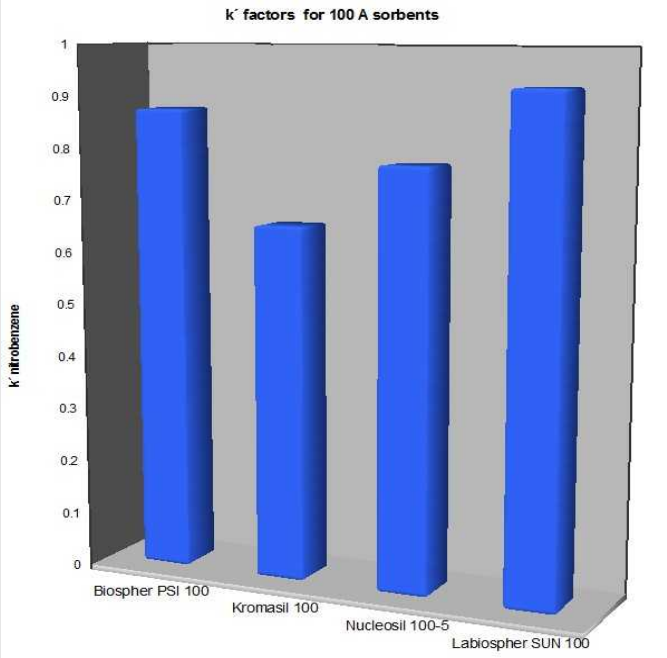
HPLC SORBENTS

LABIOSPHER

LABIOSPHER SUN

Porous silica remains nearly one hundred years the most often used sorbent in all chromatographic separations. One of the reasons is its suitability of silica for surface modifications which allows to produce new and new sorbents for range of applications. Among those the outstanding role of course play reverse phases. Their use in preparative chromatography brought new wave of interest in silica chemical stability.

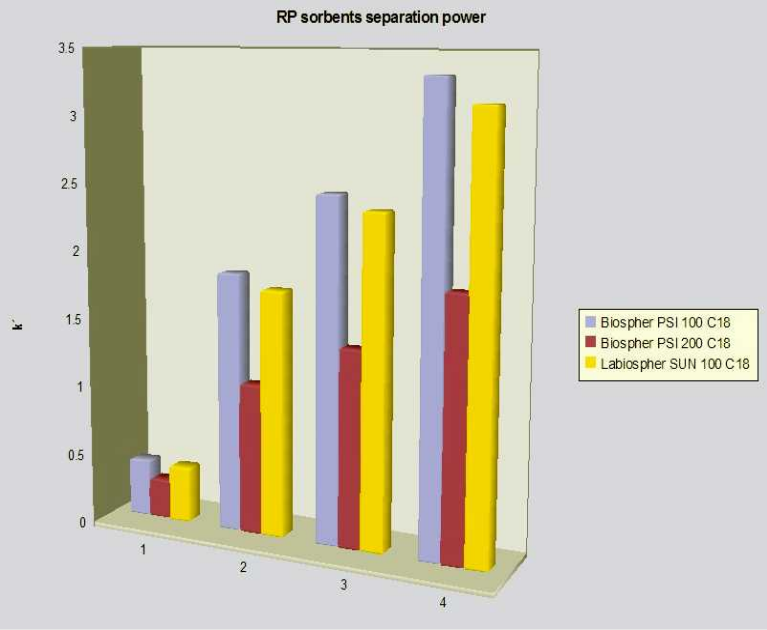
Silica is nearly ideal base for modification due to its reactivity with silanes. A price for it is unfortunately paid in a relative high solubility in water solutions, mainly under extreme pH values. In analytical applications it influences just column life time. In PLC, on the contrary, the column bed stability and life time of sorbent are critical. The price of prep column packing can reach a price of whole chromatograph.



Even that the quality of sorbent in the column is important, more important is the quantity of sorbent which is dissolved in mobile phase and can contaminate products. Dissolved silicone dioxide is usually analysed by spectrophotometry of its yellow complex with ammonium molybdate. But this reaction ignores SiO_2 which is present in the form of colloidal particles and thus gives incorrect results.

Silica particles structure is mostly made of globules. They are growing in the process of porous particles formation. Globules are often released from sorbent particle and force their destruction. Released globules - colloid particles – are drained from the column the same way as low molecular compounds. Some of them are captured by output column frit and increase the column pressure due the frit blockage. All these information support the idea of developing sorbents with lower solubility.

