Supporting Information
for

Integration of enabling methods for the automated flow preparation of piperazine-2-carboxamide

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Experimental data
Experimental section

General chemistry

$^1$H NMR spectra were recorded on a Bruker Avance DPX-400 spectrometer with the residual solvent peak as the internal reference ($\text{CDCl}_3 = 7.26 \text{ ppm}$, $d_6$-DMSO = 2.50 ppm). $^1$H resonances are reported to the nearest 0.01 ppm. $^{13}$C NMR spectra were recorded on the same spectrometers with the central resonance of the solvent peak as the internal reference ($\text{CDCl}_3 = 77.16 \text{ ppm}$, $d_6$-DMSO = 39.52 ppm). All $^{13}$C resonances are reported to the nearest 0.1 ppm. DEPT 135, COSY, HMQC, and HMBC experiments were used to aid structural determination and spectral assignment. The multiplicity of $^1$H signals are indicated as: $s =$ singlet, $d =$ doublet, $dd =$ doublet of doublet, $ddd =$ doublet of doublet of doublet, $t =$ triplet, $q =$ quadruplet, $sext =$ sextet, $m =$ multiplet, br. = broad, or combinations of thereof. Coupling constants ($J$) are quoted in Hz and reported to the nearest 0.1 Hz. Where appropriate, averages of the signals from peaks displaying multiplicity were used to calculate the value of the coupling constant. Infrared spectra were recorded neat on a PerkinElmer Spectrum One FT-IR spectrometer using Universal ATR sampling accessories. Unless stated otherwise, reagents were obtained from commercial sources and used without purification. Hydrous zirconia was kindly gifted from MEL Chemicals (cod. XZO 631/01) [1]. The removal of solvent under reduced pressure was carried out on a standard rotary evaporator. Melting points were performed on a Stanford Research Systems MPA100 (OptiMelt) automated melting point system and are uncorrected. High resolution mass spectrometry (HRMS) was performed using a Waters Micromass LCT Premier™ spectrometer using time of flight with positive ESI, or conducted by Mr Paul Skelton (Department of Chemistry, University of Cambridge) on a Bruker BioApex 47e FTICR spectrometer using (positive) ESI or EI at 70 ev to within a tolerance of 5 ppm of the theoretically calculated value. Two FlowIR™ spectrometers (silicon and diamond window respectively) from Mettler Toledo were used for the in-line analyses of the two steps [2]. BET analyses were performed using a Tristar 3000 apparatus (Micromeritics) [3] at the Department of Material Sciences and Metallurgy, University of Cambridge. The flow hydration reaction was performed using a Vapourtec R2+/R4 flow platform [4]. A Knauer K-120 HPLC pump [5] was used for the hydrogenation step, in combination with a ThalesNano H-Cube® reactor [6].
Flow procedure for the synthesis of pyrazine-2-carboxamide. A solution of nitrile 3 in ethanol/H$_2$O (0.6 M, 8:1 v/v) was passed through the column reactor R2 (100 mm × 10 mm, 5 g hydrous zirconia) heated at 100 °C, with a residence time of 20 minutes, to obtain a quantitative recovery of the primary amide 2 after concentration of the reactor output (>98% yield). White solid; m.p. 191–194 °C; δ H (400 MHz, $d_6$-DMSO, 25 °C) 7.84 (1H, br. s), 8.24 (1H, br. s), 8.70 (1H, dd, J = 2.5 Hz, J 1.5 Hz), 8.85 (1H, d, J = 2.5 Hz), 9.17 (1H, d, J = 1.5 Hz); δ C (100 MHz, CDCl$_3$, 25 °C) 143.46 (CH), 143.69 (CH), 145.18 (C), 147.46 (CH), 165.13 (C); FTIR (neat, ν): 3422, 3132, 1669, 1583, 1525, 1481, 1432, 1373, 1171, 1089, 1046, 1021, 870, 791 cm$^{-1}$; LC-MS: retention time 0.28 min, m/z [M + H]$^+$ = 124.19; HRMS (ESI): m/z calcd for C$_5$H$_6$ON$_3$+: 124.0505; found 124.0504. Elemental analysis: calcd C = 48.78%, H = 4.09%, N = 34.13%; found C = 48.60%, H = 4.19%, N = 33.70%.

