# AccuPyc® 1345

## Gas Displacement Pycnometer Series

micromeritics®

Effective Solutions for
Material Characterization

# Calculations

Dec 2019

# CORPORATE PROFILE

Micromeritics Instrument Corporation is a leading global provider of solutions for material characterization with best-in-class instrumentation and application expertise in five core areas: density; surface area and porosity; particle size and shape; powder characterization; and catalyst characterization and process development. Founded in 1962, the company is headquartered in Norcross, Georgia, USA and has more than 300 employees worldwide. With a fully integrated operation that extends from a world class scientific knowledge base through to in-house manufacture, Micromeritics delivers an extensive range of high-performance products for academic research and industrial problem-solving. The implementation of tactical partnerships to incubate and deliver valuable new technologies exemplifies the company's holistic, customer-centric approach which extends to a cost-efficient contract testing laboratory – the Particle Testing Authority (PTA). The strategic acquisitions of Freeman Technology Ltd and Process Integral Development S.L. (PID Eng & Tech) reflect an ongoing commitment to optimized, integrated solutions in the industrially vital areas of powders and catalysis.

Freeman Technology (Tewkesbury, UK) brings market-leading powder characterization technology to Micromeritics' existing portfolio of particle characterization techniques. The result is a suite of products that directly supports efforts to understand and engineer particle properties to meet powder performance targets. With over 15 years of experience in powder testing, Freeman Technology specializes in systems for measuring the flow properties of powders. In combination with detailed application know-how these systems deliver unrivalled insight into powder behavior supporting development, formulation, scale-up, processing and manufacture across a wide range of industrial sectors.

PID Eng & Tech (Madrid, Spain) complements Micromeritics' renowned offering for catalyst characterization with technology for the measurement and optimization of catalytic activity, with a product range that extends to both standard and bespoke pilot scale equipment. Launched in 2003, PID Eng & Tech is a leading provider of automated, modular microreactor systems for the detailed investigation of reaction kinetics and yield. These products are supported by a highly skilled multidisciplinary team of engineers with in-depth expertise in the design, construction and operation of laboratory units and process scale-up.

The Particle Testing Authority (PTA) provides material characterization services for fine powders and solid materials using Micromeritics' instrumentation alongside complementary solutions from other vendors. With the certification and expertise to operate across a wide range of industries the PTA offering runs from single sample analysis to complex method development, method validation, new product assessment, and the analytical support required for large scale manufacturing projects. An experienced, highly trained team of scientists, engineers, and lab technicians works closely with every client to ensure that all analytical requirements are rapidly and responsively addressed.

Micromeritics has a strong global network with offices across the Americas, Asia, and Europe complemented by a dedicated team of distributors in additional locations. This ensures that local, knowledgeable support is available for every customer, in academia or industry. Micromeritics works across a truly diverse range of industries from oil processing, petrochemicals and catalysts, to food and pharmaceuticals, and at the forefront of characterization technology for next generation materials such as graphene, metal-organic-frameworks, nanocatalysts, and zeolites. Engineering solutions that work optimally for every user is a defining characteristic of the company.

# CONTACT US

**Micromeritics Instrument Corporation**
4356 Communications Drive
Norcross, GA / USA / 30093-2901
Phone: 1-770-662-3636
Fax: 1-770-662-3696
www.Micromeritics.com

**Instrument Service or Repair**
Phone: 1-770-662-3666
International — contact your local distributor or call 1-770-662-3666
Service.Helpdesk@Micromeritics.com

**Micromeritics Learning Center**
Phone: 1-770-662-3607
www.Micro.edu

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# Table of Contents

# ANALYSIS

For derivation, see the Sample Volume Equation Derivation section of the *AccuPyc Operator Manual*.