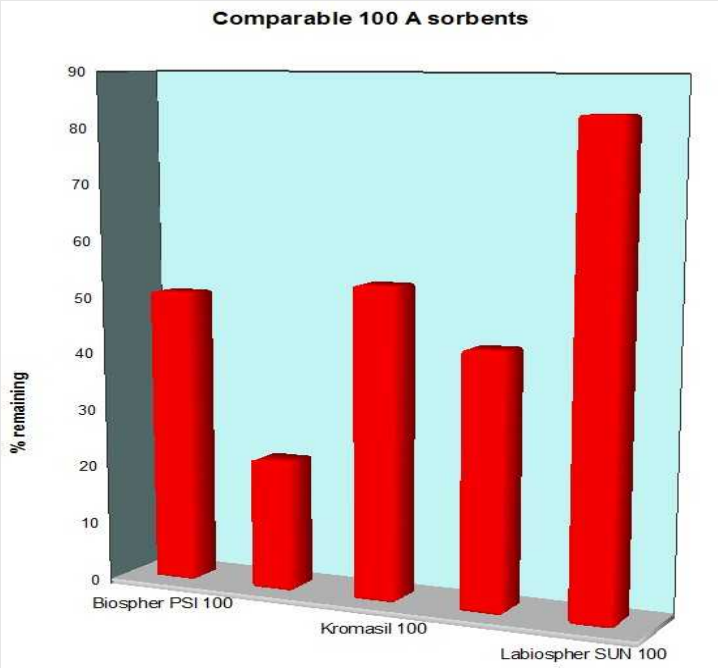
Based on silica stability studies in which dissolution in water with different NaOH concentration both in bulk and in chromatography columns was estimated, new **LABIOSPHER SUN** sorbent was developed. Due to the comparison of properties of Labio made Biospher PSI and world leading manufacturers products (spherical and unspherical) was shown that under chromatographic conditions the dissolution only slightly depends on particle size and (at least for the same manufacturing process) linearly depends on sorbent surface. We found out, that – in comparison with other properties (e.g. Sorbent density, particle size) - the major influence on dissolution of the sorbent has the silica surface. According to this knowledge we introduced a new way how to decrease the silica surface and keep in the same time the right chromatography properties. The result of this development is a new product **LABIOSPHER SUN**. Silica matrix is, as mentioned, built from



globules. Usually these globules are different in size. Their size itself and size distribution have major influence on pore distribution and surface area of porous silica. Common silica sorbents have rather broad pore distribution. Important part of pores can not be penetrated with separated molecules because of the small dimensions of pores. Thus such pores have no influence on separation power, nor can be effectively used for modifications. Nevertheless these pores are contributing to the surface measured by nitrogen adsorption and are the most active in dissolution process as well.

LABIOSPHER SUN particles are built from nearly unimodal globules, which form structure showed on the next picture. Sorbent with such structure has smaller total surface (measured with nitrogen or accessible for OH ions), but nearly all surface is accessible for molecules under the separation. It can be proved due a measurement of separation power of comparable sorbents.

LABIOSPHER SUN has the same separation power as Biospher 100 and a bit higher than Kromasil 100 and Nucleosil 100 (all sorbents were dried on 150 °C before packed to the column). The same results were found for reverse phase modifications. **LABIOSPHER SUN 100 C18** compared with Biospher PSI 100 C18 and PSI 200 C18 sorbents has shown the same separation power as his PSI 100 C18h ancestor and essentially higher than PSI 200 C18. Averages of three different lots were used. All sorbents were modified according to the same recipe. Separation factors were evaluated for phenol (1), benzene (2), dimethylaniline (3) and toluene. Column dead volume was characterized by thiourea.



Different dissolution potential of sorbents with the same chromatographic power is clearly demonstrated by a treatment with 0,01 M NaOH in water. LABIOSPHER SUN100 definitely has exhibited lowest loss of weight after the reaction.

LABIOSPHER PSI 100 & PSI 200

Spherical silica sorbents LABIOSPHER PSI (former Biospher PSI) are manufactured using technology based on colloid SiO2. Ready made silica particles are heated to the temperature 500 °C to remove all remaining organic impurities and improve mechanical stability. Subsequent acidic treatment and high quality distilled water washing is used to remove the remaining ionic substances.

LABIOSPHER PSI 100 is an universal sorbent for all applications and modifications. Its parameters (surface area 300 m^2/g, pore diameter 100 A) makes it comparable with most other manufacturers products. LABIOSPHER PSI 200 k´ factors are a bit lower and its destination is (due to relatively large pore diameter - 200 A) a chromatography of peptides (in C18 form) and other medium molecular weight compounds. LABIOSPHER PSI sorbents do not contain micropores. Internal pore volume is optimal to attain the highest possible chemical and pressure resistance and allows the access to the wholel surface.

