

Electronic Supplementary Information

Design and testing of an operando-Raman annular reactor for kinetic studies in heterogeneous catalysis

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1. Homogeneous reactions

The homogeneous reactions contribution both upstream (in the oven) and downstream (in the heating tape) has been evaluated by performing blank tests. Experiments was realized into the annular reactor (no Raman spectra have been recorded) on a clean alumina tube support, without catalyst. The operating conditions has been carefully selected, to reproduce experiments to assess the amount of homogeneous reactions. Temperature over the catalyst zone has been set at 700 °C and pressure has been kept atmospheric. The mixture was fed to assessed the catalytic partial oxidation (CPO) contribution of the homogeneous reaction in the experimental set-up. CPO has been chosen because conversions of CH₄ in heterogeneous CPO are resulted higher than in dry reforming (DR) in the same operating conditions. Methane was kept at CH₄ = 4.5 % (O₂ / CH₄ = 0.5, N₂ to balance) and the effect of three different flow rates was evaluated (66, 120 and 186 Ncc/min). In Fig. S1, it is possible to appreciate that the conversion of CH₄ decreases increasing the flow rate.

The experimental conditions described in the results and discussion section are related to feed flow rate around 180 Ncc/min. The homogeneous reactions could be considered negligible because the CH₄ conversion is 3.5% and no syngas (H₂ and CO) is formed.

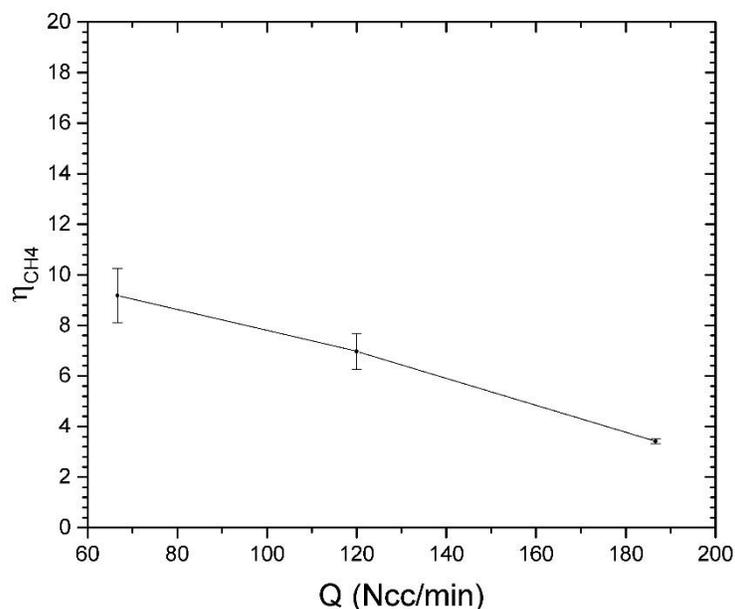


Figure S1. Conversion of CH₄ in blank test (without catalyst) in the annular reactor respect to the feed flow rate. Feeding parameters are: CH₄ = 4.6 %, CO₂ / CH₄ = 0.5, N₂ to balance. Temperature has been set at 700 °C. Standard deviation, calculated from experimental outcomes, is reported for each test.

2. Black body radiation

As shown in Fig. S2, 488 nm laser wavelength allows to measure Raman spectra at high temperature (800°C), for which the very strong black-body radiation background in the NIR/red range hinders the detection of the Raman signal.

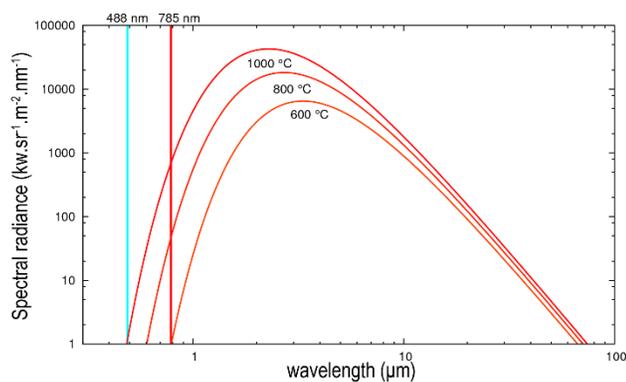


Figure S2. Black body radiation for different temperatures. The spectral radiance of a body (B) for wavelength λ at absolute temperature T is plotted based on Planck's law: $B(\lambda, T) = (2hc^2/\lambda^5)(1/(e^{\frac{hc}{\lambda kT}} - 1))$ where h the Planck constant, k the Boltzmann constant, c the speed of light. The positions of the 488nm (blue) and 785nm (red) laser wavelengths are also reported.

3. Focusing proof

The focusing proof is provided by comparing the spectrum of the blank alumina tube recorded outside the reactor, *i.e.* ex-situ, with the one recorded inside the reactor, *i.e.* in-situ (Fig. S3a). If a complete match of both Raman peak wavenumbers and relative intensities is achieved, the focusing is complete. The image of the laser spot on the alumina tube in the in-situ condition can also be taken as an additional indication (Fig. S3b): the correct alignment results in a well-defined, neat circular spot, with no blurry contours.

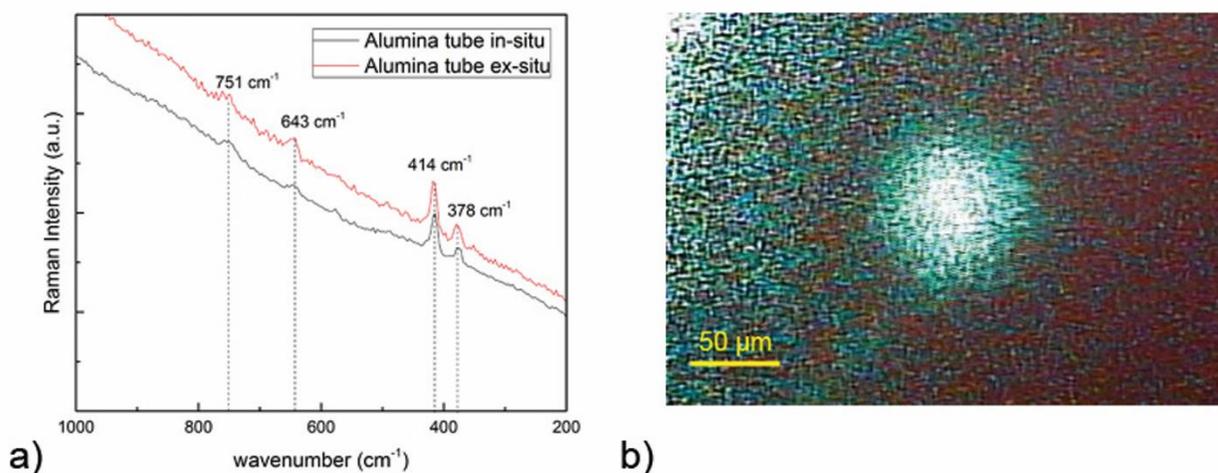


Figure S3. a) Representative Raman spectra of the alumina tube: outside the reactor (red) and inside the reactor (black), b) Image of the laser spot on the alumina tube in the in-situ condition.

4. CH₄ CPO experiments

In order to check whether the system worked properly also as annular reactor, standard CH₄ CPO tests were carried out without recording Raman spectra. The standard CH₄ CPO test were performed with diluted feed streams (CH₄ = 4.53%, O₂/CH₄ = 0.52, N₂ to balance) at increasing the temperature 50°C stepwise from 300 to 800°C. A GHSV of 4×10⁵ NI/Kgcat/h was adopted. The molar fractions of reactants (CH₄ and O₂) and products (CO, CO₂, H₂ and H₂O) at the exit of the reactor are reported in Fig. S4 as a function of the catalyst temperature. The reaction started at 300°C and by increasing the temperature, the conversion of CH₄ and O₂ increased. Below 400°C, CO₂ and H₂O were the only detectable products. At temperature higher than 400°C, the conversion of O₂ was complete, a decrease of the CO₂ and H₂O molar fractions was observed and syngas production began. The production of syngas increased steadily with temperature until reaching a stable composition, in line with thermodynamic equilibrium predictions. At 800°C, CH₄ conversion reached 92%.

Panels c and d of Fig. S4 show the axial temperature profiles measured between 300 and 400°C (*i.e.* up to complete O₂ conversion) and between 450°C and 800°C, respectively. In the first situation ($T \leq 400^\circ\text{C}$), the total oxidation of CH₄ was active along the whole catalyst layer, with no simultaneous reforming: the temperature gradients tended to arise as O₂ was progressively consumed. The most demanding condition was met at complete O₂ conversion and no syngas production, that is, at 400°C: in this case, the results show that the 10°C/cm constraint was respected. Upon further increasing the temperature ($T \geq 450^\circ\text{C}$), once O₂ was fully converted, the endothermic reforming reactions progressively activated along the catalyst layer, with consumption of H₂O and CO₂ and syngas formation. The endothermic heat input required by reforming grew with CH₄ conversion, moderating the gradients due to the exothermic CH₄ oxidation, which were never larger than 8°C/cm. Fig. S4d clearly shows that the temperature peak decreased up to an almost flat profile ($\pm 2^\circ\text{C}$) at 700°C.

