black	208.16	207.71	-0.2%
Carbon Black	499.19	520.48	4.3%

Figure 2. Summary of data.

Conclusion

As can be seen, the single-point value is very close to the multi-point value, but typically a bit lower. This is the direct result of the assumptions that are used in the single-point equation. In a production environment, that difference proves to be completely negligible.

What is key is to always measure the samples in a repeatable manner; set an acceptable tolerance on the average value; and check each batch to be certain that it is within these tolerances. The decision between using a higher or lower number becomes a moot point as the change from batch to batch is the critical control parameter. As a result, the final report is commonly produced in less than 5 minutes.