PHASE NEWS™

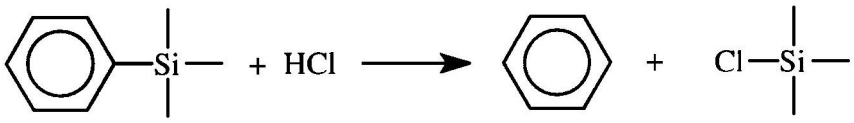
Vol. 1, No. 1

Contents:

Aromatic Phases — A Resurgence

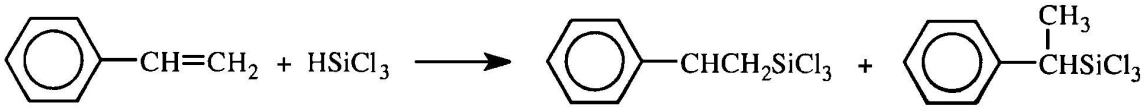
There has been a resurgence of interest in aromatic phases for LC. The high electron density and moderate polarity of aromatic groups provide significant selectivity advantages for resolution or purification of many pharmaceuticals. The historic instability of simple phenyl phases has been overcome by new molecular designs. Other approaches modify selectivity or allow compatibility with long chain alkyl test protocols.

Simple phenyl substituted silanes were utilized to create the earliest aromatic stationary phases. The stability of these materials is poor since the electropositive nature of silicon allows rupture of the silicon aromatic bond.

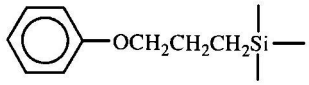
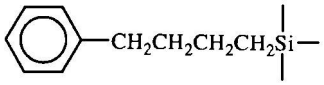
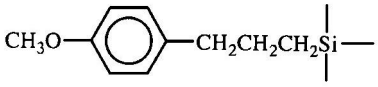
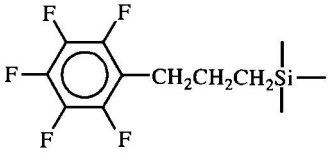
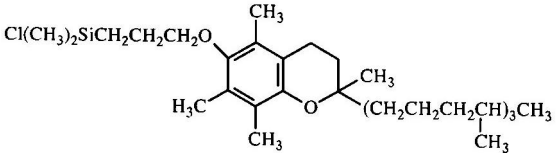


This inherent instability led many investigators to abandon phenyl-containing phases although the obvious desirability of selectivity based on aromatic interaction persisted.

Phases with enhanced hydrolytic stability can be prepared by separating the phenyl groups from silicon. The most economical method for preparing these materials is by the hydrosilation of styrene. These are the phenethyl derivatives which have two principal isomers resulting from α and β addition reactions. The phenethylsilanes are typically used as isomeric mixtures, although in some cases the purified β isomer, 2-phenethyl-, is used.

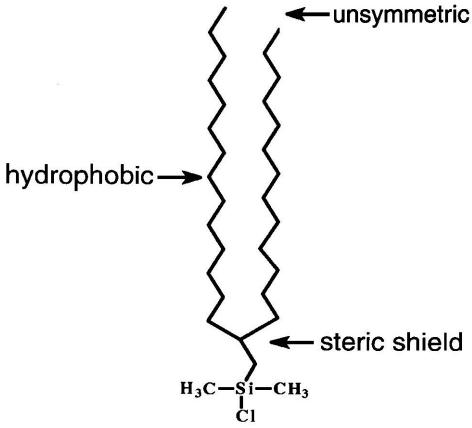


Newer aromatic silanes remove the phenyl group further from silicon and eliminate these problems. Examples are:

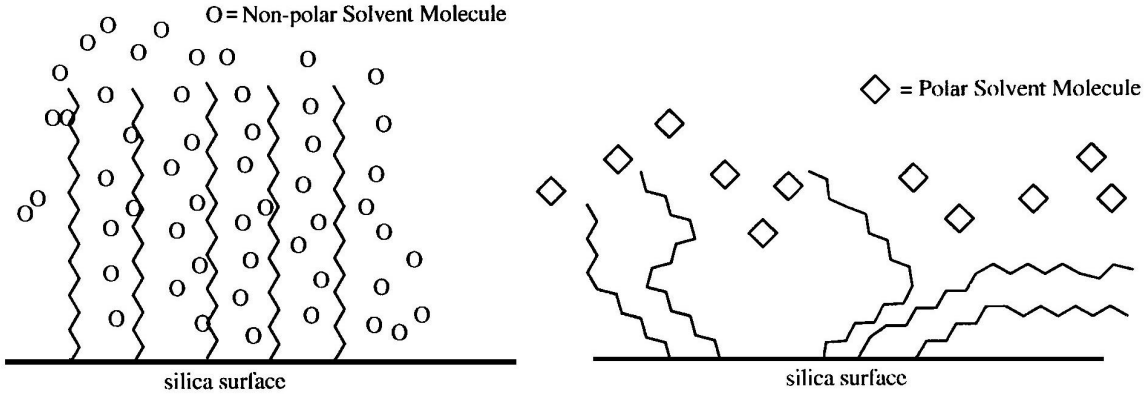
Name	Gelest Product Codes	
	-dimethyl-chlorosilane	-trichloro-silane
3-Phenoxypropyl- 	SIP6723.2 25g/$40.00	SIP6723.3 25g/$34.00
4-Phenylbutyl- 		SIP6724.9 25g/$110.00
p-Methoxyphenylpropyl- 		SIP6492.5 25g/$64.00
Pentafluorophenylpropyl- 	SIP6716.2 5g/$72.00	SIP6716.4 2.5g/$64.00
Tocopheroloxypropyl 	SIT8010.0 10g/$120.00	