Using a single stream of a Vapourtec R2/R4+ reactor, material is pumped through a polymer tubing to the glass column reactor. The output of this reactor passes through a second tubing to a 100 psi back-pressure regulator (BPR) and then through a third tubing to the switching valve V1, directing it either to waste or to be collected. All tubing is PFA with Ø 1mm.

Flow procedure for the synthesis of (R,S)-piperazine-2-carboxamide. A solution of carboxamide 2 in ethanol/H$_2$O (0.6 M, 8:1 v/v) was fed using a Knauer K-120 pump (flow rate 0.1 mL min$^{-1}$) into the H-Cube apparatus, loaded with a 10% Pd/C catalyst cartridge, heated at 100 °C to obtain a quantitative
transformation to the primary amide 1 after concentration of the reactor output (95% yield). White solid; δ H (400 MHz, MeOD, 25 °C) 1H NMR (400 MHz, MeOD) δ 2.66 (2H, ddd, J = 16.3, 7.7, 4.3 Hz), 2.80 – 2.71 (1H, m), 2.82 (1H, dt, J = 11.8, 2.7 Hz), 2.95 (1H, dt, J = 12.2, 2.7 Hz), 3.08 (1H, dd, J = 12.4, 3.4 Hz), 3.36 – 3.28 (2H, m); δ C (100 MHz, CDCl3, 25 °C) 45.56 (CH2), 46.26 (CH2), 49.63 (CH2), 59.39 (CH), 174.58 (C); FTIR (neat, ν): 3332, 3308, 3194, 2949, 2904, 2832, 1673, 1611, 1488, 1438, 1409, 1355, 1306, 1186, 1136, 1116, 1071, 1057, 1003, 959, 908, 823, 723 cm−1; LC-MS: retention time 0.26 min, m/z [M + H]+ = 130.14; HRMS (ESI): m/z calcld for C5H12ON3+: 130.0975; found 130.0979. Elemental analysis: calcd C = 46.50%, H = 8.58%, N = 32.53%; found C = 46.49%, H = 8.50%, N = 32.30%.

The reagent solution is infused using the Knauer K-120 pump into the H-Cube® via a Ø 0.5 mm PTFE tubing and Ø 0.5 mm stainless steel tubing. The output of the H-Cube® passes through Ø 0.5 mm PTFE tubing through a 100 psi BPR, then the FlowIR™ spectrometer, and then a 75 psi BPR to the collection valve V2 (Valco VICI 10-position switching valve). The second BPR was required to stop the hydrogen from blowing the solution through the FlowIR™ too rapidly, whilst not providing more pressure than the IR head can withstand.

Flow procedure for the telescoped synthesis of (R,S)-piperazine-2-carboxamide. A solution of nitrile 3 in ethanol/H2O (0.6 M, 8:1 v/v) was passed through the column reactor R2 (100 mm × 10 mm, 5 g hydrous zirconia) heated at 100 °C, with a residence time of 20 minutes. This intermediate solution was
used directly without purification in the second step. This could be performed either by matching the flow rates of the two steps, or using a reservoir arrangement as described in the main article. The intermediate solution was delivered to the H-Cube® apparatus (flow rate 0.1 mL min⁻¹) using a Knauer K-120 pump. The H-Cube® was loaded with a 10% Pd/C catalyst cartridge, which was heated at 100 °C. After concentration of the reactor output the primary amide 1 was obtained (95% yield).

*Collection reservoir*

The intermediate solution was directed into a pear shaped flask through a tube (Ø 0.5 mm PTFE, total volume 1 mL) from V1. A bent stainless steel tube allows the solution to be drawn out by the Knauer K-120 pump. An open needle allows the pressure to equalise. A plastic board gives a white background to the image captured by the camera, which is held in position relative to the flask with clamps.
### DoE run results