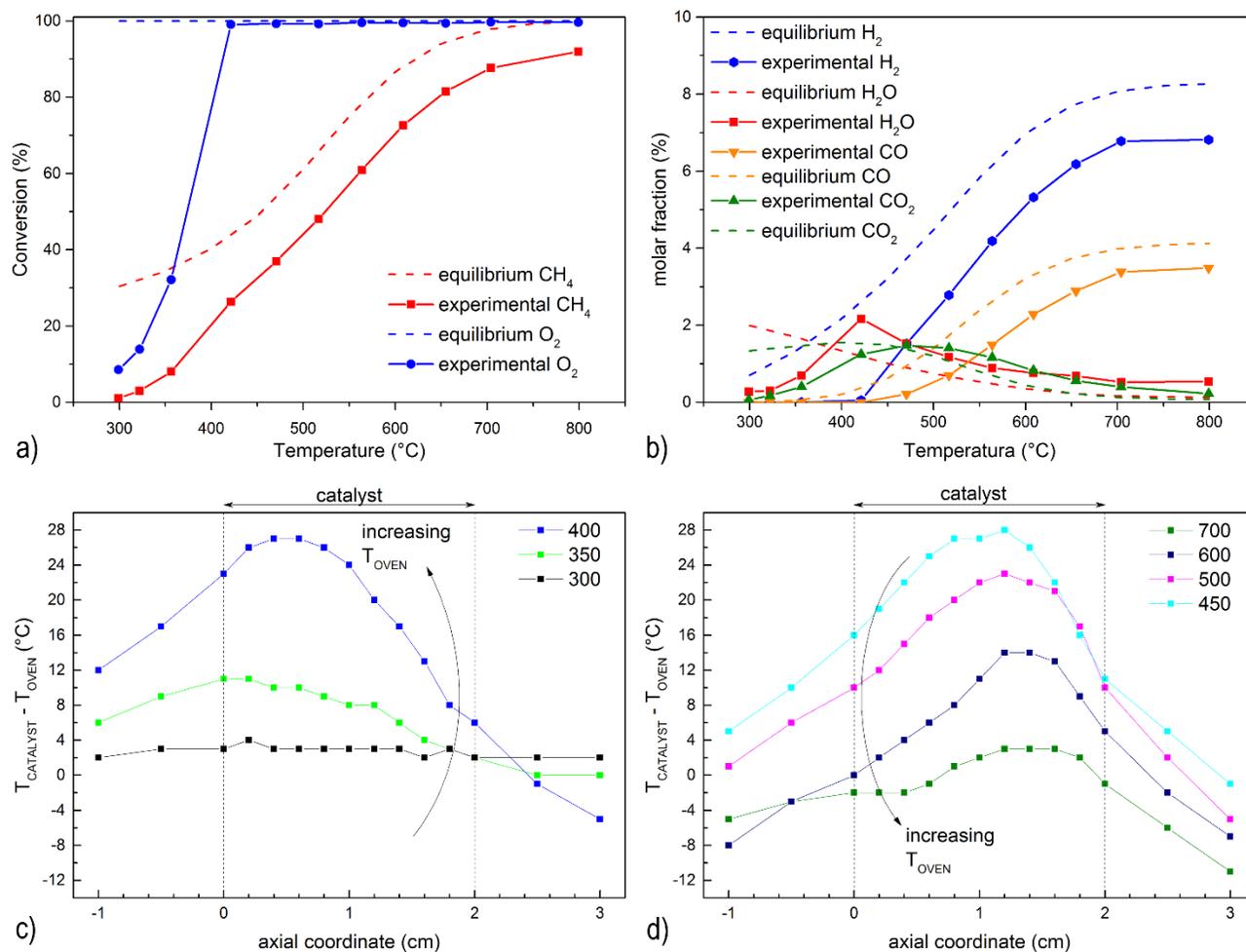


Figure S4. Results of the CH₄ CPO test at different temperature. GHSV = 4×10^5 Ni/Kgcat/h; Experimental results (symbols), equilibrium (dashed line). Feed composition: CH₄ = 4.53%, O₂/CH₄ = 0.52, N₂ to balance; atmospheric pressure. Panels: a) CH₄ and O₂ conversion; b) H₂, H₂O, CO and CO₂ molar fraction. Axial temperature profiles along the catalyst layer: c) $T \leq 400^\circ\text{C}$; d) $T \geq 450^\circ\text{C}$.

5. XRD analysis of the α -Al₂O₃

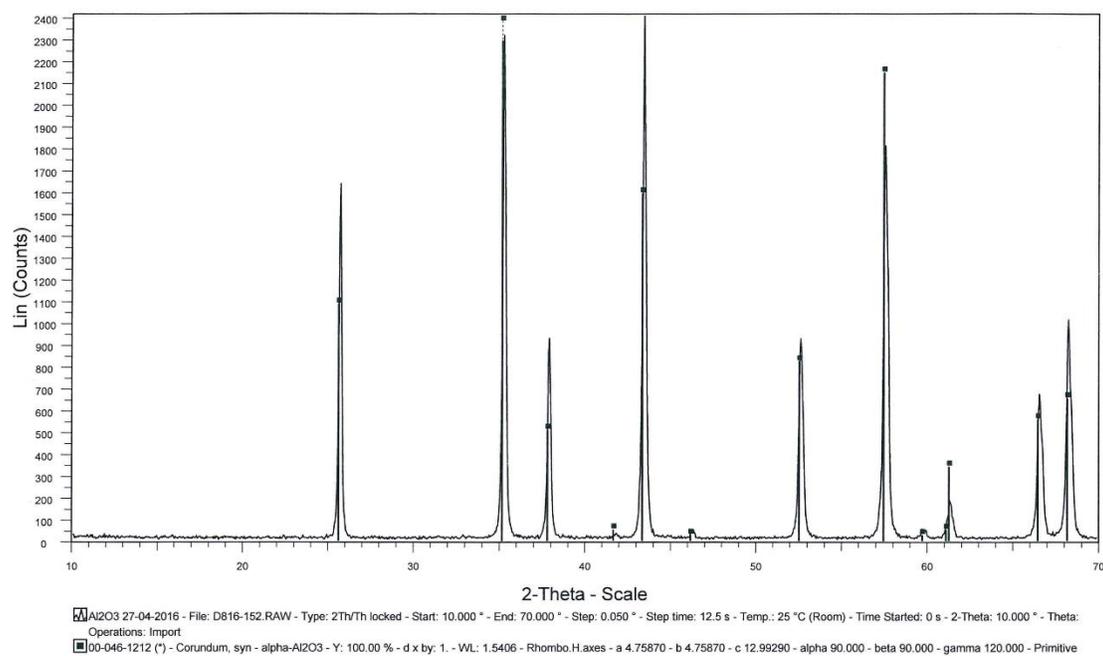


Figure S5. XRD analysis of the α -Alumina which is used as support material.

6. BET analysis of the α -Al₂O₃

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TriStar 3000 V3.01 TriStar 3000 V3.01 Unit 1 Serial # 399 Anl Port #2 Page 1

Sample: CM02216 Allumina
 Operator:
 Submitter: Maestri-Donazzi
 File Name: E:\3000_D~1\AS2016\CM02216.SMP

Started: 21/04/16 10.15.30 Analysis Adsorptive: N2
 Completed: 21/04/16 14.20.41 Analysis Bath: 77.35 K
 Report Time: 29/04/16 14.17.00 Sample Weight: 0.2789 g
 Warm Freespace: 7.0177 cm³ MEASURED Cold Freespace: 19.8294 cm³
 Equil. Interval: 10 secs Low Pressure Dose: None
 Sample Density: 1.000 cm³/g Automatic Degas: No

Analysis Log

Relative Pressure	Pressure (mmHg)	Vol Adsorbed (cm ³ /g STP)	Elapsed Time (HR:MN)	Saturation Press. (mmHg)
			01:34	787.66205
0.014121641	11.12629	1.5519	01:45	
0.048637442	38.32294	1.8387	01:47	
0.082533094	65.03378	1.9783	01:49	
0.112262471	88.46435	2.0729	01:51	
0.129773989	102.26903	2.1241	01:53	
0.151511425	119.40559	2.1852	01:55	
0.166815291	131.47340	2.2270	01:57	
0.187964842	148.14993	2.2851	01:59	
0.206963202	163.13260	2.3363	02:01	
0.252335154	198.90613	2.4621	02:03	
0.301441143	237.63319	2.6083	02:06	
0.350799438	276.55801	2.7612	02:08	
0.399879916	315.26788	2.9182	02:10	
0.449522327	354.42487	3.0768	02:12	
0.499284930	393.68073	3.2373	02:14	
0.549096845	432.97958	3.3999	02:16	
0.598821763	472.21396	3.5794	02:18	
0.648710460	511.58160	3.7803	02:20	
0.698403116	550.79877	4.0239	02:22	
0.748150536	590.06323	4.3336	02:24	
0.797860007	629.30188	4.7336	02:26	
0.818497185	645.61304	4.9492	02:28	
0.847999075	668.93610	5.3113	02:31	
0.872265802	688.11475	5.7170	02:33	
0.896857007	707.55139	6.2865	02:35	
0.920206331	726.02930	7.1706	02:38	
0.944129502	744.96289	8.5278	02:41	
0.972409059	767.41754	11.6731	02:48	
0.977247589	771.31689	13.3520	02:52	
0.982120797	775.28503	17.4773	02:58	
0.988522675	780.46130	24.8447	03:04	
0.983287240	776.36847	22.9216	03:06	
0.980280780	774.05548	20.2834	03:09	
0.976911687	771.47595	17.6587	03:13	
0.945470530	746.80298	9.2977	03:21	
0.918548703	725.59509	7.7710	03:24	
0.889454231	702.66748	6.6368	03:27	
0.875279867	691.52405	6.1778	03:30	
0.848952539	670.75897	5.4149	03:32	
0.822633374	649.99817	4.9750	03:34	

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TriStar 3000 V3.01 TriStar 3000 V3.01 Unit 1 Serial # 399 Anl Port #2 Page 2

Sample: CM02216 Allumina
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 Submitter: Maestri-Donazzi
 File Name: E:\3000_D~1\AS2016\CM02216.SMP

Started: 21/04/16 10.15.30 Analysis Adsorptive: N2
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 Equil. Interval: 10 secs Low Pressure Dose: None
 Sample Density: 1.000 cm³/g Automatic Degas: No

