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Silica Gel

Chromatographic Processing

The separation of components by absorption chromatography is a purification procedure that is widely used at all levels of organic chemistry -- from laboratory through commercial production¹.

Component separation by absorption chromatography is the result of two distinct and separate processes that occur simultaneously. The first process involves the relative affinity of each individual solute (component) for the adsorbent phase -- which, in turn, is a function of the nature of mobile phase (solvent system) and of the physio-chemical nature of the different solutes -- and is responsible for the separation of components into distinct bands. The second process involves the dispersion (diffusion) of these bands.

Optimum chromatographic resolution is obtained when the kinetics of the first process (solute adsorption/de-adsorption) are maximized and those of the second process are minimized.

Silica Gel. The column packing most widely used for adsorption chromatography is silica gel¹. The diameter of the silica gel particles range between 10 Å and 1000 Å and are linked together primarily by bridging siloxyl bonds that results in a porous, high-surface-area matrix. The small size and high porosity of these particles makes them well suited to chromatographic separation processes.

The silica gel used for liquid chromatography is generally activated by heating to 200 °C. [It has been shown that under

these conditions one molecule of water remains strongly hydrogen bonded to each silol group.] Not surprisingly, the uniformity of particle size, of porosity, and of water content (activity) can have a significant effect chromatographic on resolution – and the subsequent batch-to-batch reproducibility of product impurity profiles. The narrower the range of each these parameters, the better the chromatographic performance.

WILSHIRE Technologies offers two unmodified, high-uniformity, high-purity, chromatographic silica gels. Both are manufactured in large batch sizes to ease regulatory pressures.

Our **230-400 Mesh** (ASTM; SSA, 500-600 m²/g; MPD, 60Å) silica gel exhibits a pressure drop of 0.5 bar and is ideally suited for production-scale - gravity-feed or flash chromatography¹ - processing.

Our **200-400 Mesh** (ASTM; SSA, 300-400 m²/g; MPD, 100Å) silica gel is especially suited for situations where high-capacity, rapid gravity-feed processing is preferred. It exhibits a pressure drop of 0.1 bar.

Product Standardization and Uniformity Facilitate Direct Scale-Up. The standardization and uniformity of our silica gel pay-off in scale-up. Exhaustive analysis of such key quality factors as *Specific Surface Area* , *Pore Volume*, *Particle Size Distribution*, *Mean Pore Volume* and the continual refinement of production techniques results in highly standardized,

Wilshire Technologies

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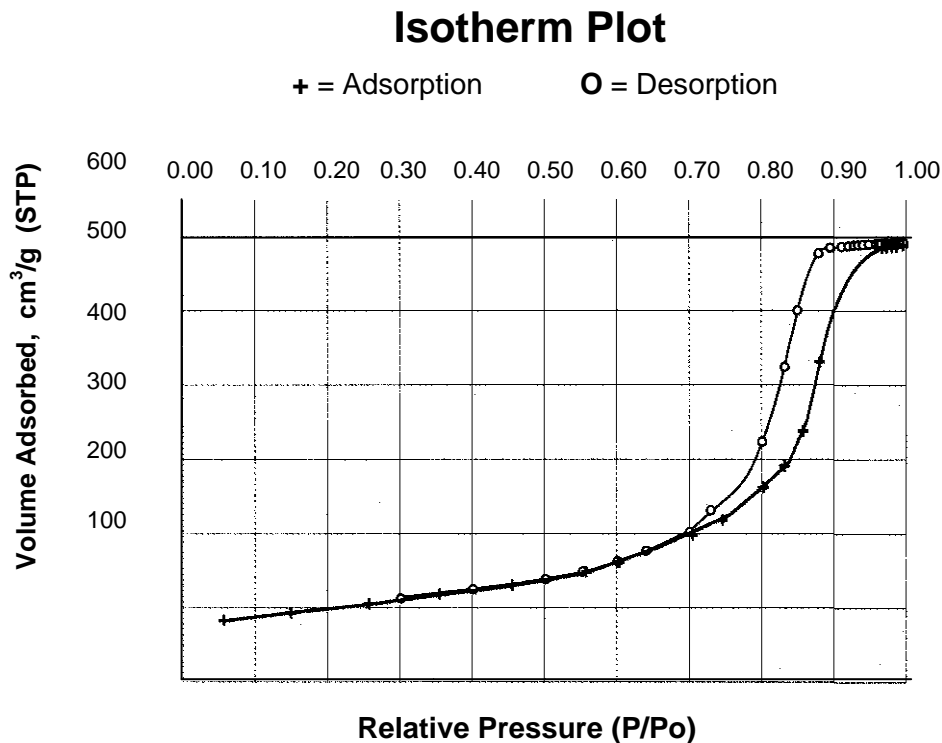
uniform silica gels which greatly simplify production scale-up.