| Run | H₂ Pressure | Temperature /°C | Flow rate /mL min⁻¹ | Conversion | Product | Side-product 1 | Side-product 2 |
|-----|-------------|-----------------|----------------------|------------|---------|----------------|----------------|
| 1   | Full        | 100             | 0.1                  | 1.00       | 1.00    | 0.00           | 0.00           |
| 2   | 20 bar      | 100             | 0.1                  | 0.53       | 0.35    | 0.08           | 0.10           |
| 3   | 20 bar      | 40              | 0.2                  | 0.85       | 0.08    | 0.07           | 0.70           |
| 4   | Full        | 40              | 0.1                  | 0.78       | 0.74    | 0.04           | 0.00           |
| 5   | 20 bar      | 40              | 0.2                  | 0.52       | 0.34    | 0.18           | 0.00           |
| 6   | 20 bar      | 40              | 0.1                  | 0.38       | 0.26    | 0.12           | 0.00           |
| 7   | 20 bar      | 100             | 0.2                  | 0.37       | 0.34    | 0.03           | 0.00           |
| 8   | Full        | 40              | 0.2                  | 0.80       | 0.54    | 0.26           | 0.00           |
| 9   | 20 bar      | 40              | 0.1                  | 0.46       | 0.42    | 0.04           | 0.00           |
| 10  | Full        | 100             | 0.1                  | 1.00       | 1.00    | 0.00           | 0.00           |
| 11  | Full        | 40              | 0.1                  | 0.73       | 0.59    | 0.14           | 0.00           |
| 12  | Full        | 100             | 0.2                  | 1.00       | 1.00    | 0.00           | 0.00           |
| 13  | Full        | 100             | 0.2                  | 1.00       | 1.00    | 0.00           | 0.00           |
| 14  | 20 bar      | 100             | 0.1                  | 0.40       | 0.37    | 0.04           | 0.00           |
| 15  | Full        | 40              | 0.2                  | 0.75       | 0.52    | 0.23           | 0.00           |
| 16  | 20 bar      | 100             | 0.2                  | 0.44       | 0.44    | 0.00           | 0.00           |

Values calculated from NMR, based on relative integration of peaks at 9.24 ppm (starting material), 3.13 ppm (product), 3.79 ppm (side-product 1) and 3.18 ppm (side-product 2).
Design-Expert® Software
Factor Coding: Actual
SP1

Design Points

X1 = A: Pressure
X2 = B: Temperature

Actual Factor
C: Flow rate = 0.10

- B- 40.00
- B+ 100.00

B: Temperature

A: Pressure

Interaction

B: Temperature

A: Pressure
Individual devices were connected as most appropriate to the control computer. The Vapourtec unit was situated a few metres from the control computer and so an Ethernet connection was most convenient. Other devices were closer and were connected by USB or USB/Serial Adapter.

The FlowIR™ has to be controlled by the Mettler-Toledo iC IR software. This is set to perform an auto-export of data to a text file. A small script running on the laptop makes this data accessible to the control computer.

The interface server can be the same machine as the control computer. In this case it was a separate machine outside the lab. The server software can also be run on a virtual machine in the cloud allowing internet access to the experimental data. Importantly the control computer can be behind a firewall and not visible from the internet, increasing the security of the laboratory devices.

This same configuration was used for all of the experiments; a Raspberry Pi® [7] computer was used for experiments not involving a camera.
BET measurements data for the hydrous zirconia catalyst (cod. XZO631/01, MEL Chemicals).