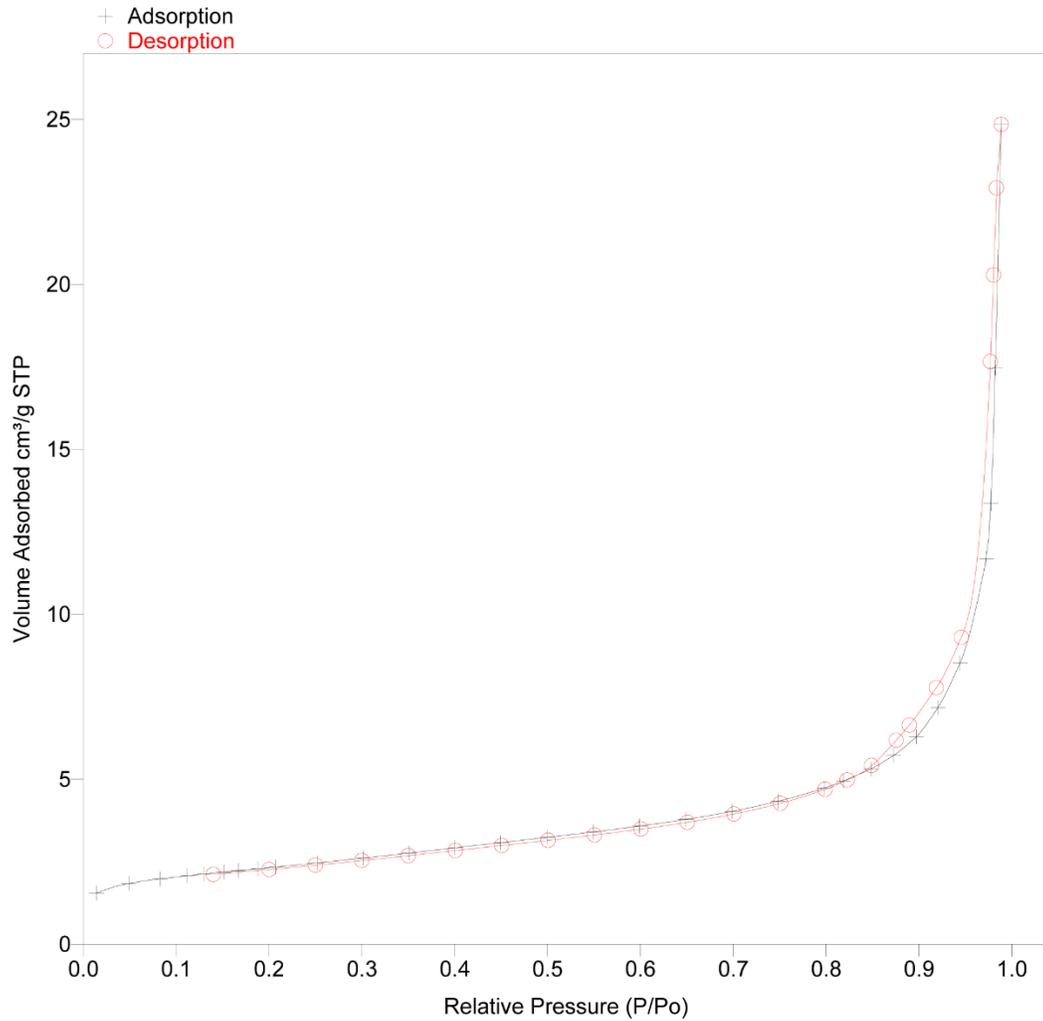
Analysis Log

Relative Pressure	Pressure (mmHg)	Vol Adsorbed (cm ³ /g STP)	Elapsed Time (HR:MN)	Saturation Press. (mmHg)
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0.750737851	593.20593	4.2707	03:39	
0.700605629	553.59326	3.9452	03:41	
0.650565868	514.05365	3.6939	03:43	
0.600575040	474.55270	3.4864	03:45	
0.550567953	435.03891	3.3083	03:47	
0.500544491	395.51218	3.1462	03:49	
0.450526512	355.98978	2.9909	03:51	
0.400503088	316.46307	2.8369	03:53	
0.349941005	276.51074	2.6811	03:56	
0.299890353	236.96252	2.5330	03:58	
0.249926754	197.48309	2.3920	04:00	
0.200115518	158.12405	2.2612	04:02	
0.140048215	110.66104	2.1098	04:04	

Sample: CM02216 Allumina
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 Submitter: Maestri-Donazzi
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Isotherm Plot



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TriStar 3000 V3.01 TriStar 3000 V3.01 Unit 1 Serial # 399 Anl Port #2 Page 4

Sample: CM02216 Allumina
 Operator:
 Submitter: Maestri-Donazzi
 File Name: E:\3000_D~1\AS2016\CM02216.SMP

Started: 21/04/16 10.15.30 Analysis Adsorptive: N2
 Completed: 21/04/16 14.20.41 Analysis Bath: 77.35 K
 Report Time: 29/04/16 14.17.00 Sample Weight: 0.2789 g
 Warm Freespace: 7.0177 cm³ MEASURED Cold Freespace: 19.8294 cm³
 Equil. Interval: 10 secs Low Pressure Dose: None
 Sample Density: 1.000 cm³/g Automatic Degas: No

BET Surface Area Report

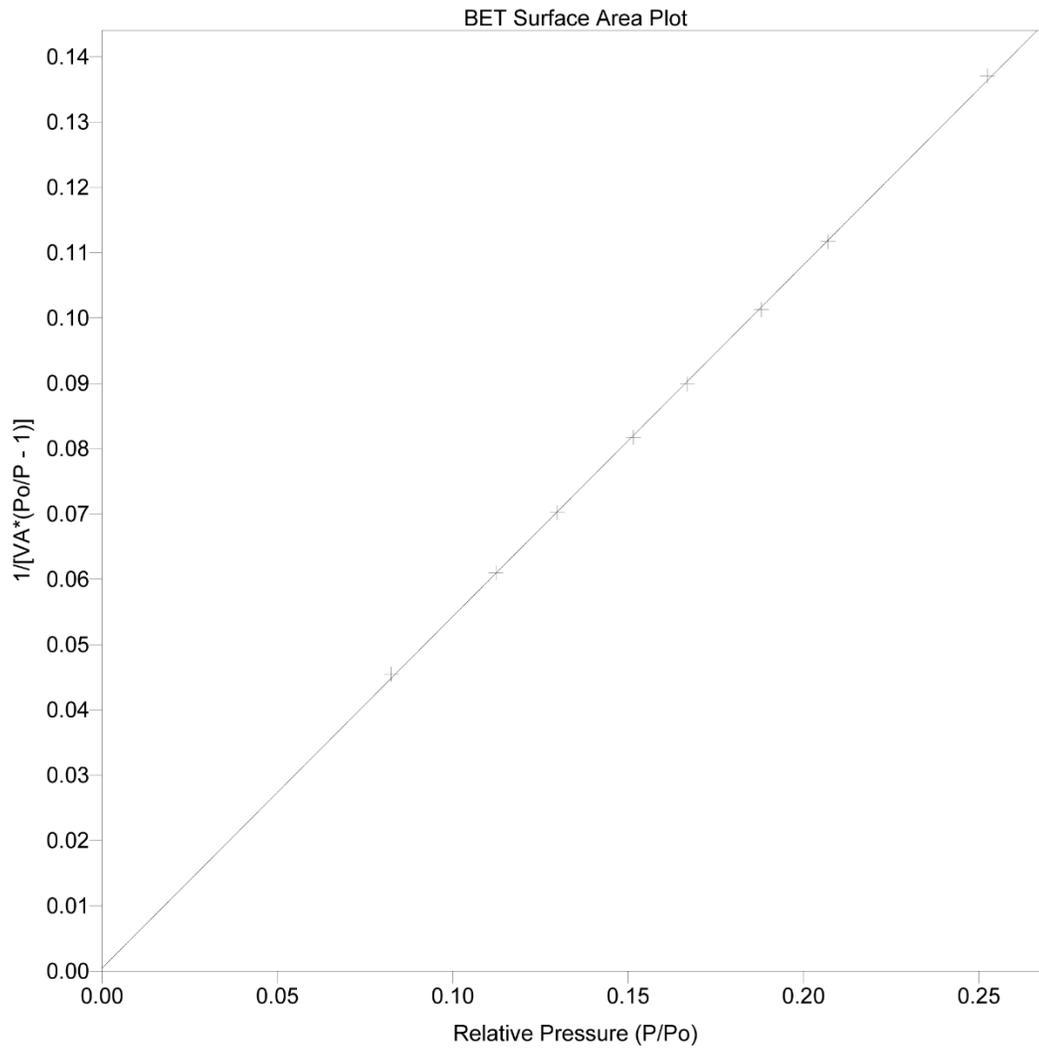
BET Surface Area: 8.0751 ± 0.0496 m²/g
 Slope: 0.538659 ± 0.003265
 Y-Intercept: 0.000429 ± 0.000552
 C: 1257.503623
 VM: 1.854984 cm³/g STP
 Correlation Coefficient: 9.998898e-01

Molecular Cross-section: 0.1620 nm²

Relative Pressure	Vol Adsorbed (cm ³ /g STP)	1/[VA*(Po/P - 1)]
0.082533094	1.9783	0.045471
0.112262471	2.0729	0.061007
0.129773989	2.1241	0.070209
0.151511425	2.1852	0.081718
0.166815291	2.2270	0.089903
0.187964842	2.2851	0.101296
0.206963202	2.3363	0.111707
0.252335154	2.4621	0.137075

Sample: CM02216 Allumina
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Equil. Interval: 10 secs Low Pressure Dose: None
Sample Density: 1.000 cm³/g Automatic Degas: No



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TriStar 3000 V3.01 TriStar 3000 V3.01 Unit 1 Serial # 399 Anl Port #2 Page 6

Sample: CM02216 Allumina
 Operator:
 Submitter: Maestri-Donazzi
 File Name: E:\3000_D~1\AS2016\CM02216.SMP

Started: 21/04/16 10.15.30 Analysis Adsorptive: N2
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 Report Time: 29/04/16 14.17.00 Sample Weight: 0.2789 g
 Warm Freespace: 7.0177 cm³ MEASURED Cold Freespace: 19.8294 cm³
 Equil. Interval: 10 secs Low Pressure Dose: None
 Sample Density: 1.000 cm³/g Automatic Degas: No

BJH Adsorption Pore Distribution Report

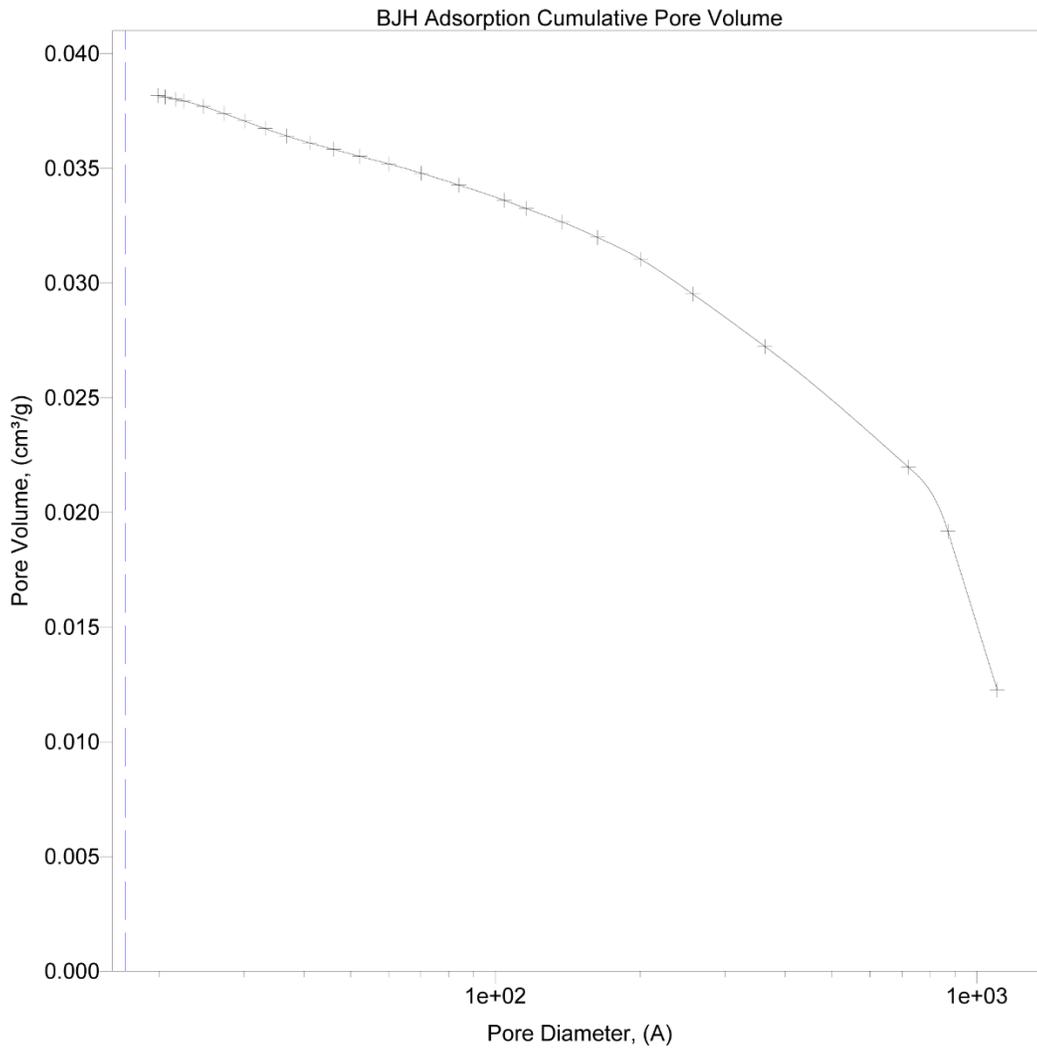
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 Adsorbate Property Factor: 9.530000 A
 Density Conversion Factor: 0.001547
 Fraction of Pores Open at Both Ends: 0.000