Heptacocylsilanes C27 Branched Silanes

Heptacocyldimethylchlorosilane is the first example of a new approach to generate more versatile hydrophobic phases for high performance and preparative liquid chromatography. The features of the silane are depicted below.



The most widely used LC phase is octadecyl which is derived from a straight chain C18 functional silane.[1] The utility of the phase has always been limited in polar media. The increasing number of acidic analytes and the drive to aqueous media have generated a high level of interest in finding a way to create hydrophobic phases without the limitations of C18. Polar solvents (i.e. water) are thought to induce a phase (bed) collapse, a process which may be caused by self-association or crystallization of the long regular C18 chains.[2]

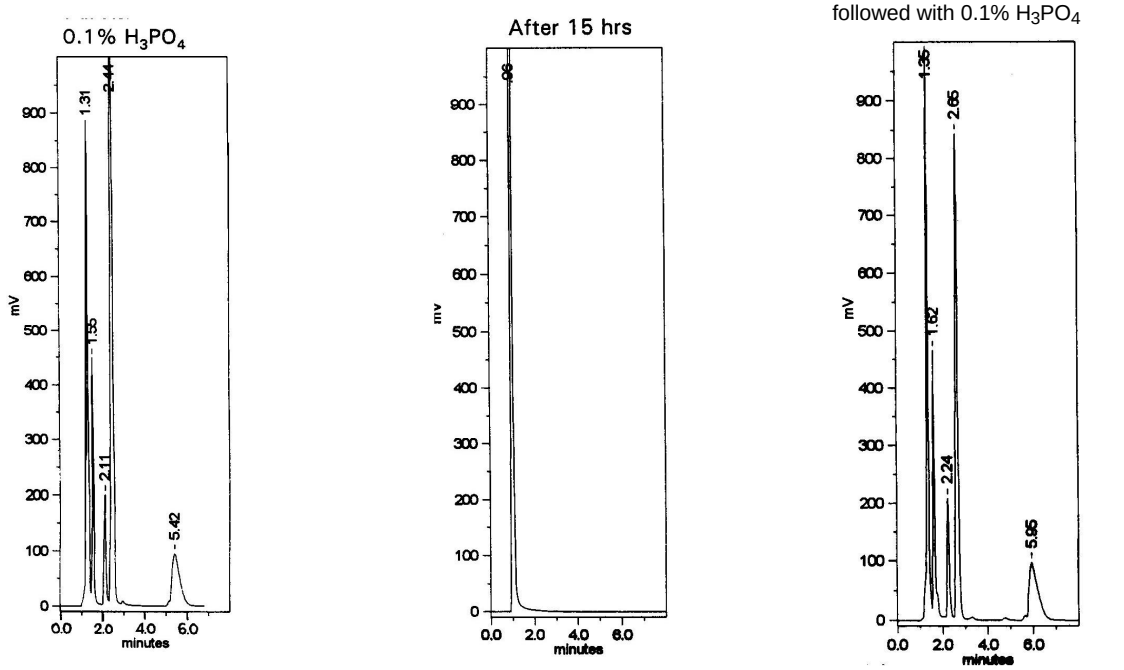


A comparative analysis of oxalic acid, tartaric acid, malic acid, ascorbic acid and succinic acid in a phosphate buffered 100% aqueous system is shown below. The octadecyl phase undergoes phase collapse, while the heptacocyl phase maintains performance.