## 10, 100, AND 350 $CM^3$ UNITS

$$V_s = V_c - \frac{V_x}{\frac{P_1}{P_2 - 1}}$$

$$\rho_s = \frac{m_s}{V_s}$$

where

| | | |
|---|---|---|
| $V_c$ | = | sample chamber volume |
| $V_x$ | = | expansion chamber volume |
| $V_s$ | = | sample volume |
| $m_s$ | = | sample mass |
| $\rho_s$ | = | sample density |
| $P_1$ | = | gauge pressure after fill |
| $P_2$ | = | gauge pressure after expansion |

## 1 CM$^3$ AND 2000 CM$^3$ UNITS

$$V_s = V_c - V_x\left(\frac{P_1}{P_2} - 1\right)$$

$$\rho_s = \frac{m_s}{V_s}$$

where

| | | |
|---|---|---|
| $V_c$ | = | sample chamber volume |
| $V_x$ | = | expansion chamber volume |
| $V_s$ | = | sample volume |
| $m_s$ | = | sample mass |
| $\rho_s$ | = | sample density |
| $P_1$ | = | gauge pressure after fill |
| $P_2$ | = | gauge pressure after expansion |

# ASPHALT DENSITY

Sample volume at 60 °F is found by multiplying the measured volume by the asphalt volume correction factor.

$$V(60) = \alpha(T)V_{\text{measured}}$$

where

$$\alpha(T) = 1.0211326242 - 3.548988118 \times 10^{4}T + 4.498813 \times 10^{-8}T^{2}$$

and T is the analysis temperature in degrees Fahrenheit.

Sample density at 60 °F is

$$\rho(60) = m/V(60)$$

Specific gravity at 60 °F is calculated by dividing the adjusted sample density by the density of water at 60 °F.

$$SG(60) = \rho(60)/\rho_{\text{H2O}}(60)$$

where

$$\rho_{\text{H2O}}(60) = 0.9990170$$

Note that the density of water at the analysis temperature is not required for this calculation.

# FOAMPYC METHODS

## CORRECTION USING CELL DIMENSIONS

$$\text{OpenCellPct} = \frac{\text{GeomVol} - V_s - \text{VolCellsCutOpen}}{\text{GeomVol}} \times 100$$

$$\text{WallPlusClosedCellPct} = 100 - \text{OpenCellPct}$$

If cell measure is **Chord Length**,

$$\text{VolCellsCutOpen} = \text{ActiveArea} \times \frac{\text{ChordLength}}{1.14}$$

If cell measure is **Diameter**,

$$\text{VolCellsCutOpen} = \text{ActiveArea} \times \frac{\text{CellDiam}}{1.4515}$$

## CORRECTION BY RECUTTING SAMPLE

$$\text{VolCellsCutOpen} = V_s[1] - V_s[2]$$

$$\text{WallPlusClosedCellVol} = V_s[1] + \text{VolCellsCutOpen}$$

$$\text{OpenCellVol} = \text{GeomVol} - \text{WallPlusClosedCellVol}$$

$$\text{WallPlusClosedCellPct} = \frac{\text{WallPlusClosedCellVol}}{\text{GeomVol}} \times 100$$

$$\text{OpenCellPct} = \frac{\text{OpenCellVol}}{\text{GeomVol}} \times 100$$

where

$V_s[1]$ = $V_s$ from the first analysis (before recutting)
$V_s[2]$ = $V_s$ from the second analysis (after recutting)
$V_s[1]$ = $V_s$ from the first analysis (before recutting)

$$\text{OpenCellVol} = \text{GeomVol} - V_S$$

$$\text{OpenCellPct} = \frac{\text{OpenCellVol}}{\text{GeomVol}} \times 100$$

## COMPRESSIBILITY TEST

Quantities appended with [i] are for cycle i, where i goes from 1 up to the number of cycles.

$$\Delta V_{P_1}[i] = \frac{V_s[i-1]-V_s[i]}{P_1[i-1]-P_1[i]}$$

$$\Delta V_{P_2}[i] = \frac{V_s[i-1]-V_s[i]}{P_2[i-1]-P_2[i]}$$

$$\mathrm{AvgVolChangeP1} = \mathrm{avg}(\Delta_{P1}[i] \ \ \mathrm{forallincludedi})$$

$$\mathrm{AvgVolChangeP2} = \mathrm{avg}(\Delta_{P2}[i] \ \ \mathrm{forallincludedi})$$

$$\mathrm{PctAvgVolChangeP1} = \frac{\mathrm{AvgVolChangeP1}}{\mathrm{GeomVol}} \times 100$$

$$\mathrm{PctAvgVolChangeP2} = \frac{\mathrm{AvgVolChangeP2}}{\mathrm{GeomVol}} \times 100$$