PSI C18, C8 and C4 are the most popular sorbents for reverse phase chromatography with bonded octadecyl, octyl or butyl groups. All sorbents are produced by a double-stage reaction with endcapping, and are used for chromatography of components containing basic or acidic groups. Reverse phase sorbents can be used in pH range 1,5 – 10,5.

LABIOSPHER PSI NH2 coded sorbents contain chemically bonded aminopropyl groups. They are determined for chromatography of carbohydrates in water–acetonitrile mixtures, but can be used in chromatography with nonpolar mobile phases as well. LABIOSPHER PSI CN phase is a polar sorbent containing chemically bonded cyanoethyl groups, which exhibits more reversible sorption then pure silica. Similar use has OH type with bonded diol groups (hydrolyzed glycidoxypropylsilane).

All LABIOSPHER PSI sorbents are delivered in a range of particle sizes (fractions with d90/d10 < 1,8 – 2,0).

LABIOSPHER DS GROUP

Under the name **LABIOSPHER DS** are delivered all famous DAISOGEL sorbents. These are high quality world wide known spherical silicas , reverse phases manufactured from them as well as other modifications. RP packings featuring maximum surface coverage are the ideal choice for a wide variety of organic compounds. Carefully controlled full endcapping leads to optimal performance with acidic, basic and chelating compounds. Two most frequently used representatives of DS group are described bellow.

LABIOSPHER DS SP ODS-AP is delivered packed into all Labio made columns and there is a choice of average pore sizes between 6 nm - SP60, 12 nm - SP120, 20 nm - SP200 and 30 nm - SP300. Phases with pore size 20 nm offer a good compromise between surface area available, separation speed and solvent consumption even in analytical high speed applications.

Sorbents delivered under **LABIOSPHER DS SP 120 ODS-BP** and **LABIOSPHER DS SP 200 ODS-BP** name are spherical silica with high surface and pore volume modificated with C18 phase. They are designed to show extended selectivity for hydrophilic and polar compounds which are either not or poorly retained on other phases. A proprietary modification technique avoids the matting-down effect of the C18 chains which conventional ODS-phases show at high water contents in the mobile phase, even if pure water is used. Typical applications are separations of biomolecules and metabolites such as oligosacharides, amino acids, small peptides, nucleotides and organic acids. ODS-BP phases are fully endcapped and show similar selectivity as conventional C18 phases when being used for separations of hydrophobic compounds with typical reversed phase eluents.

LABIOSPHER ODS-BP phases show stable base lines and high sensitivity even under neutral pH conditions and without buffer or counter-ion additives, which makes them appear especially suited for hyphenated techniques like LC-MS, where those additives disturb the detection.

LABIOSPHER FS GROUP

Under the name **LABIOSPHER FS** is presented a family of FUJI Chromatorex product line of silica sorbents. High quality and wide pore size variety of spherical and irregular sorbents are offered including pure silicas, reverse phase sorbents and other modifications including chiral types. SuperPure sorbents LABIOSPHER FS SPS are here as a new offer for analytical applications in pore size 100 A, 200 A and 300 A and in particle size 2, 3 and 5 µm. **LABIOSPHER FS SMB** is a very pure spherical silica offered both for analytical and preparative applications. These sorbents have pore size 70 A, 100 A, 150 A, 200 A, 300 A, 500 A, 800 A and 1000 A and particle size range from 3 µm and 30 µm and wide choice of modifications including chiral. Good choice for large preparative columns are spherical **LABIOSPHER FS MB** of granular **LABIOSPHER FS GS**.

ZEOPREP

Series of irregular high quality and high reproducibility silica sorbents with different pore size (4, 6, 9, 11 and 30 nm) and corresponding surfaces are delivered for prep chromatography applications as ZEOPREP 40 (60, 90, 110, 300). It is the best solution both for flash chromatography and for large low pressure column applications.

PERLOSE

Perlose is a semirigid, cellulose based spherical sorbent for size exclusion chromatography and modifications. PERLOSE MT 100 with exclusion limit 100,000 D is delivered in dry state in different particle sze from 25 to 200 um. It can be easily transformed into water slurry and packed. Pressure resistance 80 bar allows the use in high flow rates applications.