Pore Diameter Range (A)	Average Diameter (A)	Incremental Pore Volume (cm ³ /g)	Cumulative Pore Volume (cm ³ /g)	dV/dD Pore Volume (cm ³ /g-A)	dV/dlog(D) Pore Volume (cm ³ /g-A)
1704.6-1102.6	1277.7	0.012265	0.012265	2.0372e-05	6.4818e-02
1102.6- 870.6	959.0	0.006913	0.019178	2.9809e-05	6.7405e-02
870.6- 721.0	781.5	0.002787	0.021965	1.8632e-05	3.4042e-02
721.0- 362.8	431.8	0.005267	0.027233	1.4705e-05	1.7662e-02
362.8- 256.9	291.3	0.002288	0.029521	2.1598e-05	1.5259e-02
256.9- 200.4	221.3	0.001511	0.031032	2.6744e-05	1.4009e-02
200.4- 162.9	177.5	0.000951	0.031982	2.5371e-05	1.0576e-02
162.9- 137.7	148.0	0.000672	0.032654	2.6578e-05	9.1763e-03
137.7- 115.8	124.7	0.000592	0.033246	2.7119e-05	7.8947e-03
115.8- 104.3	109.4	0.000358	0.033604	3.0949e-05	7.8350e-03
104.3- 84.0	91.7	0.000664	0.034268	3.2701e-05	7.0588e-03
84.0- 70.1	75.6	0.000519	0.034788	3.7524e-05	6.6384e-03
70.1- 60.0	64.2	0.000404	0.035191	4.0056e-05	5.9901e-03
60.0- 52.3	55.5	0.000329	0.035520	4.2389e-05	5.4726e-03
52.3- 46.1	48.7	0.000304	0.035824	4.9467e-05	5.5981e-03
46.1- 41.1	43.3	0.000281	0.036105	5.5704e-05	5.5889e-03
41.1- 36.9	38.7	0.000302	0.036406	7.1324e-05	6.3967e-03
36.9- 33.3	34.8	0.000319	0.036725	8.8055e-05	7.1026e-03
33.3- 30.1	31.5	0.000335	0.037060	1.0659e-04	7.7689e-03
30.1- 27.3	28.5	0.000330	0.037390	1.1671e-04	7.7057e-03
27.3- 24.7	25.8	0.000307	0.037697	1.1988e-04	7.1712e-03
24.7- 22.5	23.5	0.000234	0.037932	1.0568e-04	5.7412e-03
22.5- 21.6	22.0	0.000084	0.038016	9.3754e-05	4.7596e-03
21.6- 20.6	21.1	0.000095	0.038111	9.5461e-05	4.6381e-03
20.6- 19.9	20.2	0.000062	0.038172	8.6257e-05	4.0218e-03

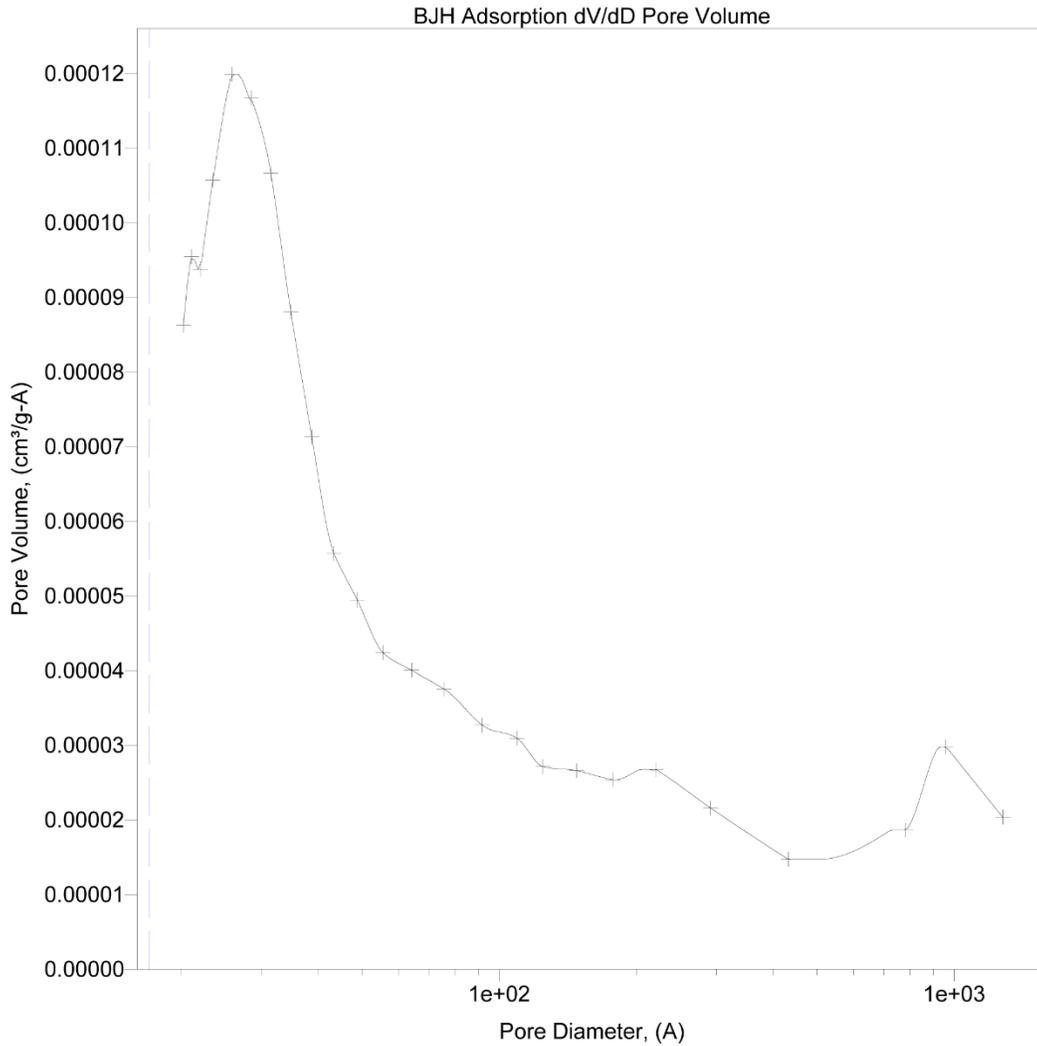
Sample: CM02216 Allumina
Operator:
Submitter: Maestri-Donazzi
File Name: E:\3000_D~1\AS2016\CM02216.SMP

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Equil. Interval: 10 secs Low Pressure Dose: None
Sample Density: 1.000 cm³/g Automatic Degas: No