## CELL FRACTURE TEST

$$\Delta\mathrm{Vol} = V_s[3] - V_s[1]$$

$$\mathrm{PctFracturedCells} = \frac{\Delta\mathrm{Vol}}{\mathrm{GeomVol}} \times 100$$

where

$V_s[1]$ = $V_s$ for the first (prefracture) cycle
$V_s[3]$ = $V_s$ for the third (postfracture) cycle

# CALIBRATION

Volume calibration uses the ultra-precise method of separate adjustments for the offset and scale factor.

## VOLUME OFFSET

$$V_c = V_{\text{celprev}} - V_{\text{sampempty}}$$

$-V_{\text{sampempty}}$ is reported as the offset in volume calibration reports.

## VOLUME SCALE

$$V_c = V_{\text{celprev}} \left( \frac{V_{\text{calib}}}{V_{\text{sampball}}} \right)$$

$$V_x = V_{\text{expprev}} \left( \frac{V_{\text{calib}}}{V_{\text{sampeball}}} \right)$$

$\left( \frac{V_{\text{calib}}}{V_{\text{sampeball}}} \right)$ is reported as the scale factor in volume calibration reports.

where

| | | |
|---|---|---|
| $V_c$ | = | sample chamber volume |
| $V_{\text{celprev}}$ | = | previously stored cell volume |
| $V_{\text{sampempty}}$ | = | average $V_{\text{samp}}$ from volume offset calibration analysis (no calibration ball) |
| $V_{\text{calib}}$ | = | calibration ball volume |
| $V_{\text{sampball}}$ | = | average $V_{\text{samp}}$ from volume scale calibration analysis (with calibration ball) |
| $V_x$ | = | expansion chamber volume |
| $V_{\text{expprev}}$ | = | previously stored expansion volume |

# GEOMETRIC VOLUME AND ACTIVE AREA

Geometric volume of the sample is calculated based on the sample shape; for Method A, Active area is also calculated.

## CUBE

$$\mathrm{GeomVol} = \mathrm{EdgeLength}^3 \times \mathrm{NumPieces}$$

$$\mathrm{ActiveArea} = \mathrm{EdgeLength}^2 \times (6 - \mathrm{NumSkins}) \times \mathrm{NumPieces}$$

## CYLINDER

$$\mathrm{GeomVol} = \pi \times \frac{\mathrm{Diam}^2}{4} \times \mathrm{Height} \times \mathrm{NumPieces}$$

$$\mathrm{ActiveArea} = [\pi \times \mathrm{Diam} \times \mathrm{Height} + \frac{\pi}{r} \times \mathrm{Diam}^2 \times (2 - \mathrm{NumSkins})] \times \mathrm{NumPieces}$$

## RECTANGLE

$$\mathrm{GeomVol} = \mathrm{LongEdge} \times \mathrm{ShortEdge} \times \mathrm{RemainEdge} \times \mathrm{NumPieces}$$

$$\begin{aligned}\mathrm{ActiveArea} = [&\mathrm{LongEdge} \times \mathrm{RemainEdge} \times (2 - \mathrm{NumLargeSkins}) + \mathrm{ShortEdge} \\ &\times \mathrm{RemainEdge} \times (2 - \mathrm{NumSmallSkins}) + \mathrm{LongEdge} \times \mathrm{ShortEdge} \\ &\times (2 - \mathrm{NumRemainSkins})] \times \mathrm{NumPieces}\end{aligned}$$

## PERCENT POROSITY

$$\mathrm{percentporosity} = (\rho_s - \rho)/\rho_s$$

# RESIN VOLUME

$$\text{ResinVol} = \frac{m_s}{\text{ResinDensity}}$$

$$\text{ResinVolPct} = \frac{\text{ResinVol}}{\text{GeomVol}} \times 100$$

where

$m_s$ = sample mass

# RUN PRECISION

Run precision requires at least five runs. Run precision criterion is met when the sample volumes calculated for the four most recent previous runs fall within the specified error band for the current run's sample volume. The error band is a specified percentage of the nominal volume of the sample chamber (1, 10, 100, 350 and/or 2000 $cm^3$).

