BET Specific Surface Area

Micromeritics TriStar II 3020

BET SURFACE AREA DATA SUMMARY

SAMPLE ID	SPECIFIC SURFACE AREA (m^2/g)	DEGASSING CONDITIONS			ADSORBATE
		DURATION	TEMPERATURE	TECHNIQUE	
Fe2O3 (Iron Oxide Powder)					
0338	106.13	24 Hours	100°C	Vacuum	Nitrogen

2023-05-16
CSL 2023-05-17

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INTERPRETING YOUR BET SPECIFIC SURFACE AREA ANALYSIS BY GAS PHYSISORPTION
(Static Volumetric Method)

Introduction

The surface area of your sample(s) has been measured via gas physisorption analysis, using the static volumetric method. For a typical multipoint determination, the amount of inert gas that adsorbs to the surface of a sample is measured at varying relative pressures. The surface area is calculated using the Brunauer–Emmett–Teller (BET) equation below:

$$\frac{1}{V_a\left(\left(\frac{P}{P_0}\right)-1\right)} = \frac{C-1}{V_m C} \; x \; \frac{P}{P_0} + \frac{1}{V_m C}$$

where $\frac{P}{P_0}$ = relative pressure,
V_a = volume of gas adsorbed,
V_m = volume of gas adsorbed to produce an apparent monolayer on the surface,
C = BET constant.

For the BET transform, the quantity $\frac{1}{V_a\left(\left(\frac{P}{P_0}\right)-1\right)}$ is plotted against the relative pressure (P/P_0). The BET plot should yield a straight line around the relative pressures where monolayer adsorption occurs, typically in the range of 0.05-0.3. The volume of gas needed to produce a monolayer on the sample surface (V_m) and the BET constant (C) are derived from the slope and y-intercept of the line from the BET plot. The specific surface area is determined using the calculated V_m, the cross sectional area of the adsorbate gas and the mass of the test powder.

The approximate BET surface area can also be determined at one single relative pressure only (commonly at $P/P_0 = 0.3$), either assuming the y-intercept of the BET plot is zero or using certain previously established parameters through multipoint determination for the sample type.

Sample Preparation

As the amount of gas adsorbed onto the sample surface is critical to the analysis, moisture and other impurities must be removed from the sample surface prior to analysis. This process, called degassing, may involve pulling vacuum on, flowing inert gas over the sample, or both. Degassing can also be done at different temperatures depending on the thermal stability of the material. It is extremely important to choose a temperature and technique such that the sample is not altered physically or chemically since either could affect the surface area of the sample. The degassing conditions used in your analysis are included on the preliminary results summary page and the final report.

The Data

The header section contains various user-entered information specific to each project. It generally includes the client name, sample identification, and analysis notes. Each project is given its own unique seven-digit code (PTL Project #) which can be found on these data pages. Please refer to this PTL Project # when contacting us with any questions regarding the analysis.

The data includes a BET tabular report and the BET plot. A table of the relative pressure and quantity of gas adsorbed at that relative pressure is provided in the tabular portion of the data. The last column on the right is the BET transform of the data, which can also be visualized in the BET plot.

Commonly reported values from your surface area data are explained below:

- **BET Specific Surface Area**: the surface area calculated based on the BET model normalized by the sample mass.
- **C Value (also called the BET constant)**: a value related to the affinity of the solid with the adsorbate gas. Since a negative C value has no physical meaning, the C value must be positive. A very low C value indicates the adsorbate gas did not interact well with the surface, suggesting the gas may not have formed a complete monolayer. On the other hand, a high C value may indicate a strong interaction between the surface and adsorbate gas, e.g. chemisorption sites or presence of micropores.
- **Correlation Coefficient**: a value that indicates the linear transform fit of the adsorption data. The BET surface area should only be evaluated over the linear region of the BET plot. At PTL, as per the current USP <846>, the correlation coefficient must be ≥ 0.9975 for data to be considered acceptable.

For additional questions specific to your analysis results, please contact us directly.