| Relative Pressure (P/Po) | Absolute Pressure (mmHg) | Quantity Adsorbed (cm$^3$/g STP) | Elapsed Time (h:min) | Saturation Pressure (mmHg) |
|--------------------------|---------------------------|---------------------------------|----------------------|---------------------------|
| 0.010564699              | 8.25021                   | 57.8743                         | 01:09                | 781.13434                 |
| 0.030148522              | 23.54193                  | 68.0283                         | 01:17                |                           |
| 0.060813265              | 47.48395                  | 77.2127                         | 01:24                |                           |
| 0.085770453              | 66.96606                  | 83.1222                         | 01:32                |                           |
| 0.098558777              | 76.94717                  | 85.8828                         | 01:37                |                           |
| 0.117526064              | 91.74615                  | 89.8118                         | 01:43                |                           |
| 0.137303355              | 107.18528                 | 93.6985                         | 01:48                |                           |
| 0.157206557              | 122.71706                 | 97.4922                         | 01:53                |                           |
| 0.177275022              | 138.37518                 | 101.1974                        | 01:59                |                           |
| 0.197381660              | 154.06279                 | 104.8489                        | 02:04                |                           |
| 0.248717216              | 194.11777                 | 113.5836                        | 02:12                |                           |
| 0.297637232              | 232.28601                 | 121.1762                        | 02:18                |                           |
| 0.352207416              | 274.85944                 | 128.2285                        | 02:24                |                           |
| 0.397142912              | 309.91544                 | 132.6440                        | 02:28                |                           |
| 0.447467828              | 349.17444                 | 136.0688                        | 02:32                |                           |
| 0.497209032              | 387.97864                 | 138.5256                        | 02:35                |                           |
| 0.547574017              | 427.27133                 | 140.4535                        | 02:37                |                           |
| 0.597697991              | 466.37454                 | 142.1346                        | 02:39                |                           |
| 0.648074148              | 505.67145                 | 143.7969                        | 02:42                | 780.27753                 |
| 0.697307297              | 544.07977                 | 145.5344                        | 02:44                |                           |
| 0.746892425              | 582.76178                 | 147.5260                        | 02:46                |                           |
| 0.796559342              | 621.50281                 | 150.0012                        | 02:49                |                           |
| 0.818991347              | 638.99713                 | 151.3472                        | 02:51                |                           |
| 0.847971249              | 661.59570                 | 153.3857                        | 02:54                |                           |
| 0.872695282              | 690.87567                 | 155.5836                        | 02:56                |                           |
| 0.896994950              | 699.82288                 | 158.4901                        | 02:59                |                           |
| 0.921022295              | 718.55536                 | 162.5055                        | 03:02                |                           |
| 0.950040325              | 741.16235                 | 171.1658                        | 03:09                |                           |
| 0.973998912              | 759.03571                 | 186.6551                        | 03:17                |                           |
| 0.980257366              | 764.49371                 | 197.0335                        | 03:22                |                           |
| 0.991029177              | 773.07245                 | 212.1142                        | 03:23                |                           |
| 0.991359953              | 773.32092                 | 218.5023                        | 03:25                |                           |
| 0.989001368              | 771.47632                 | 208.7006                        | 03:26                |                           |
| 0.976407608              | 761.63837                 | 202.5224                        | 03:29                |                           |
| 0.970459723              | 756.96474                 | 197.0186                        | 03:32                |                           |
| 0.951428327              | 742.10150                 | 182.1891                        | 03:40                |                           |
| 0.927109244              | 723.11212                 | 171.0871                        | 03:45                |                           |
| 0.892979680              | 696.46216                 | 162.1981                        | 03:52                |                           |
| 0.867330651              | 676.44098                 | 158.2733                        | 03:56                |                           |
| 0.839817773              | 654.97528                 | 155.2783                        | 03:58                |                           |
| 0.827915726              | 645.68488                 | 154.1757                        | 04:00                |                           |
| 0.802199907              | 625.62155                 | 152.3462                        | 04:02                |                           |
| 0.754393518              | 586.24933                 | 149.6367                        | 04:05                |                           |
| 0.703653787              | 548.75024                 | 147.3666                        | 04:07                |                           |
| 0.653385425              | 509.54175                 | 145.4824                        | 04:09                |                           |
| 0.603061856              | 470.29117                 | 143.7837                        | 04:11                |                           |
| 0.552622023              | 430.90158                 | 142.2303                        | 04:14                |                           |
| 0.502505861              | 391.86157                 | 140.7400                        | 04:16                |                           |
| 0.453866465              | 353.92075                 | 138.6243                        | 04:19                |                           |
| 0.405920740              | 316.52740                 | 134.7340                        | 04:24                |                           |
Isotherm Tabular Report

| Relative | Absolute | Quantity | Elapsed | Saturation |
| Pressure | Pressure | Adsorbed | Time | Pressure |
| (P/Po) | (mmHg) | (cm³/g STP) | (h:min) | (mmHg) |
| 0.354818260 | 276.67035 | 129.2679 | 04:29 | 779.69928 |
| 0.304357259 | 237.31447 | 122.8228 | 04:35 | |
| 0.255522172 | 199.23045 | 115.2801 | 04:40 | |
| 0.204967040 | 159.82825 | 106.4465 | 04:41 | |
| 0.145712509 | 119.61194 | 95.9960 | 04:49 | |

