

Product Sales Announcement

Product: AutoFlow BET+™

Date: June 9, 2017

PSA#

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NEW STOCK CODES

12 New Models:

02160-110-1ST-1
02160-110-1ST-1HT
02160-110-2ST-1
02160-110-2ST-1HT
02160-110-3ST-1
02160-110-3ST-1HT
02160-220-1ST-1
02160-220-1ST-1HT
02160-220-2ST-1
02160-220-2ST-1HT
02160-220-3ST-1
02160-220-3ST-1HT

4 Ship Kits:

03160-AF-BET
03160-AF-BET-HT
03160-AF-CPU
03160-AF-CPU-HT

KEYWORDS:

BET Analysis
Dynamic Flow
Surface Area
High Throughput
Krypton Adsorption
Statistical Thickness STSA
US Pharmacopeia

EXISTING PRODUCTS IMPACTED:

The **AutoFlow BET+™** is expected to replace the Monosorb in labs requiring high throughput surface area analyses.

SUMMARY:

The **AutoFlow BET+™** is a fully automated dynamic flow surface area analyzer designed to maximize analysis speed and throughput.

The instrument consists of one control module and 1, 2 or 3 independent analysis stations. The control module houses three independent degassing stations and a touchscreen. Each analysis station can perform preset or user-programmable Single Point BET analyses as quickly as < 5 minutes per sample, and Multipoint BET analyses as quickly as <15 minutes per sample. Higher throughput is attained by combining additional analysis modules: a set of 3 samples will generate three simultaneous Single Point BET and Multipoint BET analyses in as little as 5 and 15 minutes, respectively.

BENEFITS SUMMARY:

The **AutoFlow BET+** is ideally suited to perform very fast, precise and high throughput BET surface area analyses of solid samples.

Easy to use, compact, and free of vacuum pump noise and maintenance, the **AutoFlow BET+** can potentially analyze up to 36 samples per hour.

Such high throughput is of great benefit to highly dynamic QA/QC and Central Analytical laboratories, where rapid analysis turnaround is essential to ensure high product quality, minimize manufacturing process adjustment costs, and ultimately maximize profits.



Figure 1. **AutoFlow BET+™** with a Control Module (Far Left) and Three Independent Analysis Stations.



Figure 2. **AutoFlow BET+™** with a Control Module (Far Left) and Three Independent Analysis Stations.

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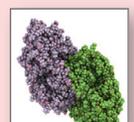
Catalysts



Ceramics



Energy



Carbon



Pigment



Pharma

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POSITIONING & SALES STRATEGY:

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OVERVIEW.

The **AutoFlow BET+™** is arguably the *highest throughput surface area analyzer available in the market today*. The instrument includes a control module (with three independent degassing stations) and 1, 2 or 3 independent analysis stations (see Figures 1 and 2). Single point BET surface areas can be evaluated in <5 min per sample. A three-station **AutoFlow BET+™** triples this output to as many as 36 sample analyses per hour, yielding an average of <2 min per sample. This translates to as many as 36 sample analyses per hour.

This outstanding analysis throughput is equally extended to Multipoint BET determinations. For instance, 3-point BET analyses for single samples can be collected in <15 min per sample. A three-station **AutoFlow BET+™** could thus yield 3 multipoint BET analyses in <15 min, or up to 12 multipoint BET analyses per hour.

Such a high analysis throughput would of course need to be paralleled by high throughput sample preparation (degassing) capabilities. So each **AutoFlow BET+™** includes three independent degassing ports (see Figures 1 and 2). Each degassing port has its own heating mantle and flow regulation valve, and can be independently programmed with up to six degassing steps (each step including target temperature, ramp rate and hold time).

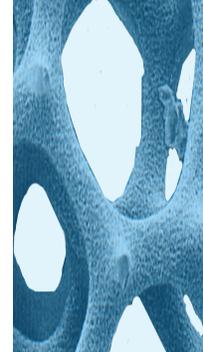
Standard degassing temperatures up to 350°C are standard, but high temperature (HT) **AutoFlow BET+™** models extend the upper limit to 450°C using quartz heating mantles and glassware.

The **AutoFlow BET+™** is ideally suited to perform very fast, precise and high throughput BET surface area analyses of solid samples. This is of great benefit to highly dynamic QA/QC and Central Analytical laboratories, where rapid analysis turnaround is essential to ensure high product quality, minimize manufacturing process adjustment costs, and ultimately maximize profits.

- Easy to use
- Compact
- Free of vacuum pump noise
- Free of vacuum pump maintenance

Suggested positioning strategies for the **AutoFlow BET+™** :

- (1) In existing Monosorb markets;
- (2) against dynamic flow BET competitors; and
- (3) against vacuum volumetric BET products.