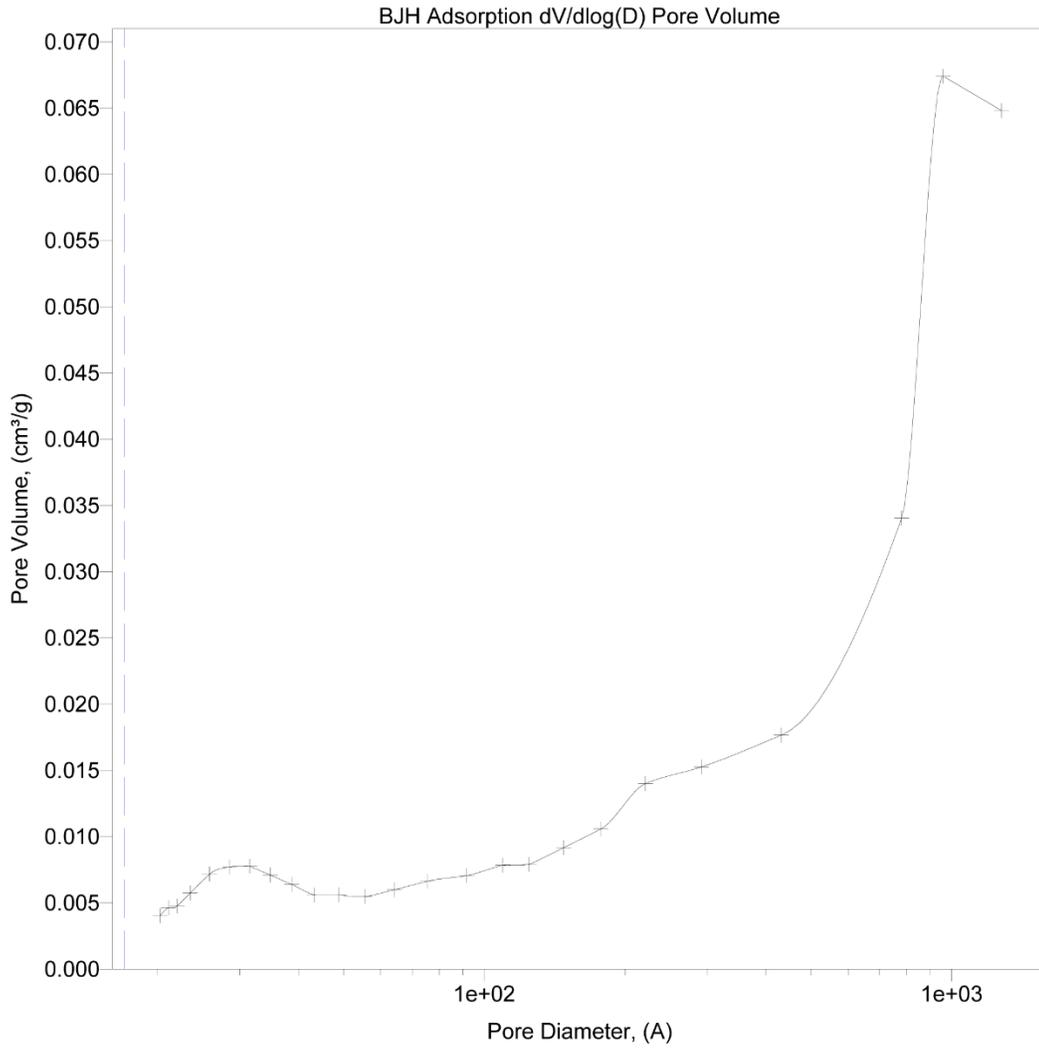
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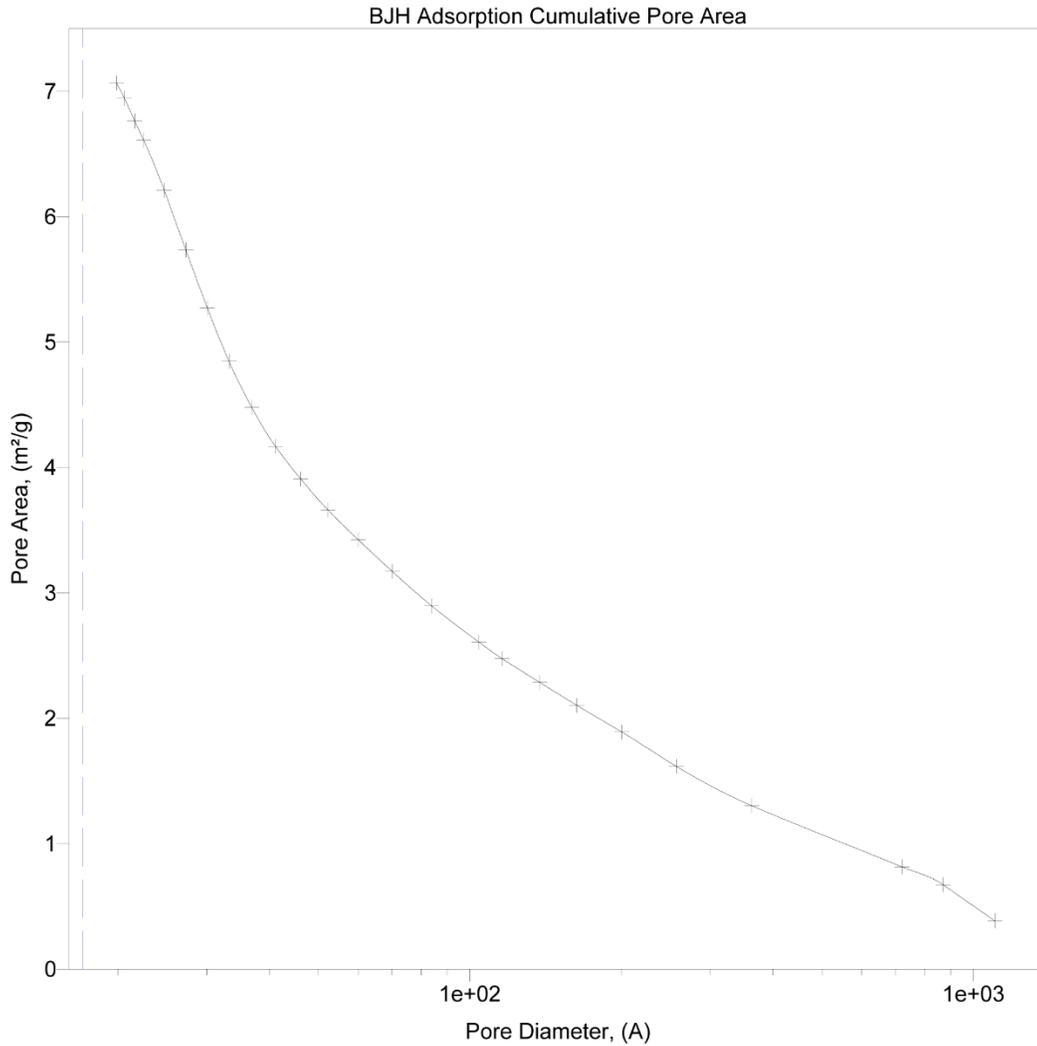
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Sample: CM02216 Allumina
Operator:
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TriStar 3000 V3.01 TriStar 3000 V3.01 Unit 1 Serial # 399 Anl Port #2 Page 11

Sample: CM02216 Allumina
 Operator:
 Submitter: Maestri-Donazzi
 File Name: E:\3000_D~1\AS2016\CM02216.SMP

Started: 21/04/16 10.15.30 Analysis Adsorptive: N2
 Completed: 21/04/16 14.20.41 Analysis Bath: 77.35 K
 Report Time: 29/04/16 14.17.00 Sample Weight: 0.2789 g
 Warm Freespace: 7.0177 cm³ MEASURED Cold Freespace: 19.8294 cm³
 Equil. Interval: 10 secs Low Pressure Dose: None
 Sample Density: 1.000 cm³/g Automatic Degas: No

BJH Desorption Pore Distribution Report

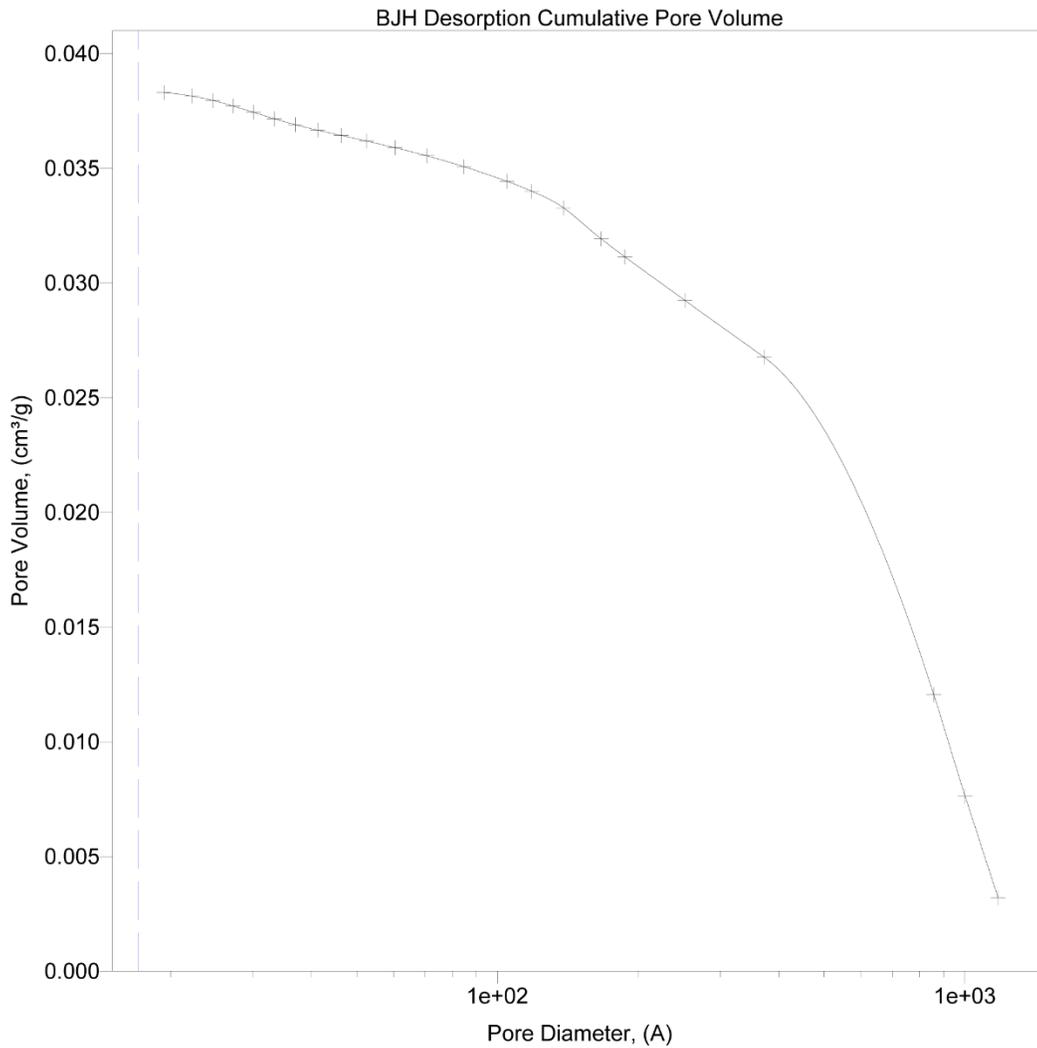
$$t = 3.5400 \times [-5.0000 / \ln(P/P_0)]^{0.3330}$$

Diameter Range: 17.0000 to 3000.0000 A
 Adsorbate Property Factor: 9.530000 A
 Density Conversion Factor: 0.001547
 Fraction of Pores Open at Both Ends: 0.000

Pore Diameter Range (A)	Average Diameter (A)	Incremental Pore Volume (cm ³ /g)	Cumulative Pore Volume (cm ³ /g)	dV/dD Pore Volume (cm ³ /g-A)	dV/dlog(D) Pore Volume (cm ³ /g-A)
1704.6-1178.0	1346.3	0.003194	0.003194	6.0666e-06	1.9907e-02
1178.0-1001.6	1075.2	0.004429	0.007623	2.5102e-05	6.2852e-02
1001.6- 858.2	918.6	0.004429	0.012053	3.0898e-05	6.6027e-02
858.2- 371.5	443.9	0.014719	0.026772	3.0241e-05	4.0477e-02
371.5- 251.8	288.3	0.002467	0.029239	2.0621e-05	1.4615e-02
251.8- 187.4	209.7	0.001896	0.031135	2.9423e-05	1.4772e-02
187.4- 166.8	175.8	0.000787	0.031922	3.8114e-05	1.5523e-02
166.8- 138.5	149.8	0.001354	0.033276	4.7917e-05	1.6792e-02
138.5- 118.5	126.8	0.000721	0.033998	3.6002e-05	1.0629e-02
118.5- 104.8	110.7	0.000437	0.034435	3.1963e-05	8.2051e-03
104.8- 84.8	92.4	0.000633	0.035068	3.1694e-05	6.8929e-03
84.8- 70.6	76.2	0.000477	0.035544	3.3590e-05	5.9953e-03
70.6- 60.4	64.6	0.000355	0.035899	3.4516e-05	5.1950e-03
60.4- 52.5	55.8	0.000288	0.036186	3.6713e-05	4.7637e-03
52.5- 46.3	48.9	0.000245	0.036431	3.9316e-05	4.4676e-03
46.3- 41.2	43.4	0.000230	0.036661	4.5237e-05	4.5533e-03
41.2- 37.0	38.8	0.000238	0.036899	5.5744e-05	5.0119e-03
37.0- 33.3	34.9	0.000259	0.037158	7.0890e-05	5.7279e-03
33.3- 30.1	31.5	0.000284	0.037442	8.7718e-05	6.3922e-03
30.1- 27.2	28.4	0.000270	0.037713	9.4646e-05	6.2339e-03
27.2- 24.6	25.7	0.000245	0.037958	9.4240e-05	5.6152e-03
24.6- 22.2	23.2	0.000190	0.038148	7.8466e-05	4.2216e-03
22.2- 19.4	20.5	0.000155	0.038303	5.5085e-05	2.6299e-03