Specific Surface Area and Pore Volume. Specific morphological characteristics for each batch of silica gel are derived from determination of the nitrogen adsorption isotherm associated with each

1. *JOC*, **43**, 2923 (1978).

production run. Analysis of the adsorption branch of the adsorption isotherm hysteresis (**Figure 1**) yields the specific surface area and the pore volume while evaluation of the desorption portion of the curve provides the pore size distribution information

Figure 1.



Particle Size Distribution (PSD). Perhaps no single physical parameter can effect the run-to-run performance of silica gel as much as particle size distribution. Until relatively recently, however, meaningful comparison of PDS data has not been readily possible. The advent of modern instrumentation for PSD determination has changed this. With such measurements, meaningful comparisons of PDS data are now readily and routinely carried out.

Figures 2 and 3 below show a comparison of the particle size distributions for two equivalent Silica Gels: Pore Volume, 0.80 + 0.05 ml/g; Surface Area (BET), 500-600 m²/g; Mean Pore Diameter, 60Å ; Particle Size, 230-400 mesh (ASTM). **Figure 2** presents the PSD observed for the product offered by a leading supplier; **Figure 3** is the PSD of the equivalent product manufactured to our specifications (see below).

Figure 2

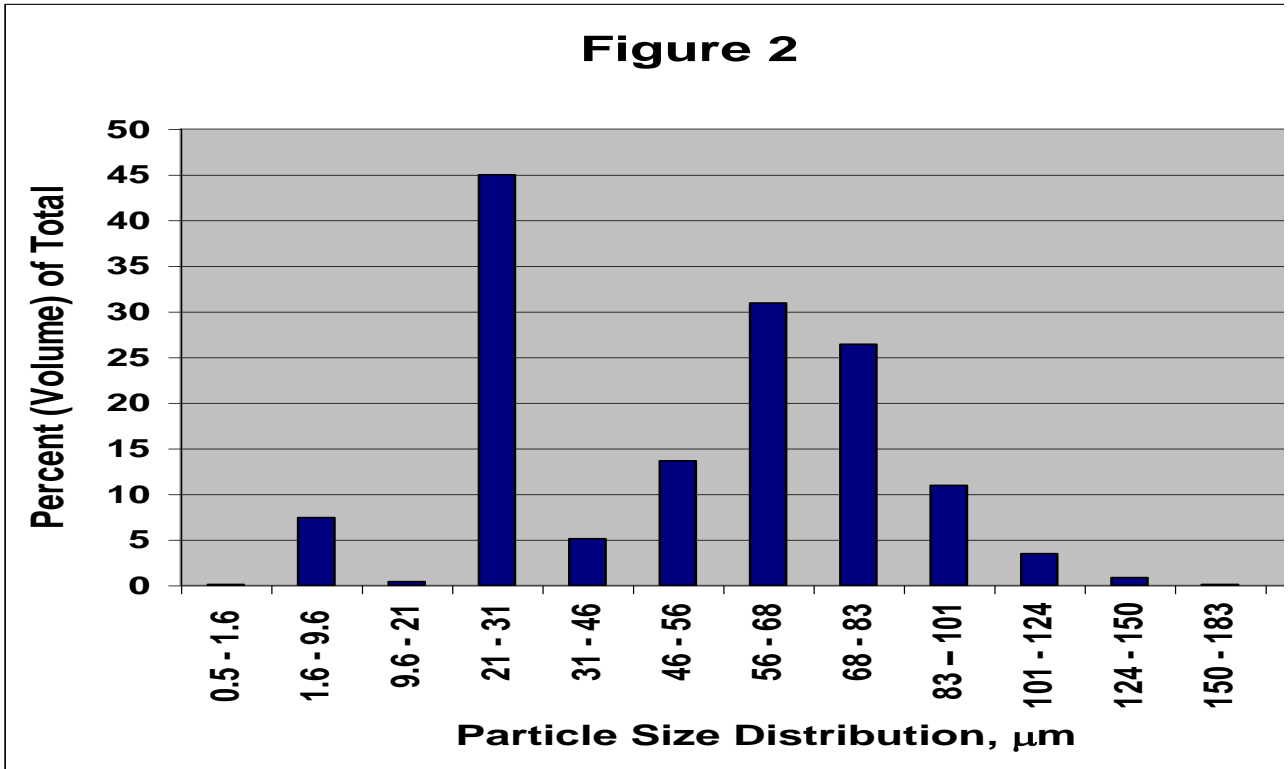
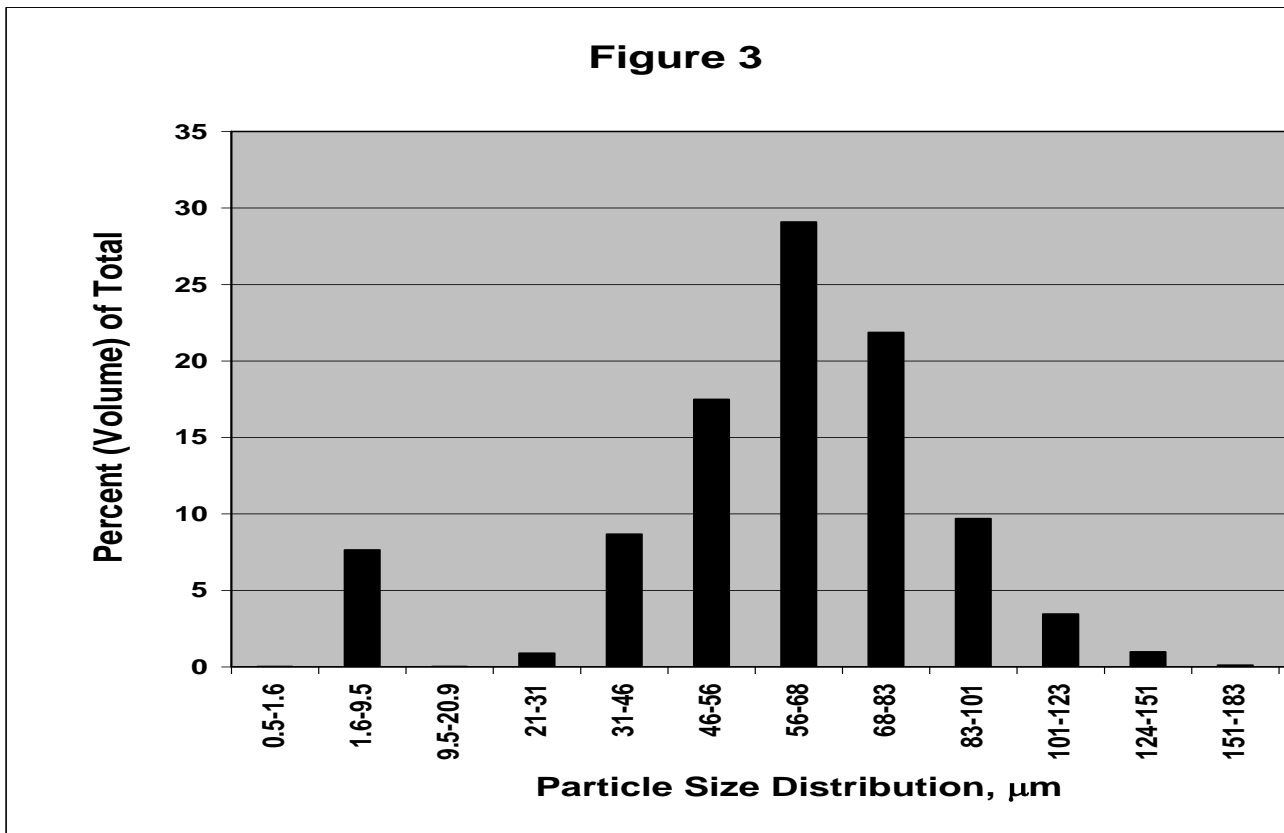