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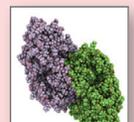
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1. AutoFlow BET+ Positioning as a Next Generation Monosorb.

For decades the Monosorb dynamic flow analyzer has enjoyed wide acceptance in high throughput markets where Single Point BET (SP-BET) approximations were adequate for QA/QC purposes. In those markets, the ability to generate SP-BET approximations in < 5 minutes per sample outweighed the inaccuracies introduced by the SP-BET method assumptions (e.g., SP-BET surface areas ~2% lower than the more accurate MultiPoint BET areas (MP-BET) for materials with BET "C" constants of ~100 [refer to Lowell et al.'s "Characterization of Porous Solids and Powders" 2010 book for detailed discussions of SP-BET and MP-BET analyses]). SP-BET results could be generated quickly, reproducibly, and easily, on the robust and time-tested Monosorbs. However, Monosorbs were long overdue for modernization. The **AutoFlow BET+™** units can be introduced to existing Monosorb customers as a modern, improved, and much more capable version of their Monosorbs. **AutoFlow BET+™** units can provide the following distinct advantages over Monosorbs:

(a) Faster SP-BET analysis results overall, because:

(i) Users will not need to wait 20-30 minutes for instrument warm-up every day (or even turn off/on their instruments each day), and can start tests as soon as their degassed samples are ready (e.g., after programmed overnight preparation and purging).

(ii) Users do not need to spend time zeroing and recalibrating the TCD signal responses with manual syringe or loop injections, since the **AutoFlow BET+™** responses are internally pre-calibrated to read gas concentrations directly.

(iii) Users do not need to purchase certified gas mixtures (e.g., 30% N₂ in He), since the built-in mass flow controllers of the **AutoFlow BET+™** enable them to use pure gases instead, or they can continue to use their pre-calibrated 30% N₂/He gas mixtures if so desired.

(b) Fast and more accurate multipoint BET analyses, compared to reproducible but less accurate SP-BET results. High accuracy would be most advantageous when comparing analysis results of a material in different laboratories. Each lab may produce highly precise (or highly repeatable) SP-BET areas, but each lab could potentially produce different SP-BET areas due to the assumptions inherent in the SP-BET method. Reporting MP-BET areas removes the uncertainties related to SP-BET assumptions.

(c) Confidence that the highly sensitive MEMS-TCD detector will not be damaged, as conventional TCD detectors are, if for any reason the flow of inert gases was interrupted (say, if a tank ran out of gas), and air slowly reached and oxidized the energized TCD filaments (thus avoiding potential downtime and repair costs).

(d) Additional technical capabilities that go well beyond standard SP-BET analyses (see item 2 below).

The equivalence of Monosorb vs. **AutoFlow BET+™** analysis results is demonstrated in Figure 3.

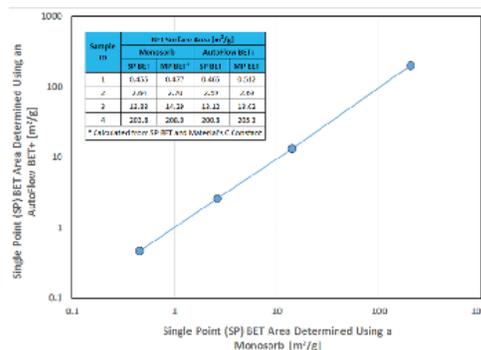
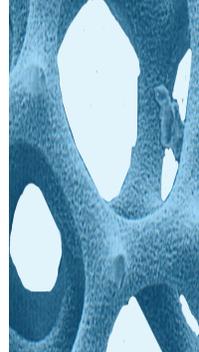
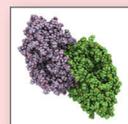


Figure 3. Comparison of Industry Standard (Monosorb) vs. Advanced **AutoFlow BET+™** Surface Areas for Selected Alumina Samples. Excellent agreement is obtained in all cases. Note: Unlike the **AutoFlow BET+™**, the Monosorb only measures single point BET (SP BET) surface areas. Multipoint BET (MP BET) Surface Areas for the Monosorb were calculated from SP BET values and average BET C constants for the materials tested.



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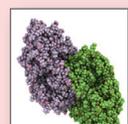
Catalysts



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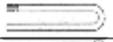
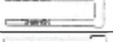
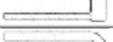
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However, one limitation of **AutoFlow BET+™** vs. Monosorb units should be noted: Due to space constraints in the 3-station degasser ports, the **AutoFlow BET+™** heating mantles cannot accommodate the larger or 2-piece sample cells that some Monosorb clients may use on occasion. The types of sample cells that are available for use in Monosorbs and **AutoFlow BET+™** units are listed in **Table 1**.