File name: 5919991A
Chemist: AL
Submitter: Particle Technology Labs
File: R:\Tristar II 3020\win3020\data\59199-91\5919991A.SMP

Started: 5/16/2023 2:15:46 PM
Completed: 5/16/2023 4:32:24 PM
Report Time: 5/16/2023 4:47:12 PM
Sample Mass: 0.1036 g
Cold Free Space: 40.1750 cm³
Low Pressure Dose: None
Automatic Degas: No

Analysis Adsorptive: N2
Analysis Bath Temp.: 77.350 K
Thermal Correction: No
Warm Free Space: 13.2812 cm³ Measured
Equilibration Interval: 20 s
Sample Density: 1.000 g/cm³

Comments: Fe2O3 (Iron Oxide Powder) 0338 PTL Project #59199-91 PTL ID:517609-91

BET Report

BET Surface Area: 106.1271 ± 1.4155 m²/g
Slope: 0.040225 ± 0.000536 g/cm³ STP
Y-Intercept: 0.000788 ± 0.000108 g/cm³ STP
C: 52.049675
Qm: 24.3826 cm³/g STP
Correlation Coefficient: 0.9999112
Molecular Cross-Sectional Area: 0.1620 nm²

Relative Pressure (P/Po)	Quantity Adsorbed (cm³/g STP)	1/[Q(Po/P - 1)]
0.055874329	19.7464	0.002997
0.173737743	26.7809	0.007851
0.297902997	33.3190	0.012735

2023-05-16
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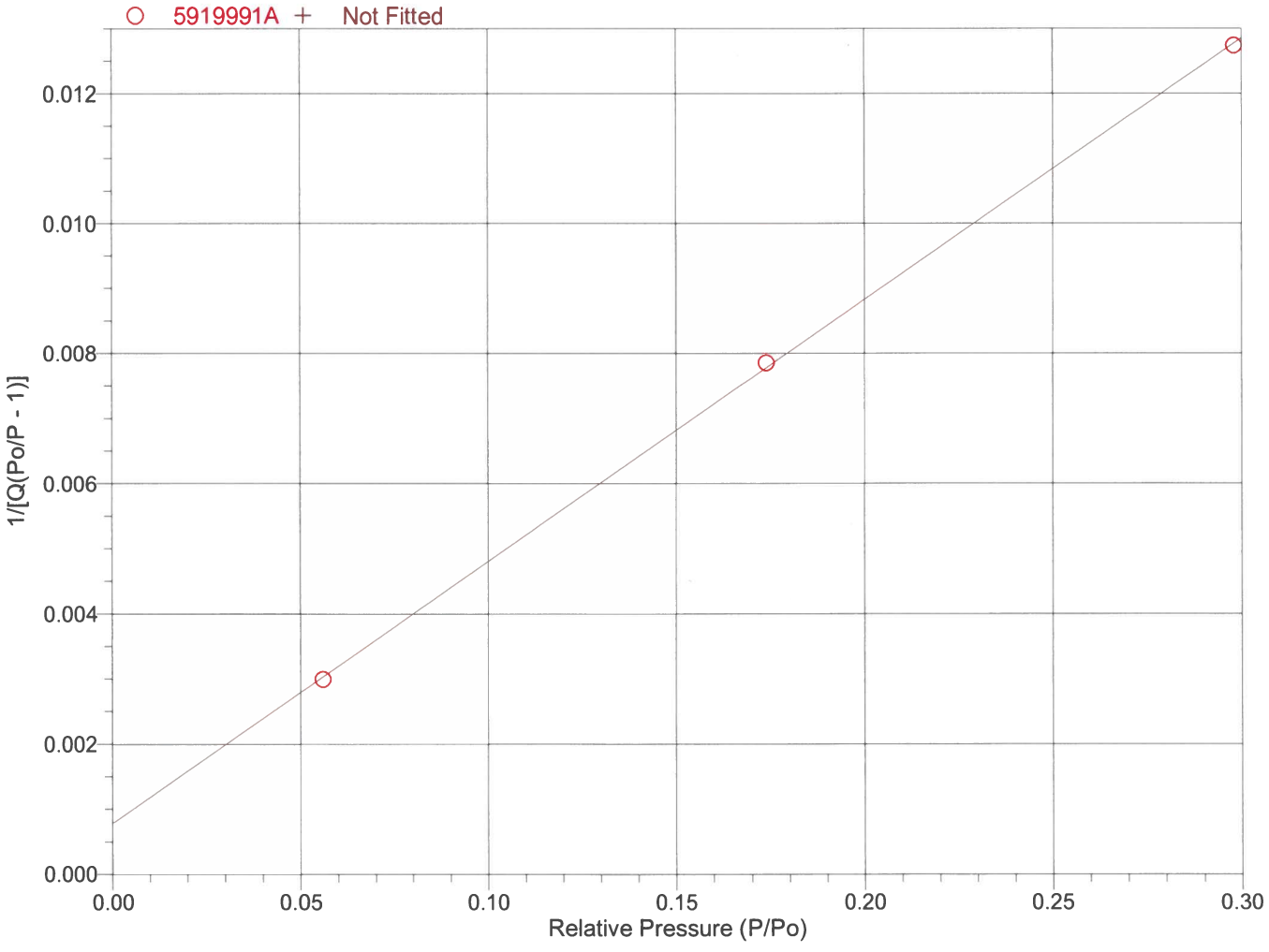
BET Surface Area Plot