LABIOSPHER SUN

Modifications available

	C18	C8	C4	NH2	OH	PH	CN
LABIOSPHER SUN 100	yes	yes	yes	yes	yes	yes	yes

Physical and chemical properties

	Specific surface area [m^2/g]	Pore volume [ml/g]	Average pore diameter {nm]	pH (5 % suspension)	Loss of drying on 150 °C [wt %]
LABIOSPHER SUN 100	180 - 220	0,9 - 1,1	9 - 12	4,0 - 6,5	< 2

Chromatographic properties

	SUN 100	**SUN 100 CN, OH**	**SUN 100 NH**	**SUN 100 C18**	**SUN 100 C8**
Nitrobenzene [k']	0,6 - 1,1	0,6 - 1,1	0,6 - 1,1	n.a.	n.a.
Phenol [k']	n.a.			0,3 – 0,4	n.a.
Toluene [k']	n.a.			3,0 - 3,6	2,0 - 2,6
Dimethylaniline [k']				1,4 - 1,7	0,7 - 1,1

For Labiospher SUN 100 and SUN 100 CN heptane with 0,1 % of isopropanole was used as mobile phase (k' of biphenyle = 0). For Labiospher SUN 100 C18 and Labiospher SUN 100 C8 a mixture of methanole and water (7:3 v/v) was used as mobile phase (k' of thiourea = 0).

Typical efficiency of columns

Particle size [um]	**5**	**7**	**10**	**15**	**20**	**40**
LABIOSPHER SUN 100 [TP/m]	55000	50000	40000	20000	14000	5000
LABIOSPHER SUN 100 C18 [TP/m]	70000	60000	45000	25000	16000	8000

Conditions: column 4,6 x 250 mm, flow rate 0,6 ml/min, measured for nitrobenzene (SUN 100) or toluene (SUN 100 C18).

LABIOSPHER PSI

Modifications available

Modification / **Sorbent**	**C18**	**C8**	**C4**	**SRP**	**C18N**	**NH2**	**OH**	**PH**	**CN**
PSI 100	**yes**	**yes**	**yes**	**yes**	**yes**	**yes**	**yes**	**yes**	**yes**
PSI 200	**yes**	**yes**	**yes**			**yes**	**yes**		**yes**

Physical and chemical properties

Sorbent	Specific surface area [m^2/g]	Pore volume [ml/g]	Average pore diameter {nm]	pH (5 % suspension)	Loss of drying on 150 °C [wt %]
PSI 100	240 - 360	0,6 - 0,8	9 - 14	4,0 - 6,5	< 2
PSI 200	140 - 240	0,7 - 0,9	15 - 21	4,0 - 6,5	< 2

Chromatographic properties

	PSI 100	**PSI 100 CN, OH**	**PSI 100 NH**	**PSI 100 C18**	**PSI 100 C8**	**PSI SRP**
Nitrobenzene [k']	0,6 - 1,1	0,6 - 1,1	0,6 - 1,1	n.a.	n.a.	n.a.
Phenol [k']	n.a.			0,3 – 0,5	n.a.	n.a.
Toluene [k']	n.a.			2,8 - 3,9	n.a.	4,4 - 5,2
	PSI 200	**PSI 200 CN**	**PSI 200 NH**	**PSI 200 C18**	**PSI 200 C8**	

	PSI 100	PSI 100 CN, OH	PSI 100 NH	PSI 100 C18	PSI 100 C8	PSI SRP
Dimethylaniline [k']				1,4 - 1,9	0,7 - 1,1	
Nitrobenzene [k']	0,35 - 0,60	0,3 - 0,6	0,3 - 0,6			
Phenol [k']				0,2 - 0,4	0,1 - 0,3	
Toluene [k']				1,7 - 2,4	0,8 - 1,3	

For Biospher PSI 100 and Biospher PSI 100 CN heptane with 0,1 % of isopropanole was used as mobile phase, k' of biphenyle = 0. For Biospher PSI 100 C18 and Biospher PSI 100 C8 a mixture of methanole and water (7:3 v/v) was used as mobile phase, k' of thiourea = 0.

Typical efficiency of analytical columns (4.6x250 mm)

Particle size [um]	5	7	10	15	20	40
Labiospher PSI 100 [TP/m]	55000	50000	40000	20000	14000	5000
Labiospher PSI 100 C18 [TP/m]	70000	60000	45000	25000	16000	8000
Labiospher PSI 200 [TP/m]	55000	50000	40000	25000	14000	5000
Labiospher PSI 200 C18 [TP/m]	75000	60000	45000	30000	16000	8000

Conditions: column 4,6 x 250 mm, flow rate 0,6 ml/min, measured for nitrobenzene (PSI) or toluene (PSI C18).

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