Table 1. Sample Cells Available for Use with Monosorb and **AutoFlow BET+™** Analyzers.

P/N	Description	Image	Can be Used with This Instrument	
			Monosorb	AutoFlow
74000	Standard Flow Cell		Yes	Yes
74001	Microcell		Yes	Yes
74002	Capillary Flow Cell		Yes	Yes
74003	Narrow Flow Cell Etc		Yes	No
74004	Wide Bore Flow Cell		Yes	Yes
74005-10	2pc 2-Piece Solid Bulk Flow Cell		Yes	No
74005-20	6pc 2-Piece Solid Bulk Flow Cell		Yes	No
74008	Monolith 2-Piece Flow Cell		Yes	No

2. AutoFlow BET+™ Positioning Against Competitors in Dynamic Flow BET Markets.

2.1. Established Dynamic Flow BET Markets. The dynamic flow technique is particularly popular in markets where:

(a) high analysis speed and throughput are important; and

(b) samples may be difficult to analyze by vacuum techniques (see Section 3).

Such markets include, but are not limited to, the following: Pharmaceutical, Food, Polymers, Metal Powders, Metal Oxides (Alumina, Silica, Zinc Oxide, etc.), Catalysts, Catalyst Supports, Ceramics, and Minerals.

Table 2 lists examples of industrial methods currently employed for the characterization of BET surface areas by the dynamic flow method. The particular case of the Carbon Black industry is discussed in Section 2.2.

Table 2. Selected Examples of Dynamic Flow Standard Test Methods for Various Materials.

Material(s)	Standard Method(s) ^a
Metal Powders	ASTM B922
Alumina, Silica	ASTM C1069
Advanced Ceramics	ASTM C1274
Catalysts, Catalyst Support	ASTM D4567
General	ISO 9277
Pharma (Active Pharmaceutical Ingredients (API) and Excipients)	USP <846> Method I, European Pharmacopeia 2.9.26 Method I

^aNote: Carbon Black Total (BET) and External (STSA) Surface Areas evaluated by ASTM D6556 can also be determined by the **AutoFlow BET+™** dynamic flow method and can yield equivalent results in extremely short analysis times.

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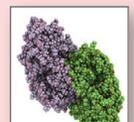
Catalysts



Ceramics



Energy



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Pigment



Pharma

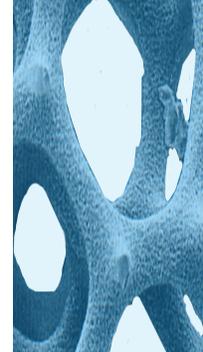
2.2. Dynamic Flow Methods for the Carbon Black Market.

Because of its high sales volume, the Carbon Black market should also be considered. In the past, dynamic flow methods (e.g., Monosorb and other chromatography-type techniques) were standard for the determination of surface areas of industrial carbon blacks (e.g., refer to ASTM Test Method D3037, withdrawn in 1999). At that time the introduction of fast and economical static vacuum techniques (such as MM's Gemini and QI's NOVAe) shifted the Carbon Black industry focus from Single-Point BET alone to Multipoint BET (MP-BET) and Statistical Thickness Surface Area (STSA) determinations. At that time, the Carbon Black industry adopted ASTM Method D6556, which only includes the static vacuum test method for BET and STSA determinations.

Regardless of the test method, STSA is simply the external surface area determined by the t-plot method, but using a t-curve customized for carbon black particles. In fact, the t-plot method provides both external/STSA areas and micropore volumes. Coupling these fast measurements with SP-BET and MP-BET areas makes the **AutoFlow BET+™** a very powerful characterization tool for the Carbon Black industry. We have therefore included STSA calculations in the **AutoFlow BET+™**, and expect the Carbon Black industry to shift their attention back to the dynamic flow method once the **AutoFlow BET+™**'s many advantages (fast speed, high throughput, operational simplicity, and ready availability of MP-BET and STSA values) are realized (see **Table 3**).

Table 3. Carbon Black Industry Reference Materials Tested Using an **AutoFlow BET+™**. **Table 3** lists average values obtained from five analyses of each reference material on an **AutoFlow BET+™**. Results equivalent to ASTM D6556 can be obtained with the **AutoFlow BET+™** in extremely short analysis times.