Laser Diffraction

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INTERPRETING YOUR MALVERN MASTERSIZER FINE PARTICLE ANALYSIS

Introduction

The particle size analysis of your sample(s) has been conducted on a Malvern® MasterSizer LASER diffractor. This instrument is considered an ensemble analyzer that calculates a volume distribution from the LASER (**L**ight **A**mplification by **S**timulated **E**mission of **R**adiation) diffraction pattern of a suspension of particles. This raw scatter data are then processed using a complex algorithm and presented on the basis of **EQUIVALENT SPHERICAL DIAMETER**. The Malvern equipment currently in use at Particle Technology Labs (PTL) are the *MasterSizer 2000 and the MasterSizer 3000*.

The Data

The header section contains various user-entered information including client name, sample identification, and analysis notes. Each project submitted to PTL is given a unique seven-digit code (PTL Project #) which can be found on these data pages, although its location is dependent on the instrumentation used. On the MasterSizer 2000 and 3000 data, the PTL Project # appears at the bottom of the page and is identified as File Name or within the File. Please refer to this PTL Project # when contacting us with any questions regarding the analysis.

The data output also provides the parameters specific to the instrument being used, as well as the parameters specific to the analysis. The optical model (composed of the sample's refractive index as well as the imaginary absorption value) is required if determining the particle size using Mie theory. This theory can be used on any size particles, but is specifically recommended over the use of the Fraunhofer approximation according to ISO 13320:2020 for material in the size region less than approximately 50 µm. Please note that, unless otherwise requested or provided by the client, the analysis of an unknown material is generally conducted using the standard instrument default optical values as detailed below.

System Default Values

SETTINGS	MASTERSIZER 2000 / MASTERSIZER 3000	
	WET	DRY
Analysis Model	General Purpose	General Purpose
Presentation	Default	Default
Sample RI Value	1.520	1.520

The Fraunhofer approximation, which does not require specific knowledge of the optical properties of the sample, can be applied for large particles if a known refractive index of the material is not available or the sample is composed of multiple components. If the actual RI of the sample material is provided at a later date, the raw data can be recalculated to adjust for the refractive index.

