

SUPELCOTM Preparative HPLC Columns

Product Specification

SUPELCOSM 10mm, 21.2mm, and 50.8mm ID preparative columns enable you to scale up an analysis from microgram quantities of material to several hundred milligrams or more. Using these columns, you can rapidly and cost effectively obtain large amounts of high purity material.

SUPELCOSM preparative columns are packed with 5 μ m or 12 μ m silica and silica-based bonded phase packings. High efficiency columns containing 5 μ m packings are available in 10mm, 21.2mm, and 50.8mm ID. Lower efficiency columns containing 12 μ m packings are available in 21.2mm and 50.8mm ID. The 21.2mm ID columns have standard compression-type fittings and are rated to use up to 3000psi; the 50.8mm ID columns have flanged fittings and are rated for use up to 2000psi.

The most important features of preparative HPLC analyses on SUPELCOSM columns are *speed* and *efficiency*. Rapid elution of compounds of interest and the ability to load large amounts of material onto the column ensure substantial savings of time and solvents. When used to isolate products from relatively complex mixtures, SUPELCOSM preparative columns are more time efficient than columns containing larger (30-50 μ m) silica particles. Furthermore, the high efficiency of SUPELCOSM preparative columns enables you to resolve closely eluting peaks, as shown in the example application on the reverse side of this specification sheet.

Silica Characteristics

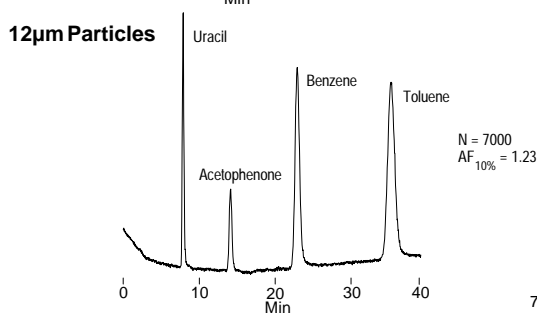
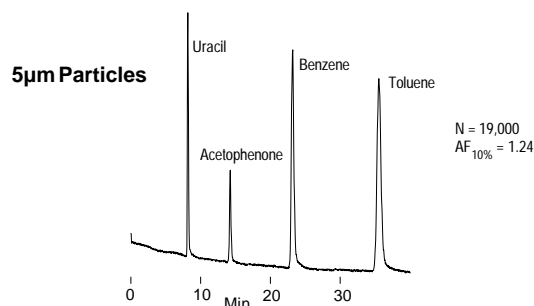
Particle Size:	5 μ m	12 μ m
Packing Composition:	silica, spherical	silica, spherical
Pore Size:	100Å	120Å
Pore Volume:	0.6mL/g	0.7mL/g
Surface Area:	170m ² /g	190m ² /g
pH Range:	2-7	2-7

Select from High or Medium Efficiency Columns

In the last 10 years, the average particle size of packings in preparative LC columns has become smaller. Modern preparative LC equipment can deliver mobile phase flow at pressures that allow the user to take full advantage of the high efficiencies of 5 μ m packings. Thus, it is possible to speed up the isolation of compounds in complex matrices. The increased speed is paid for by higher column cost. Using 10 μ m or 12 μ m particles makes sense when the compounds of interest are well separated (as is usually the case when dealing with less complex samples) or when a lower back pressure is required.

Column Test Mix on 5 μ m and 12 μ m Packings

Columns: **SUPELCOSM LC-18, 25cm x 50.8mm**
 Cat. Nos.: **54850** (5 μ m particles) **54859** (12 μ m particles)
 Mobile Phase: methanol:water, 66:34
 Flow Rate: 40mL/min
 Det.: UV, 254nm
 Inj.: 100 μ L reversed phase test mix (Cat. No. 58278)



795-0068, 0069

Ordering Information:

SUPELCOSM Preparative Columns (All columns 25cm long)

SUPELCO SM Phase	Particles (μ m)	Pores (Å)	4.6mm ID Column	10mm ID Column	21.2mm ID Column	50.8mm ID Column
LC-Si	5	100	58295	58365	54843	54844
LC-Si	12	120	59180-U	—	59183	54857
LC-CN	5	100	58231	58369	—	—
LC-1	5	100	58296	58366	—	—
LC-8	5	100	58297	58367	54845	54846
LC-8	12	120	59181	—	59184	54858
LC-8-DB	5	100	58354	58357	54847	54848
LC-18	5	100	58298	58368	54849	54850-U
LC-18	12	120	59182	—	59185	54859
LC-18-DB	5	100	58355-U	58358	54851	54852
Suplex TM pKb-100	5	100	58934	59172	—	—
LC-ABZ	5	100	59142	59170	54853	54854
ABZ ⁺ Plus	5	100	59197	—	54855	54856

We suggest developing a method by using a standard 4.6mm ID SUPELCO SIL column, then scaling the analysis up to a larger diameter column. SUPELCO SIL analytical-scale columns containing the same packings as the preparative columns listed here are described in the Supelco catalog. See the Applications section of the Supelco catalog for many examples of separations on SUPELCO SIL analytical columns.