Isotherm Linear Plot

- zro27: Adsorption
- zro27: Desorption
Langmuir Surface Area Report

Langmuir Surface Area: 485.0454 ± 19.6167 m²/g
Shape: 0.999976 ± 0.000303 g/cm³ STP
Y-Intercept: 0.155174 ± 0.034650 mmHg g/cm³ STP
b: 0.057837 l/mmHg
Qm: 111.4227 cm³/g STP
Correlation Coefficient: 0.993521
Molecular Cross-Sectional Area: 0.1620 nm²

| Pressure (mmHg) | Quantity Adsorbed (cm³/g STP) | PIQ (mmHg g/cm² STP) |
|-----------------|-------------------------------|----------------------|
| 8.25021         | 57.8743                       | 0.143                |
| 23.54193        | 68.0253                       | 0.346                |
| 47.48395        | 77.2127                       | 0.615                |
| 66.96606        | 83.1222                       | 0.806                |
| 76.94717        | 85.8026                       | 0.896                |
| 91.74615        | 89.8118                       | 1.022                |
| 107.18528       | 93.6985                       | 1.144                |
| 122.71706       | 97.4922                       | 1.259                |
| 156.37516       | 101.1974                      | 1.367                |
| 154.96279       | 104.9489                      | 1.499                |
Langmuir Surface Area Plot

P/Q (mmHg g/cm² STP) vs Pressure (mmHg)
## t-Plot Report

- **Micropore Volume:** 0.019061 cm³/g STP
- **Micropore Area:** *
- **External Surface Area:** 415.0610 m²/g
- **Slope:** 268.336260 ± 2.478795 cm³/g-nm STP
- **Y-Intercept:** -12.322642 ± 1.033230 cm³/g STP
- **Correlation Coefficient:** 0.999702

### Surface Area Correction Factor
- **Density Conversion Factor:** 0.0015468

### Total Surface Area (SET):
- 376.5479 m²/g

### Thickness Range:
- 0.35000 nm to 0.50000 nm

### Thickness Equation:
- Harkins and Jura

\[
t = \left[ \frac{13.99}{(0.034 - \log(P/P₀))} \right] ^ {0.5}
\]

| Relative Pressure (P/P₀) | Statistical Thickness (nm) | Quantity Adsorbed (cm³/g STP) |
|--------------------------|-----------------------------|--------------------------------|
| 0.010654699              | 0.26381                     | 57.8743                        |
| 0.030148522              | 0.29997                     | 68.0283                        |
| 0.060813285              | 0.33454                     | 77.2127                        |
| 0.085770453              | 0.35652                     | 83.1222                        |
| 0.098555777              | 0.36271                     | 85.5628                        |
| 0.117320648              | 0.38097                     | 89.6118                        |
| 0.137303355              | 0.39507                     | 93.6965                        |
| 0.157226557              | 0.40870                     | 97.4922                        |
| 0.177276022              | 0.42206                     | 101.1974                       |
| 0.197381660              | 0.43519                     | 104.8489                       |
| 0.248717216              | 0.46816                     | 113.5838                       |
| 0.297637232              | 0.49968                     | 121.1762                       |
| 0.352207416              | 0.53586                     | 128.2265                       |
| 0.397142912              | 0.56707                     | 132.6440                       |
| 0.447467828              | 0.60419                     | 136.6868                       |
| 0.497200032              | 0.64387                     | 138.3256                       |
| 0.547574017              | 0.68800                     | 140.4535                       |
| 0.597697991              | 0.73706                     | 142.1346                       |
| 0.649074146              | 0.79317                     | 143.7969                       |