Please note the following commonly reported values when reviewing your data:

- **Tabulated Data:** Always appears as a Cumulative % less than (*Volume Under %*) unless otherwise requested.
- **Span:** Value related to the width of the curve, expressed as $\frac{[Dv(90) - Dv(10)]}{Dv(50)}$ or $\frac{d(0.9) - d(0.1)}{d(0.5)}$ depending on the instrument.
- **D[3,2]:** Surface-weighted mean diameter (Sauter diameter)
- **D[4,3]:** Volume-weighted MEAN
- **10%, 50%, 90% size values:** Indicates the size median which 10%, 50%, or 90% of the particles within the distribution is smaller than (example: Dv(90) or d(0.9): 140 µm, this means that 90% of the particulate is smaller than 140 µm on a volume basis).
- **Specific Surface Area:** If a calculated Specific Surface Area value is reported, consider this value only as an approximate surface area since calculations are based upon **non-porous spheres**. It is not a replacement for a result produced from an actual gas adsorption instrument due to the above assumption.

Also included is the Particle Size Distribution DIFFERENTIAL HISTOGRAM. This histogram shows the Tabulated Data as a **Differential Volume Percent Less Than Indicated Size**. Please note the histogram for the *MasterSizer 3000* also generally includes a CUMULATIVE Curve representative of the Tabulated Data.

For additional questions specific to your analysis results, please contact us directly.

Malvern 3000 Liquid Analysis v.2

ptl Particle Technology Labs

Measurement Details	
Client	ENGINEERED DATA, LLC
Test Method	N/A
Operator Name	aredman
SOP File Name	HydroMV.cfg
Carrier	non-aqueous
Notes	N/A

Measurement Details	
Sample Name	Average of 'Fe2O3 (iron oxide powder)'
Sample ID	0338
PTL ID	517609-91
Analysis Date Time	5/16/2023 1:29:04 PM
Measurement Date Time	5/16/2023 1:29:04 PM
Result Source	Averaged

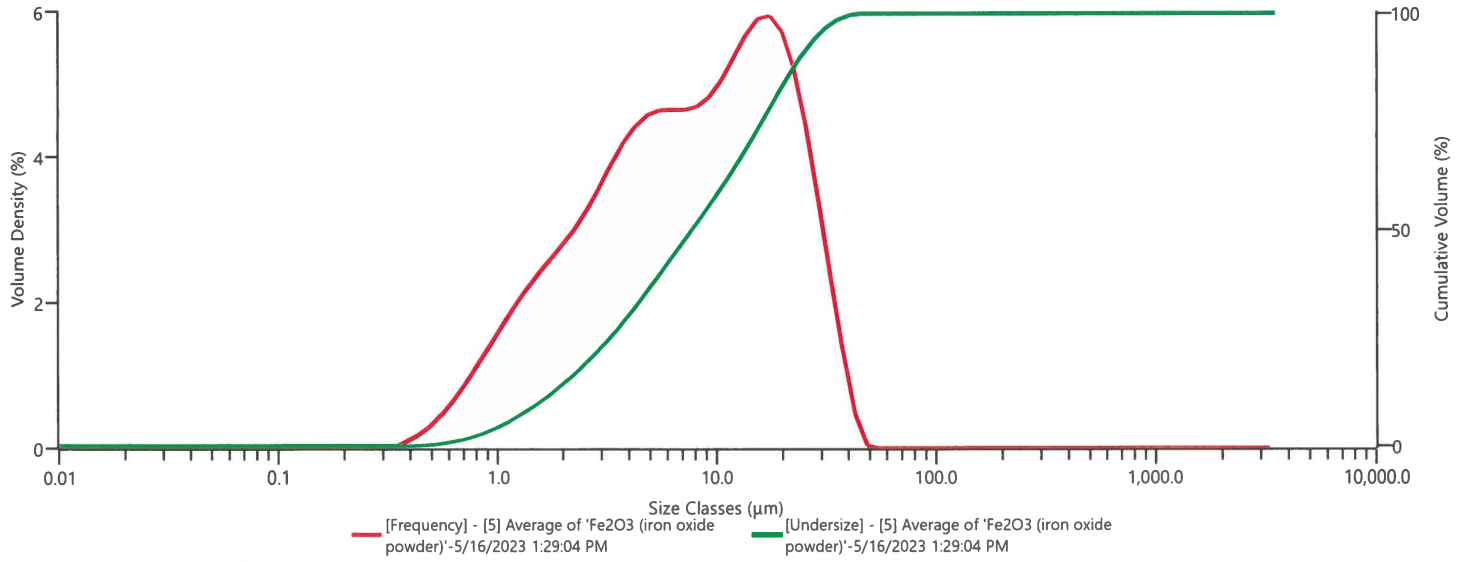
Analysis	
Particle Name	Iron III Oxide Fe2O3
Particle Refractive Index	2.918
Particle Absorption Index	1.000
Weighted Residual	0.51 %
Laser Power	79.74 %
Laser Obscuration	8.81 %
Accessory Name	Hydro MV
Software Version	3.88.2211.150