Figure 3



Comparison of the particle size distributions for two equivalent Silica Gels: Pore Volume, 0.80 + 0.05 ml/g; Surface Area (BET), 500-600 m²/g; Mean Pore Diameter, 60Å ; Particle Size, 230-400 mesh (ASTM). **Figure 2** presents the PSD observed for the product offered by a leading supplier; **Figure 3** is the PSD of the equivalent product manufactured to our specifications (see below).

Silica Gel

Optimized for Production-scale (Gravity-feed or Flash Chromatography) Processing of Molecules up to 1,000 Daltons

<u>Test</u>	<u>Specification</u>
Fe	<0.02%
Cl	< 0.01%
Loss on Drying	<4.0%
pH (10% aq. suspension)	6 – 7
Pore Volume	0.80 ± 0.05 ml/g
Surface area (BET)	500-600 m ² /g
Mean pore dia.	60 Å
Particle size	230-400 mesh (ASTM)
Pressure Drop ‡	0.5 bar

‡ @ 25°C, 100 mL/min.; Hexane/EtOH, 90/10; column: 50 x 250 mm

Silica Gel

Optimized for High-Capacity Production-scale Chromatography of Molecules up to 1,000 Daltons

<u>Test</u>	<u>Specification</u>
Fe	<0.02%
Cl	< 0.01%
Loss on Drying	< 4.0%
pH (10% aq. suspension)	6 – 7
Surface area (BET)	300-400 m ² /g
Mean pore dia.	90-100 Å
Particle size	200-400 mesh (ASTM)
Pressure Drop ‡	0.1 bar

‡ @ 25°C, 100 mL/min.; Hexane/EtOH, 90/10; column: 50 x 250 mm