Large-Scale Separation of α - and δ -Tocopherols

The capability of SUPELCO SIL preparative columns packed with 12 μ m spherical silica is illustrated by the isolation of α - and δ -tocopherols.

Eight forms of vitamin E (α -, β -, γ -, and δ -tocopherol and α -, β -, γ -, and δ -tocotrienol) occur naturally in oils extracted from corn, soybeans, wheat, and other grains, at concentrations from 0.1 to 100mg/mL. Although tocopherols can be analyzed by LC, TLC, or GC (after derivatization), only LC offers a sample capacity that makes the purification on a preparative scale cost effective.

We injected 1mL of a synthetic mixture of α - and δ -tocopherols in hexane (250mg/mL each isomer) onto a 25cm x 21.2mm ID SUPELCO SIL LC-Si preparative column containing 12 μ m silica particles (no bonded phase). Figure A shows the resolution of the tocopherols. Reinjection of the separated isomers onto the column revealed that the purity of each fraction was greater than 98% (Figure B). From these results, we estimate that at least 1 gram of each isomer can be purified by using a 50.8mm ID column filled with the same packing.

SUPELCO SIL LC-Si columns provide equivalent results in many other normal phase preparative applications, including separations and purifications of pharmaceuticals, steroids, triglycerides, and vitamins.

Column Care

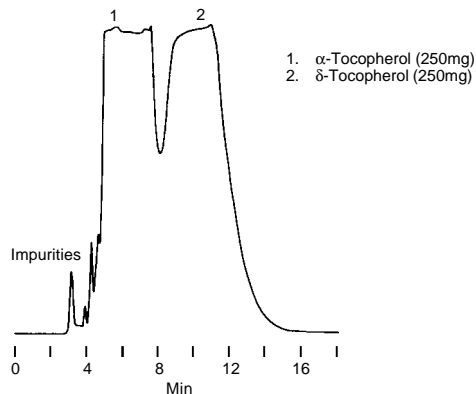
Column care is as important in preparative-scale operations as it is at analytical levels. Our tocopherol mix contained many materials (including oxidation products) that were not eluted from the column. To maintain good performance from the column, we injected 2-3mL of isopropyl alcohol after every fifth sample injection and flushed the system with methanol after every 50 injections.

If your samples might contain irreversibly retained compounds, we recommend using a guard column. We can prepare 5cm x 2.5cm ID or 5.08cm ID guard columns on request. Please call for price and delivery information.

Contact our Technical Service Department (phone 800-359-3041 or 814-359-3041, FAX 814-359-5468) for expert answers to your questions.

Figure A. Separate Large Amounts of Tocopherol Isomers

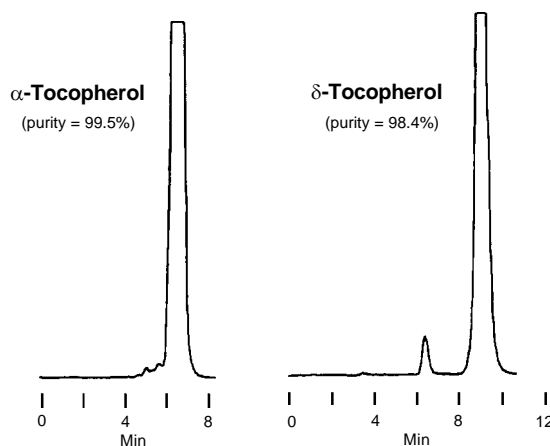
Column: **SUPELCO SIL LC-Si, 25cm x 21.2mm ID, 12 μ m particles**
 Cat. No.: **59183**
 Mobile Phase: hexane:isopropanol, 99:1
 Flow Rate: 20mL/min
 Det.: UV, 295nm (2.0 AUFS)
 Inj.: 0.5g synthetic mixture of tocopherols in 1mL hexane



713-0511

Figure B. Isomer Purity Confirmed

Column: **SUPELCO SIL LC-Si, 25cm x 4.6mm ID, 12 μ m particles**
 Cat. No.: **59180**
 Mobile Phase: hexane:isopropanol, 99:1
 Flow Rate: 1.6mL/min
 Det.: UV, 295nm (2.0 AUFS)
 Inj.: 20 μ L of peak 1 or peak 2 from A



713-0512, 0513

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For more information, or current prices, contact your nearest Supelco subsidiary listed below. To obtain further contact information, visit our website (www.sigma-aldrich.com), see the Supelco catalog, or contact Supelco, Bellefonte, PA 16823-0048 USA.

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