Result	
Dispersant Name	In House Hydrocarbon
Dispersant Refractive Index	1.420
Analysis Model	General Purpose
Analysis Sensitivity	Normal
Are particles non-spherical?	Yes
Scattering Model	Mie
Accessory Serial No.	MAL1137700
Instrument Serial No.	MAL1139295
Virtual Lens Range	

Dv (10)	1.53 µm
Dv (50)	7.70 µm
Dv (90)	24.0 µm

D [4,3]	10.6 µm
Span	2.922

Frequency (compatible) and Undersize



Measurement Details	
File Path	R:\Malvern 3000\Measurement Data\59199-91.mmes
Record Number	5
Average Result Records	1, 2, 3, 4

AR 2023-05-16
BF 2023-05-16

Malvern 3000 Liquid Analysis v.2

ptl Particle Technology Labs

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Measurement Date Time	5/16/2023 1:29:04 PM
Result Source	Averaged

Result

Size (µm)	% Volume Under	Size (µm)	% Volume Under	Size (µm)	% Volume Under	Size (µm)	% Volume Under	Size (µm)	% Volume Under
0.0100	0.00	0.166	0.00	2.75	20.89	45.6	100.00	756	100.00
0.0114	0.00	0.188	0.00	3.12	23.86	51.8	100.00	859	100.00
0.0129	0.00	0.214	0.00	3.55	27.10	58.9	100.00	976	100.00
0.0147	0.00	0.243	0.00	4.03	30.60	66.9	100.00	1110	100.00
0.0167	0.00	0.276	0.00	4.58	34.30	76.0	100.00	1260	100.00
0.0189	0.00	0.314	0.00	5.21	38.12	86.4	100.00	1430	100.00
0.0215	0.00	0.357	0.00	5.92	41.99	98.1	100.00	1630	100.00
0.0244	0.00	0.405	0.08	6.72	45.86	111	100.00	1850	100.00
0.0278	0.00	0.460	0.22	7.64	49.73	127	100.00	2100	100.00
0.0315	0.00	0.523	0.47	8.68	53.64	144	100.00	2390	100.00
0.0358	0.00	0.594	0.86	9.86	57.64	163	100.00	2710	100.00
0.0407	0.00	0.675	1.42	11.2	61.84	186	100.00	3080	100.00
0.0463	0.00	0.767	2.16	12.7	66.30	211	100.00	3500	100.00
0.0526	0.00	0.872	3.12	14.5	71.04	240	100.00		
0.0597	0.00	0.991	4.30	16.4	75.96	272	100.00		
0.0679	0.00	1.13	5.71	18.7	80.94	310	100.00		
0.0771	0.00	1.28	7.32	21.2	85.72	352	100.00		
0.0876	0.00	1.45	9.15	24.1	90.07	400	100.00		
0.0995	0.00	1.65	11.15	27.4	93.74	454	100.00		
0.113	0.00	1.88	13.32	31.1	96.58	516	100.00		
0.128	0.00	2.13	15.66	35.3	98.52	586	100.00		
0.146	0.00	2.42	18.17	40.1	99.64	666	100.00		

Measurement Details	
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