* The micropore area is not reported because either the micropore volume is negative or the calculated external surface area is larger than the total surface area.
| Pore Diameter Range (nm) | Average Diameter (nm) | Incremental Porous Volume (cm$^3$) | Cumulative Porous Volume (cm$^3$) | Incremental Porous Area (m$^2$) | Cumulative Porous Area (m$^2$) |
|-------------------------|-----------------------|-----------------------------------|---------------------------------|-------------------------------|-------------------------------|
| 2.5 - 21.7             | 21.3                  | 0.010424                           | 0.010424                        | 0.186                         | 0.186                         |
| 21.7 - 51.9            | 51.7                  | 0.025299                           | 0.035723                        | 0.844                         | 1.030                         |
| 51.9 - 73.0            | 72.7                  | 0.017480                           | 0.053203                        | 0.898                         | 1.928                         |
| 73.0 - 125.0           | 125.2                 | 0.026706                           | 0.080033                        | 2.240                         | 4.181                         |
| 125.0 - 159.0          | 159.1                 | 0.015150                           | 0.115180                        | 6.138                         | 6.138                         |
| 159.0 - 221.0          | 221.0                 | 0.007005                           | 0.112185                        | 7.399                         | 7.399                         |
| 221.0 - 250.0          | 250.0                 | 0.005125                           | 0.117310                        | 8.551                         | 8.551                         |
| 250.0 - 318.0          | 318.0                 | 0.003864                           | 0.111164                        | 9.603                         | 9.603                         |
| 318.0 - 416.0          | 416.0                 | 0.002859                           | 0.114023                        | 10.775                        | 10.775                        |
| 416.0 - 104            | 104.9                 | 0.002458                           | 0.117033                        | 11.675                        | 11.675                        |
| 104.9 - 9.4            | 9.1                   | 0.004681                           | 0.121714                        | 13.721                        | 13.721                        |
| 9.4 - 7.0              | 7.5                   | 0.003862                           | 0.125576                        | 15.787                        | 15.787                        |
| 7.0 - 5.2              | 5.5                   | 0.003252                           | 0.128828                        | 17.996                        | 17.996                        |
| 5.2 - 4.6              | 4.9                   | 0.003492                           | 0.132320                        | 20.507                        | 20.507                        |
| 4.6 - 4.1              | 4.3                   | 0.004885                           | 0.137205                        | 23.075                        | 23.075                        |
| 4.1 - 3.7              | 3.9                   | 0.006526                           | 0.143731                        | 25.343                        | 25.343                        |
| 3.7 - 3.3              | 3.5                   | 0.010512                           | 0.154243                        | 30.002                        | 30.002                        |
| 3.3 - 3.0              | 3.1                   | 0.015348                           | 0.179601                        | 39.017                        | 39.017                        |
| 3.0 - 2.7              | 2.8                   | 0.020738                           | 0.200339                        | 59.466                        | 59.466                        |
| 2.7 - 2.5              | 2.6                   | 0.023585                           | 0.224024                        | 79.037                        | 79.037                        |
| 2.5 - 2.2              | 2.3                   | 0.035009                           | 0.260333                        | 108.307                       | 108.307                       |
| 2.2 - 2.1              | 2.1                   | 0.046725                           | 0.270058                        | 141.084                       | 141.084                       |
| 2.1 - 2.0              | 2.0                   | 0.014781                           | 0.284885                        | 297.197                       | 297.197                       |

BJH Adsorption Pore Distribution Report

Diameter Range: 1.7000 nm to 300.0000 nm
Adsorbate Property Factor: 0.06300 nm
Density Conversion Factor: 0.0015-0.088
Fraction of Pores Open at Both Ends: 0.00
### BJH Desorption Pore Distribution Report

\[ t = 3.54 \frac{1}{(-5 \ln(P/P_0))^{0.333}} \]