Sample ID	Surface Area Method	Surface Area Analysis Statistics					
		ASTM D6556-16			AutoFlow BET+™		
		Area [m ² /g]	Std. Dev. [m ² /g]	CoV [%]	Area [m ² /g]	Std. Dev. [m ² /g]	CoV [%]
SRB8-F	MP BET	36.7	1.3	3.6	36.5	0.5	0.2
	STSA	35.4	2.3	6.6	35.0	0.4	1.1
SRB8-G	MP BET	9.1	0.4	4.0	9.4	0.0	0.1
	STSA	8.4	0.6	7.1	8.6	0.1	1.3



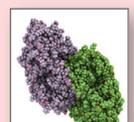
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2.3. Advantages of the AutoFlow BET+™ Over Competitors.

Based on information publicly available at this time, the **AutoFlow BET+™** is the fastest dynamic flow BET surface area analyzer available commercially today. Technical advantages over specific competitors are highlighted in the following subsections.

2.3.1 AutoFlow BET+™ vs. Horiba SA-9600 Series.

The Horiba SA-9600 Series is described in the following link:

<http://www.horiba.com/scientific/products/particle-characterization/surface-area-analysis/details/sa-9600-series-938/>.

The various **AutoFlow BET+™** modules provide distinct speed advantages over the Horiba products. This is in part due to the fact that Horiba analyses require an initial pulse calibration step. In contrast, the accurate pre-calibrations of the **AutoFlow BET+™** save time, and the responses of its unique MEMS-TCD sensors do not vary with environmental conditions or gradual changes as with conventional TCD filament performance.

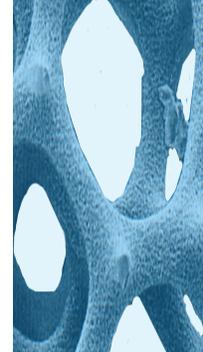
The **AutoFlow BET+™** also features automated gas flow path switching among three different paths (short, medium, and long path). The path required to minimize analysis time and optimize sensor performance is automatically decided by the instrument. The decision is made based on optional user input of the approximate/anticipated BET surface area in m²/g. Crude estimates are sufficient to ensure flow path optimization. This ability also makes the **AutoFlow BET+™** able to analyze sample surface areas between 0.05 and 350 m² in the cell, as opposed to only 0.01 to 50 m² in Horiba sample cells. Accordingly, **AutoFlow BET+™** users can confidently add much more sample to the cells in order to get more representative analysis results in optimum time.

AutoFlow BET+™ users can also extend the MP-BET test range to P/Po < 0.05, and as low as P/Po of 0.005. This feature is most useful for microporous/high surface area materials, since the MP-BET range of linearity for microporous materials often falls below the standard P/Po ~0.05-0.33 range.

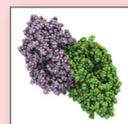
In addition, **AutoFlow BET+™** users can also collect data over a wide range of P/Po target pressures > 0.33, and thus generate STSA and sorption isotherm data as well.

Other advantages of the **AutoFlow BET+™** over Horiba competitors include:

- (a) programmable degassing steps for each station;
- (b) maximum degassing temperature of 350° C standard [450° C optional], vs. 300° C in Horiba units;
- (c) maximum degassing time limited to a maximum of 3 hours in Horiba units, vs. up to 500 hours on **AutoFlow BET+™** degassers;
- (d) software availability in up to 11 languages; and
- (e) built-in safety shielding doors to protect operators from potential cryogen splashes and moving system components (Dewar lift).



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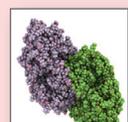
Catalysts



Ceramics



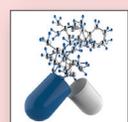
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2.3.2 AutoFlow BET+™ vs. JW-DA Analyzer (China).

The JW-DA Analyzer is described in the following link:
http://www.jwgb.cn/en/Products_View.Asp?ID=60.

The 4-Stations-in-Series JA-DA analyzer differs from the more limited JD-WX analyzer in one important respect: The JW-DX analyzer can only provide BET surface areas by comparison to the signal from a reference sample. In contrast, the JW-DA can perform BET measurements directly, but taking a relatively long time (25 minutes) per sample. The more rapid "Reference Method" can yield "reference" (vs. BET) surface areas in 5-8 minutes per sample, but the results are highly dependent on the characteristics (quality, homogeneity and repeatability) of the sample chosen as the "reference."

Besides having slower BET analysis times, the JW-DA and JW-DX units are not supplied with integrated degassers. This forces their clients to purchase separate degassers in order to use these instruments. The added cost and bench-space loss of having separate degasser dedicated to pre-treating samples are clearly undesirable.