$V_s[0]$ to $V_s[4]$ are the five most recent sample volumes, $V_s[4]$ being the most recent.

$$\mathbf{ErrBand} = \mathbf{PctFullScale} \times \frac{\mathbf{V_{nominal}}}{\mathbf{100}}$$

where

$V_{nominal}$ = nominal sample cell volume

If $(|V_s[i] - V_s[4]| \leq \mathbf{ErrBand})$ for $i$ = 0 to 3, run precision is achieved.

# SPC REPORT VARIABLES

## REGRESSION CHART VARIABLES

The line of best fit for the Regression Chart is calculated by the usual least squares method. [1] If there is only a single point or all *N* points have the same x-value, there can be no line of best fit in the standard form.

$$\bar{x} = \frac{\sum x_i}{N}$$

$$\bar{y} = \frac{\sum y_i}{N}$$

$$\text{Slope} = \frac{\sum (x_i - \bar{x})(y_i - \bar{y})}{\sum (x_i - \bar{x})^2}$$

$$\text{Intercept} = \bar{y} - \text{Slope} \cdot \bar{x}$$

The coefficient of correlation for this line is also calculated in the usual way. [2]

$$\sigma_x = \sqrt{\frac{\sum (x_4 - \bar{x})^2}{N}}$$

$$\sigma_y = \sqrt{\frac{\sum (y_i - \bar{y})^2}{N}}$$

$$\text{Cov}(x, y) = \frac{\sum (x_i - \bar{x})(y_i - \bar{y})}{N}$$

$$\text{CorrelationCoeff} = \frac{\text{Cov}(x,y)}{\sigma_x \sigma_y}$$

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[1] *BASIC Scientific Subroutines Vol II*, by F.R. Ruckdeschel, Copyright 1981 BYTE Publications/McGraw Hill, p. 16.

[2] *Mathematical Handbook for Scientists and Engineers*, G.A. Korn and T.M. Korn, McGraw Hill, Sec. 18.4. (1968)

## CONTROL CHART VARIABLES

$$\text{Mean} = \frac{\sum y_i}{N}$$

$$\text{StandardDeviation} = \sqrt{\frac{\sum (y_i - \text{Mean})^2}{N-1}}$$

$$\text{C. V.} = \frac{\text{StdDev}}{\text{Mean}}$$

$$+n\sigma = \text{Mean} + n \cdot \text{StandardDeviation}$$

$$-n\sigma = \text{Mean} - n \cdot \text{StandardDeviation}$$

# SPECIFIC GRAVITY

$$\mathrm{SG} = \rho_s / rho_w$$

where

$\rho_w$ = water density at analysis temperature

# TOTAL PORE VOLUME

Total pore volume is per gram of sample.

$$\text{TotalPoreVol} = \frac{\rho_s - \rho_{\text{bulk}}}{\rho_s \times \rho_{\text{bulk}}}$$

where

$\rho_{\text{bulk}}$ = entered bulk density

# TOTAL SOLIDS CONCENTRATION

$$\mathrm{WeightPercentSolids} = \left(1 - \frac{\rho_{\mathrm{liq}}}{\rho_s}\right) \frac{\rho_{\mathrm{sol}}}{\rho_{\mathrm{sol}} - \rho_{\mathrm{liq}}} \times 100$$

where

| | | |
|---|---|---|
| $\rho_{\mathrm{liq}}$ | = | entered liquid density |
| $\rho_{\mathrm{sol}}$ | = | entered solid density |
| $\rho_s$ | = | sample density |