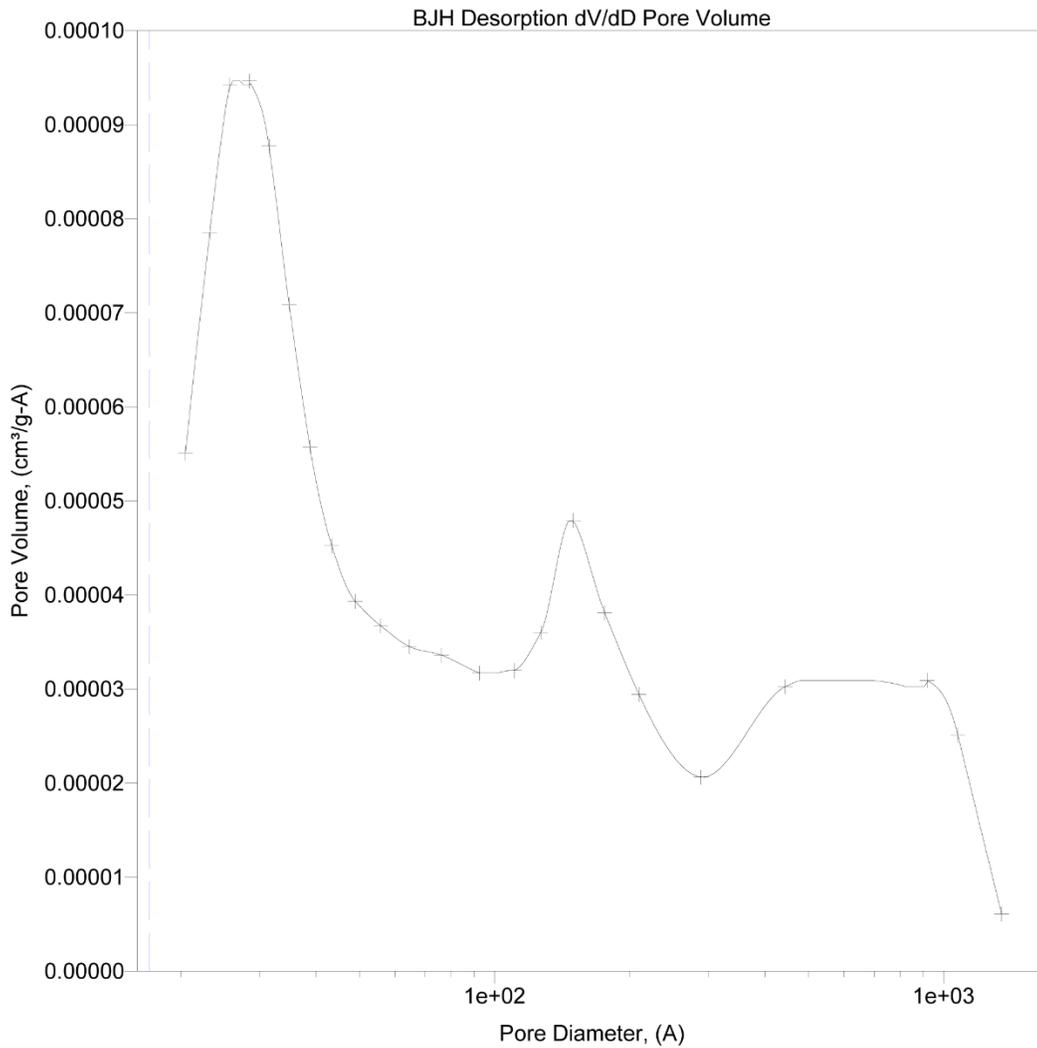
Sample: CM02216 Allumina
Operator:
Submitter: Maestri-Donazzi
File Name: E:\3000_D~1\AS2016\CM02216.SMP

Started: 21/04/16 10.15.30 Analysis Adsorptive: N2
Completed: 21/04/16 14.20.41 Analysis Bath: 77.35 K
Report Time: 29/04/16 14.17.00 Sample Weight: 0.2789 g
Warm Freespace: 7.0177 cm³ MEASURED Cold Freespace: 19.8294 cm³
Equil. Interval: 10 secs Low Pressure Dose: None
Sample Density: 1.000 cm³/g Automatic Degas: No



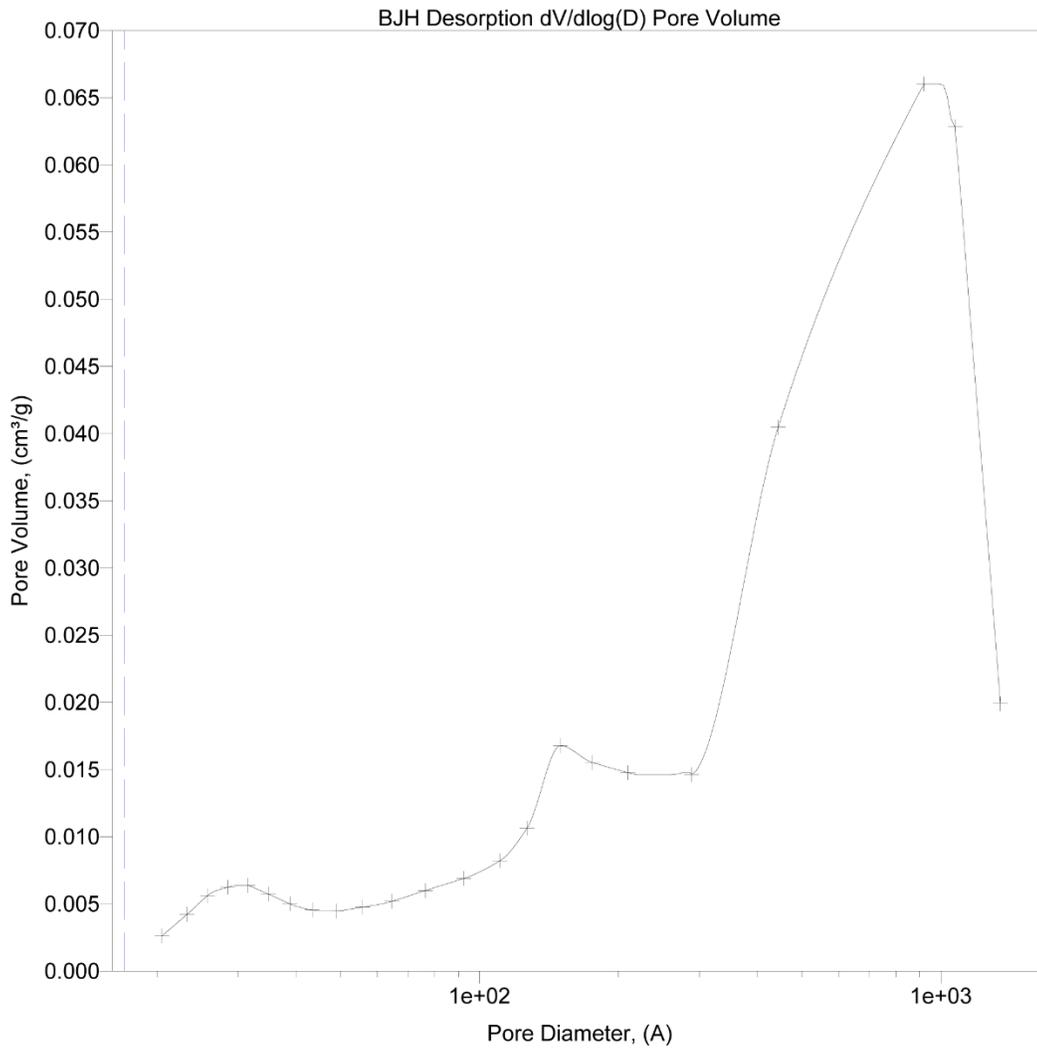
Sample: CM02216 Allumina
Operator:
Submitter: Maestri-Donazzi
File Name: E:\3000_D~1\AS2016\CM02216.SMP

Started: 21/04/16 10.15.30 Analysis Adsorptive: N2
Completed: 21/04/16 14.20.41 Analysis Bath: 77.35 K
Report Time: 29/04/16 14.17.00 Sample Weight: 0.2789 g
Warm Freespace: 7.0177 cm³ MEASURED Cold Freespace: 19.8294 cm³
Equil. Interval: 10 secs Low Pressure Dose: None
Sample Density: 1.000 cm³/g Automatic Degas: No