2.3.3. AutoFlow BET+™ vs. Belsorp-MR6 Analyzer (Japan).

The Belsorp-MR6 Analyzer is described in the following link:

<http://www.microtrac-bel.com/en/product/surface/bel-sorp-mr6.html>.

The 6-Station-in-Series carousel design of the Belsorp-MR6 allows it to start batch analyses with up to six samples that can be pretreated and analyzed one-by-one. Even though a minimum analysis time of 10 minutes per sample is claimed, the parallel batch design prevents more than 6 degassed samples from being analyzed in less than 1 hour. The **AutoFlow BET+™** far exceeds this claimed throughput capacity.

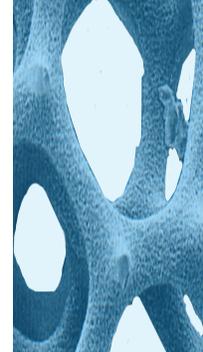
Besides having slower BET analysis times, a significant limitation of the Belsorp-MR6 is its being designed to yield Single Point BET results only, and only add Multipoint BET capabilities (and external gas mixers) as an option.

2.3.4. AutoFlow BET+™ vs. Other Dynamic Flow BET Competitors.

Additional instruments that may compete against the **AutoFlow BET+™** analyzer, albeit with more limited capabilities, include: the FlowSorb III by Micromeritics (more manually operated Monosorb competitor, with some versions excluding the automated Dewar lift mechanism), the Smart Analyzer (sold in India, with a claimed accuracy and reproducibility of only $\pm 5\%$ and $\pm 3\%$, respectively), and the Monosorb (designed as a single station, Single Point BET analyzer; see Section 1).

3. AutoFlow BET+™ Positioning of Dynamic Flow vs. Static Volumetric Analyzers.

The latest IUPAC recommendations for surface area evaluation (Thommes et al., Pure Appl. Chem. 87 (2015) 1051) continue to favor vacuum over flow techniques for physisorption analyses. Dynamic flow methods are deemed suitable for analyses when the adsorption of the carrier gas (helium) can be neglected. This is a reasonable assumption for helium, except for its potential entrapment in narrow micropores. To avoid such potential uncertainties, pore size distributions are preferably determined by static vacuum techniques (NOVA, QDS, ASiQ). However, the dynamic flow technique has been shown to provide very reliable BET analysis results. The dynamic flow method is generally faster, simpler, and cheaper to implement, than static vacuum techniques. This is why the **AutoFlow BET+™** may be most appealing to laboratories where high BET analysis throughput can improve their productivity.



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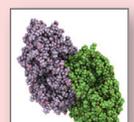
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Another advantage of flow vs. vacuum techniques stems from the sensitivity of some materials to pressure/vacuum swings. For example, in the pharmaceutical and food markets it is not unusual to encounter samples whose structure may change upon degassing and/or thermal cycling, or solids that have a non-negligible vapor pressure. These factors can complicate the attainment of repeatable vacuum conditions during sample preparation or analysis. In contrast, dynamic flow techniques target changes in the concentration of gases flowing through the sample. Once the TCD detects constant (baseline) gas concentrations, any deviation is attributed to adsorption or desorption, caused by, e.g., raising or lowering the liquid nitrogen bath, respectively. Therefore, even if the samples have not attained constant vacuum levels, they still can reach constant dynamic flow signals that permit the measurement of TCD signal deviations with good repeatability. This is why many organic materials, such as active pharmaceutical ingredients (APIs), or polymers are often easier to analyze quickly and reliably by dynamic flow methods.

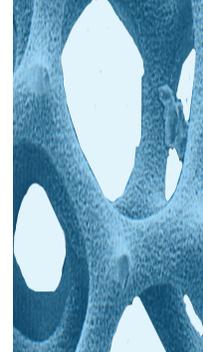
Other benefits of dynamic flow over static vacuum methods include the following:

- No vacuum pump required (minimizing noise, maintenance, and potential oil mist effects);
- No need for time-consuming void volume measurements;
- No need for gas non-ideality corrections;
- No need for bath coolant level control devices; and
- No cumulative errors, since each data point is independent of all others.

The **AutoFlow BET+™** also includes pressure and temperature sensors to correct all measurements for changes in P_0 and ambient conditions. All values are then conveniently reported at standard temperature and pressure (STP) conditions.

Field Upgrades.

The standard units (max. temperature = 350° C) can be field-upgraded to HT units (max. temperature = 450° C). The upgrade involves software changes and the use of quartz sample cells. The heating mantles do not need to be replaced during upgrades.



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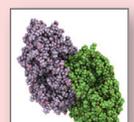
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