Diameter Range: 1.7000 nm to 300.0000 nm

Adsorbate Property Factor: 0.95300 nm

Density Conversion Factor: 0.0013468

Fraction of Pores Open at Both Ends: 0.00

| Pore Diameter Range (nm) | Average Diameter (nm) | Incremental Pore Volume (cm³/g) | Cumulative Pore Volume (cm³/g) | Incremental Pore Area (m²/g) | Cumulative Pore Area (m²/g) |
|--------------------------|-----------------------|---------------------------------|--------------------------------|------------------------------|-----------------------------|
| 225.5 - 1778             | 196.0                 | 0.016037                        | 0.016037                       | 0.327                        | 0.327                       |
| 177.8 - 840              | 100.6                 | 0.010193                        | 0.026230                       | 0.405                        | 0.732                       |
| 84.0 - 675               | 73.9                  | 0.009365                        | 0.035694                       | 0.507                        | 1.239                       |
| 67.5 - 416               | 48.5                  | 0.025980                        | 0.061574                       | 2.144                        | 3.383                       |
| 41.6 - 280               | 32.1                  | 0.019882                        | 0.081436                       | 2.472                        | 5.855                       |
| 28.0 - 193               | 22.0                  | 0.016239                        | 0.097675                       | 2.947                        | 8.803                       |
| 19.3 - 157               | 17.1                  | 0.007118                        | 0.104793                       | 1.863                        | 10.466                      |
| 15.7 - 13.1              | 14.1                  | 0.009435                        | 0.112028                       | 1.538                        | 12.004                      |
| 13.1 - 12.2              | 12.6                  | 0.002016                        | 0.114244                       | 0.640                        | 12.644                      |
| 12.2 - 107               | 11.3                  | 0.003276                        | 0.115520                       | 1.159                        | 13.803                      |
| 10.7 - 8.6               | 9.4                   | 0.004954                        | 0.120474                       | 2.112                        | 15.914                      |
| 8.6 - 7.1                | 7.7                   | 0.004252                        | 0.124725                       | 2.205                        | 18.119                      |
| 7.1 - 6.1                | 6.5                   | 0.003625                        | 0.128350                       | 2.228                        | 20.345                      |
| 6.1 - 5.3                | 5.6                   | 0.003413                        | 0.131763                       | 2.429                        | 22.774                      |
| 5.3 - 4.7                | 4.9                   | 0.003226                        | 0.134989                       | 2.651                        | 25.406                      |
| 4.7 - 4.1                | 4.4                   | 0.003280                        | 0.138280                       | 3.010                        | 28.414                      |
| 4.1 - 3.7                | 3.9                   | 0.005721                        | 0.144001                       | 5.864                        | 34.278                      |
| 3.7 - 3.4                | 3.5                   | 0.012572                        | 0.156573                       | 14.281                       | 48.559                      |
| 3.4 - 3.0                | 3.2                   | 0.018965                        | 0.175538                       | 23.886                       | 72.425                      |
| 3.0 - 2.7                | 2.9                   | 0.023533                        | 0.199071                       | 32.793                       | 105.218                     |
| 2.7 - 2.5                | 2.6                   | 0.028021                        | 0.228093                       | 44.661                       | 149.879                     |
| 2.5 - 2.2                | 2.3                   | 0.035220                        | 0.263313                       | 60.037                       | 209.918                     |
| 2.2 - 2.0                | 2.1                   | 0.044453                        | 0.307766                       | 85.663                       | 295.579                     |
Summary Report

Surface Area
Single point surface area at P/Po = 0.197381660: 366.3377 m²/g

BET Surface Area: 376.5479 m²/g

Langmuir Surface Area: 485.0454 m²/g

t-Plot External Surface Area: 415.0610 m²/g

BJH Adsorption cumulative surface area of pores between 1.7000 nm and 300.0000 nm diameter: 297.8170 m²/g

BJH Desorption cumulative surface area of pores between 1.7000 nm and 300.0000 nm diameter: 295.5793 m²/g

Pore Volume
Single point adsorption total pore volume of pores less than 73.6426 nm diameter at P/Po = 0.972998912: 0.288718 cm³/g

t-Plot micropore volume: -0.019061 cm³/g

BJH Adsorption cumulative volume of pores between 1.7000 nm and 300.0000 nm diameter: 0.309870 cm³/g

BJH Desorption cumulative volume of pores between 1.7000 nm and 300.0000 nm diameter: 0.307766 cm³/g

Pore Size
Adsorption average pore width (4V/A by BET): 3.06700 nm

BJH Adsorption average pore diameter (4V/A): 4.1619 nm

BJH Desorption average pore diameter (4V/A): 4.1649 nm
References

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