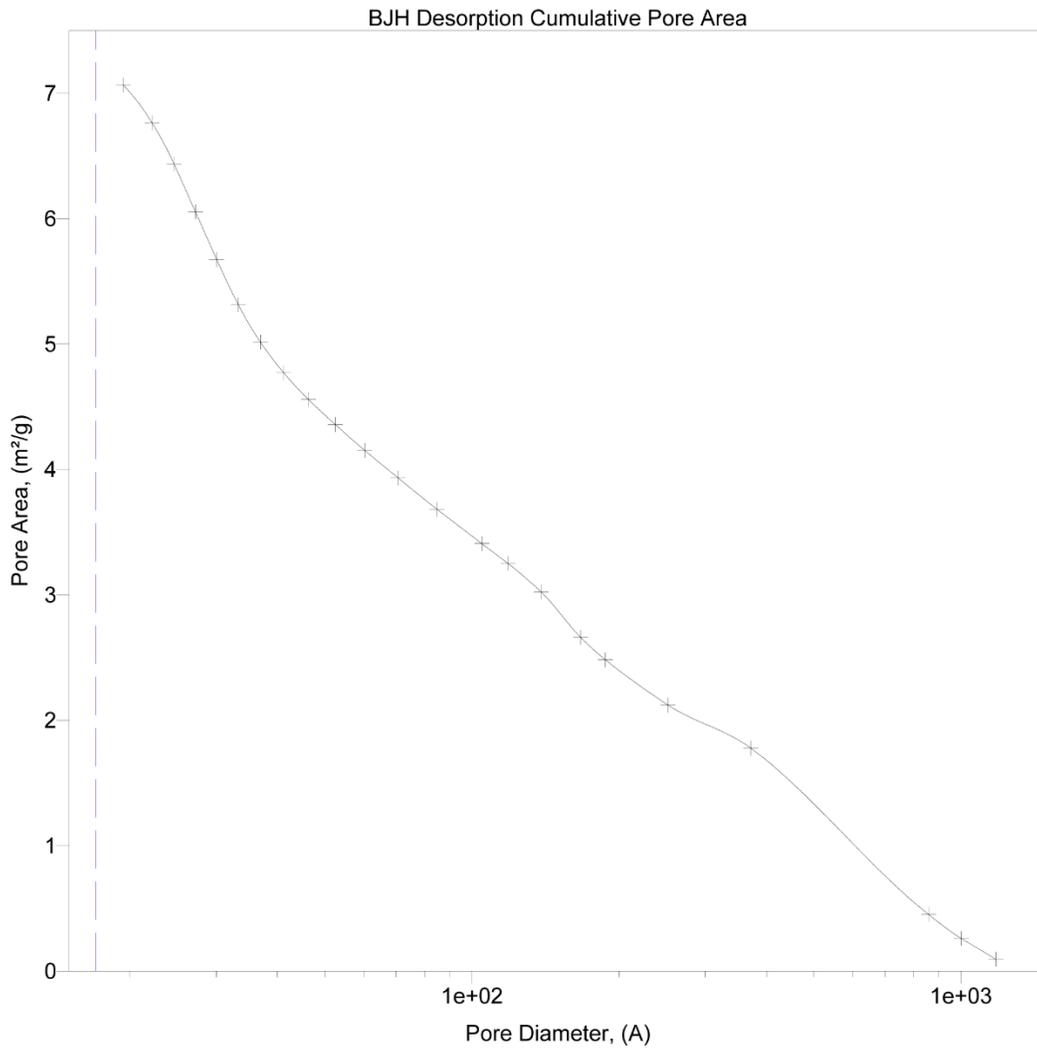
Sample: CM02216 Allumina
Operator:
Submitter: Maestri-Donazzi
File Name: E:\3000_D~1\AS2016\CM02216.SMP

Started: 21/04/16 10.15.30 Analysis Adsorptive: N2
Completed: 21/04/16 14.20.41 Analysis Bath: 77.35 K
Report Time: 29/04/16 14.17.00 Sample Weight: 0.2789 g
Warm Freespace: 7.0177 cm³ MEASURED Cold Freespace: 19.8294 cm³
Equil. Interval: 10 secs Low Pressure Dose: None
Sample Density: 1.000 cm³/g Automatic Degas: No



Sample: CM02216 Allumina
Operator:
Submitter: Maestri-Donazzi
File Name: E:\3000_D~1\AS2016\CM02216.SMP

Started: 21/04/16 10.15.30 Analysis Adsorptive: N2
Completed: 21/04/16 14.20.41 Analysis Bath: 77.35 K
Report Time: 29/04/16 14.17.00 Sample Weight: 0.2789 g
Warm Freespace: 7.0177 cm³ MEASURED Cold Freespace: 19.8294 cm³
Equil. Interval: 10 secs Low Pressure Dose: None
Sample Density: 1.000 cm³/g Automatic Degas: No



TriStar 3000 V3.01 TriStar 3000 V3.01 Unit 1 Serial # 399 Anl Port #2 Page 16

Sample: CM02216 Allumina
 Operator:
 Submitter: Maestri-Donazzi
 File Name: E:\3000_D~1\AS2016\CM02216.SMP

Started: 21/04/16 10.15.30 Analysis Adsorptive: N2
 Completed: 21/04/16 14.20.41 Analysis Bath: 77.35 K
 Report Time: 29/04/16 14.17.00 Sample Weight: 0.2789 g
 Warm Freespace: 7.0177 cm³ MEASURED Cold Freespace: 19.8294 cm³
 Equil. Interval: 10 secs Low Pressure Dose: None
 Sample Density: 1.000 cm³/g Automatic Degas: No

Summary Report

Area

Single Point Surface Area at P/Po 0.25233515 :	8.0136	m ² /g
BET Surface Area:	8.0751	m ² /g
Langmuir Surface Area:	12.9136	m ² /g
Micropore Area:	1.6798	m ² /g
BJH Adsorption Cumulative Surface Area of pores between 17.000000 and 3000.000000 A Diameter:	7.0658	m ² /g
BJH Desorption Cumulative Surface Area of pores between 17.000000 and 3000.000000 A Diameter:	7.0666	m ² /g

Volume

Single Point Adsorption Total Pore Volume of pores less than 721.0424 A Diameter at P/Po 0.97240906:	0.018056	cm ³ /g
BJH Adsorption Cumulative Pore Volume of pores between 17.000000 and 3000.000000 A Diameter:	0.038172	cm ³ /g
BJH Desorption Cumulative Pore Volume of pores between 17.000000 and 3000.000000 A Diameter:	0.038303	cm ³ /g

Pore Size

Adsorption Average Pore Diameter (4V/A by BET):	89.4397	A
BJH Adsorption Average Pore Diameter (4V/A):	216.0950	A
BJH Desorption Average Pore Diameter (4V/A):	216.8110	A

7. Hg Porosimetry results of α -Al₂O₃

micromeritics

MICROMERITICS INSTRUMENT CORPORATION

AutoPore IV 9500 V1.09

Serial: 335

Port: 2/1

Page 1

Sample ID: Alumina
Operator:
Submitter: Donazzi-Maestri
File: E:\9500_D~1\2016\PO2316.SMP

LP Analysis Time: 27/04/2016 14.59.45
HP Analysis Time: 28/04/2016 10.21.51
Report Time: 29/04/2016 14.14.24

Sample Weight: 0.3400 g
Correction Type: Formula
Show Neg. Int: No

Summary Report

Penetrometer parameters

Penetrometer:	polveri 0.366 2016		
Pen. Constant:	11.117 $\mu\text{L/pF}$	Pen. Weight:	58.3147 g
Stem Volume:	0.3660 mL	Max. Head Pressure:	4.4500 psia
Pen. Volume:	5.3111 mL	Assembly Weight:	126.5512 g

Hg Parameters

Adv. Contact Angle:	130.000 degrees	Rec. Contact Angle:	130.000 degrees
Hg Surface Tension:	485.000 dynes/cm	Hg Density:	13.5335 g/mL

User Parameters

Param 1:	0.000	Param 2:	0.000	Param 3:	0.000
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Low Pressure:

Evacuation Pressure:	50 μmHg
Evacuation Time:	5 mins
Mercury Filling Pressure:	0.51 psia
Equilibration Time:	10 secs

High Pressure:

Equilibration Time:	10 secs
---------------------	---------

Blank Correction by Formula

(From Pressure 0.10 to 60000.00 psia)

Intrusion Data Summary

Total Intrusion Volume =	0.6270 mL/g
Total Pore Area =	10.326 m^2/g
Median Pore Diameter (Volume) =	46237 A
Median Pore Diameter (Area) =	794 A
Average Pore Diameter (4V/A) =	2429 A
Bulk Density at 0.51 psia =	1.1558 g/mL
Apparent (skeletal) Density =	4.1973 g/mL
Porosity =	72.4640 %
Stem Volume Used =	59 %



MICROMERITICS INSTRUMENT CORPORATION

AutoPore IV 9500 V1.09

Serial: 335

Port: 2/1

Page 2

Sample ID: Allumina
Operator:
Submitter: Donazzi-Maestri
File: E:\9500_D~1\2016\PO2316.SMP

LP Analysis Time: 27/04/2016 14.59.45
HP Analysis Time: 28/04/2016 10.21.51
Report Time: 29/04/2016 14.14.24

Sample Weight: 0.3400 g
Correction Type: Formula
Show Neg. Int: No

Tabular Report

Pressure (psia)	Pore Diameter (A)	Cumulative Pore Volume (mL/g)	Incremental Pore Volume (mL/g)	dV/dD Pore Volume (mL/g/A)	dV/dlogD Pore Volume (mL/g)
0.51	3522693	0.0000	0.0000	0.000×10^0	0.000×10^0
1.99	909888	0.0389	0.0389	1.487×10^{-8}	6.608×10^{-2}
2.99	605488	0.0542	0.0153	5.041×10^{-8}	8.675×10^{-2}
3.99	453474	0.0676	0.0134	8.809×10^{-8}	1.067×10^{-1}
5.49	329639	0.0818	0.0142	1.146×10^{-7}	1.025×10^{-1}
5.98	302235	0.0844	0.0026	9.647×10^{-8}	7.014×10^{-2}
7.48	241852	0.0940	0.0095	1.580×10^{-7}	9.856×10^{-2}
8.48	213254	0.0983	0.0044	1.527×10^{-7}	7.992×10^{-2}
10.48	172658	0.1063	0.0079	1.954×10^{-7}	8.648×10^{-2}
12.96	139504	0.1145	0.0082	2.479×10^{-7}	8.875×10^{-2}
15.96	113326	0.1241	0.0096	3.666×10^{-7}	1.063×10^{-1}
19.98	90522	0.1478	0.0237	1.038×10^{-6}	2.427×10^{-1}
24.98	72415	0.2186	0.0709	3.913×10^{-6}	7.311×10^{-1}
29.97	60354	0.2635	0.0449	3.722×10^{-6}	5.673×10^{-1}
36.16	50019	0.2987	0.0352	3.402×10^{-6}	4.311×10^{-1}
46.77	38668	0.3469	0.0482	4.248×10^{-6}	4.314×10^{-1}
58.03	31165	0.3723	0.0254	3.387×10^{-6}	2.712×10^{-1}
75.48	23961	0.3915	0.0192	2.672×10^{-6}	1.686×10^{-1}
89.76	20149	0.4006	0.0091	2.384×10^{-6}	1.208×10^{-1}
116.30	15552	0.4089	0.0083	1.808×10^{-6}	7.390×10^{-2}
140.53	12870	0.4130	0.0041	1.512×10^{-6}	4.934×10^{-2}
175.16	10325	0.4162	0.0032	1.242×10^{-6}	3.303×10^{-2}
219.78	8229	0.4186	0.0025	1.169×10^{-6}	2.487×10^{-2}
269.90	6701	0.4204	0.0018	1.182×10^{-6}	2.025×10^{-2}
329.97	5481	0.4219	0.0015	1.254×10^{-6}	1.753×10^{-2}
419.92	4307	0.4233	0.0013	1.121×10^{-6}	1.257×10^{-2}
519.91	3479	0.4245	0.0013	1.535×10^{-6}	1.371×10^{-2}
639.45	2828	0.4254	0.0008	1.307×10^{-6}	9.455×10^{-3}
799.88	2261	0.4264	0.0010	1.738×10^{-6}	1.014×10^{-2}
989.49	1828	0.4271	0.0007	1.723×10^{-6}	8.080×10^{-3}
1199.97	1507	0.4282	0.0011	3.359×10^{-6}	1.286×10^{-2}
1498.73	1207	0.4319	0.0037	1.220×10^{-5}	3.796×10^{-2}
1899.87	952	0.4542	0.0223	8.756×10^{-5}	2.166×10^{-1}
2350.10	770	0.5512	0.0970	5.320×10^{-4}	1.051×10^0



MICROMERITICS INSTRUMENT CORPORATION

AutoPore IV 9500 V1.09

Serial: 335

Port: 2/1

Page 3

Sample ID: Allumina
Operator:
Submitter: Donazzi-Maestri
File: E:\9500_D~1\2016\PO2316.SMP

LP Analysis Time: 27/04/2016 14.59.45
HP Analysis Time: 28/04/2016 10.21.51
Report Time: 29/04/2016 14.14.24

Sample Weight: 0.3400 g
Correction Type: Formula
Show Neg. Int: No

Tabular Report

Pressure (psia)	Pore Diameter (A)	Cumulative Pore Volume (mL/g)	Incremental Pore Volume (mL/g)	dV/dD Pore Volume (mL/g/A)	dV/dlogD Pore Volume (mL/g)
2898.22	624	0.6174	0.0662	4.549×10^{-4}	7.272×10^{-1}
3598.35	503	0.6262	0.0088	7.264×10^{-5}	9.386×10^{-2}
4497.89	402	0.6270	0.0008	7.522×10^{-6}	7.802×10^{-3}
5598.07	323	0.6270	0.0000	0.000×10^0	0.000×10^0
6897.69	262	0.6270	0.0000	0.000×10^0	0.000×10^0
8596.67	210	0.6270	0.0000	0.000×10^0	0.000×10^0
10595.98	171	0.6270	0.0000	0.000×10^0	0.000×10^0
13194.36	137	0.6270	0.0000	0.000×10^0	0.000×10^0
14794.05	122	0.6270	0.0000	0.000×10^0	0.000×10^0
16390.96	110	0.6270	0.0000	0.000×10^0	0.000×10^0
19985.98	90	0.6270	0.0000	0.000×10^0	0.000×10^0
24994.60	72	0.6270	0.0000	0.000×10^0	0.000×10^0
29995.93	60	0.6270	0.0000	0.000×10^0	0.000×10^0
27299.74	66	0.6069	-0.0201	3.379×10^{-3}	4.920×10^{-1}
21002.69	86	0.6063	-0.0006	2.906×10^{-5}	5.068×10^{-3}
16001.31	113	0.6063	0.0000	0.000×10^0	0.000×10^0
12395.28	146	0.6063	0.0000	0.000×10^0	0.000×10^0
9588.91	189	0.6063	0.0000	0.000×10^0	0.000×10^0
7299.01	248	0.6063	0.0000	0.000×10^0	0.000×10^0
5710.54	317	0.6063	0.0000	0.000×10^0	0.000×10^0
4300.52	421	0.6063	0.0000	0.000×10^0	0.000×10^0
3305.61	547	0.6063	0.0000	0.000×10^0	0.000×10^0
2596.56	697	0.6063	0.0000	0.000×10^0	0.000×10^0
1994.70	907	0.6063	0.0000	0.000×10^0	0.000×10^0
1501.20	1205	0.6063	0.0000	0.000×10^0	0.000×10^0
1199.75	1508	0.6063	0.0000	0.000×10^0	0.000×10^0
900.51	2008	0.6063	0.0000	0.000×10^0	0.000×10^0
699.21	2587	0.6063	0.0000	0.000×10^0	0.000×10^0
499.81	3619	0.5951	-0.0112	1.082×10^{-5}	7.659×10^{-2}
400.38	4517	0.5475	-0.0476	5.294×10^{-5}	4.939×10^{-1}
300.09	6027	0.4649	-0.0827	5.475×10^{-5}	6.601×10^{-1}
239.90	7539	0.4560	-0.0089	5.855×10^{-6}	9.107×10^{-2}
189.74	9532	0.4540	-0.0021	1.039×10^{-6}	2.032×10^{-2}
144.37	12528	0.4530	-0.0010	3.171×10^{-7}	8.004×10^{-3}



MICROMERITICS INSTRUMENT CORPORATION

AutoPore IV 9500 V1.09

Serial: 335

Port: 2/1

Page 4

Sample ID: Allumina
Operator:
Submitter: Donazzi-Maestri
File: E:\9500_D~1\2016\PO2316.SMP

LP Analysis Time: 27/04/2016 14.59.45
HP Analysis Time: 28/04/2016 10.21.51
Report Time: 29/04/2016 14.14.24

Sample Weight: 0.3400 g
Correction Type: Formula
Show Neg. Int: No

Tabular Report

Pressure (psia)	Pore Diameter (A)	Cumulative Pore Volume (mL/g)	Incremental Pore Volume (mL/g)	dV/dD Pore Volume (mL/g/A)	dV/dlogD Pore Volume (mL/g)
109.25	16555	0.4521	-0.0009	2.189×10^{-7}	7.282×10^{-3}
84.92	21299	0.4512	-0.0009	1.934×10^{-7}	8.385×10^{-3}
65.40	27656	0.4499	-0.0013	2.033×10^{-7}	1.139×10^{-2}
51.49	35123	0.4483	-0.0016	2.126×10^{-7}	1.529×10^{-2}
33.17	54520	0.4439	-0.0044	2.288×10^{-7}	2.324×10^{-2}
19.70	91829	0.4320	-0.0119	3.194×10^{-7}	5.263×10^{-2}

Sample ID: Allumina
Operator:
Submitter: Donazzi-Maestri
File: E:\9500_D~1\2016\PO2316.SMP

LP Analysis Time: 27/04/2016 14.59.45
HP Analysis Time: 28/04/2016 10.21.51
Report Time: 29/04/2016 14.14.24

Sample Weight: 0.3400 g
Correction Type: Formula
Show Neg. Int: No





MICROMERITICS INSTRUMENT CORPORATION

AutoPore IV 9500 V1.09

Serial: 335

Port: 2/1

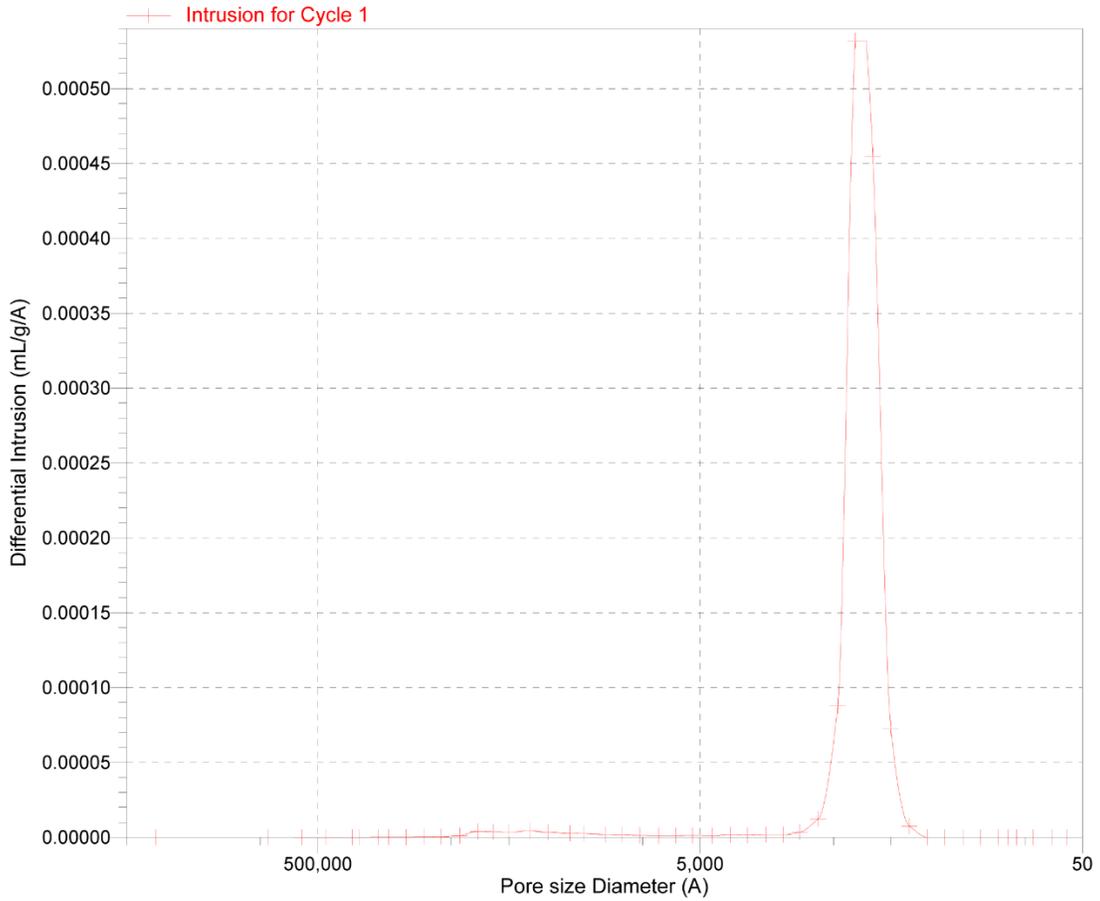
Page 7

Sample ID: Allumina
Operator:
Submitter: Donazzi-Maestri
File: E:\9500_D~1\2016\IP02316.SMP

LP Analysis Time: 27/04/2016 14.59.45
HP Analysis Time: 28/04/2016 10.21.51
Report Time: 29/04/2016 14.14.24

Sample Weight: 0.3400 g
Correction Type: Formula
Show Neg. Int: No

Differential Intrusion vs Pore size





MICROMERITICS INSTRUMENT CORPORATION

AutoPore IV 9500 V1.09

Serial: 335

Port: 2/1

Page 8

Sample ID: Allumina
Operator:
Submitter: Donazzi-Maestri
File: E:\9500_D~1\2016\IP02316.SMP

LP Analysis Time: 27/04/2016 14.59.45
HP Analysis Time: 28/04/2016 10.21.51
Report Time: 29/04/2016 14.14.24

Sample Weight: 0.3400 g
Correction Type: Formula
Show Neg. Int: No