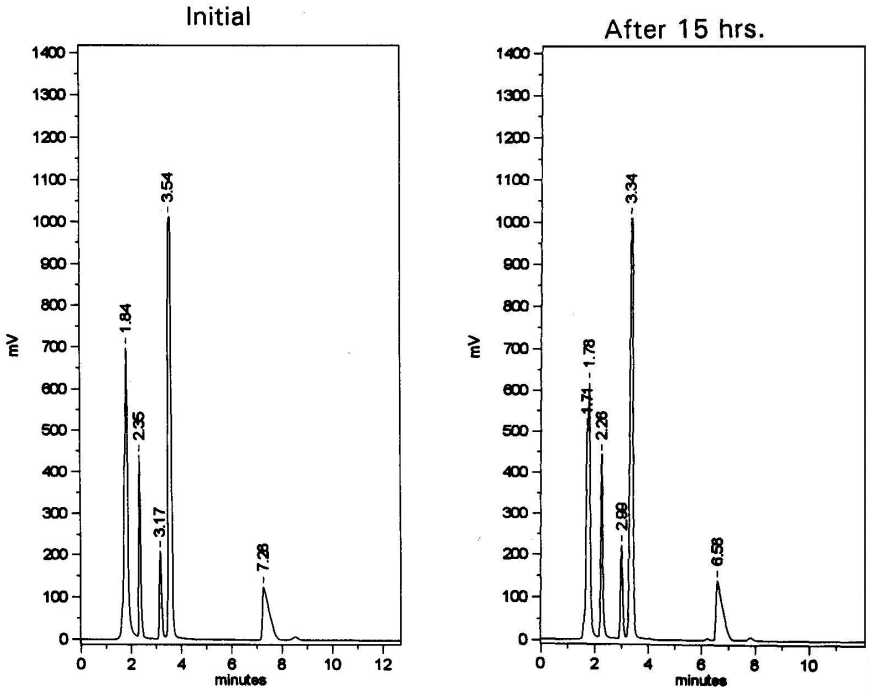
SIC2266.0
13-(CHLORODIMETHYLSILYLMETHYL)HEPTACOSANE, 95%
10g/$110.00

1. W. Caldwell et al., HPLC '98, St. Louis, Mo.
2. L. Duff, American Laboratory, Feb. 1998.

Octadecyl C_{18} Phase



Heptacocyl C27 Phase

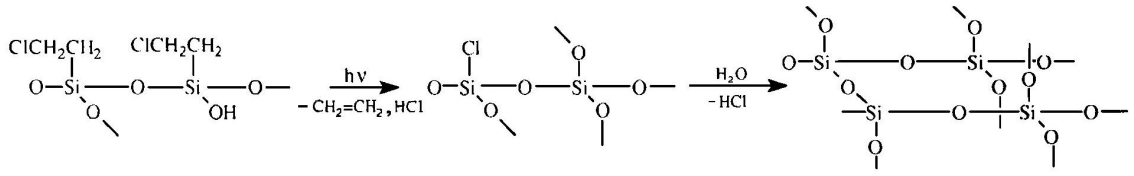


New Method Provides Uniform SiO2

The ability to provide silicon dioxide as a conformal coating or in discrete patterns has potential applications in separation science. The applications range from coating of capillaries to producing patterned arrays. The silicon dioxide surfaces generated can then be functionalized by established techniques.

At relatively low temperature (300°C) a deep UV exposure provides uniform high density by conversion of a silsesquioxane precursor. Films can be generated by dip, spin-on or direct-write methods including micro-contact printing. The technology allows the deposition of architectures which can be derivatized with organosilanes for diagnostic applications.

The technology exploits the rearrangement reaction of 2-chloroethylsilsesquioxane. At temperatures above 300°C or under the influence of IV irradiation, the chlorine migrates to the silicon atom, forming ethylene as a byproduct, and then is displaced by SiOH or reacts with water to form the final siloxane bond, generating HCl.[3,4] The technology allows deposition on substrates ranging from plastic to silicon.[5] Patterns can be created by writing or printing deposition or, in the case of UV exposure, washing off non-exposed areas with a variety of solvents.



3. B. Arkles, D. Berry, L. Figge, US Pat. 5,853,808, 1988
4. B. Arkles, D. Berry, L. Figge, J. Sol-Gel Sci. & Technol., *8*, 465, 1997
5. R. Composto *et al.*, Thin Solid Films, *345*, 244, 1999
6. J. Sharma, D. Berry, R. Composto, H. Dai, J. Mater. Res., *14*, 990, 1999

Seramic™ SI Silicon Dioxide Precursor

Description

Searmic™ SI is a 2-chloroethylsilsesquioxane solution in methoxypropanol.

Film Properties

Color	clear
Dielectric Constant	3.2–3.6

Solution Properties

Form	solution
Solids	14-16%
Density	0.96g/cc
Viscosity	3-5 cSt.
Flashpoint	35°C

Application Methods

Thermal — Gelest Seramic™ SI is applied as a coating by dipping or spin-on. After solvent evaporation, the system cures in 30-60 minutes at 300°C. As supplied typical film deposition is 1500-2000Å by spin-on application. Thinner films may be prepared by diluting with methoxypropanol or diglyme. The cure process liberates small amounts of ethylene and hydrogen chloride.

UV — Gelest Seramic™ SI is converted to silicon dioxide on exposure to deep UV (<240nm). Exposed areas are converted, while unexposed areas may be removed by a solvent wash.

Seramic™ SI

PP1-SESI 100g/$84.00

PRODUCT SHORTS

Silicone Gum in Solvent

High molecular weight purified and filtered polydimethylsiloxane gum is available predispersed in volatile cyclic siloxanes. Those who have worked with silicone elastomer gums will appreciate the “good behavior” of this predispersed material. The solvent is D5, a volatile cyclic siloxane.

DMS-T72B15
Polydimethylsiloxane 15-20x10^6 cSt. 15% in D5 100g/$39.00

Octaphenylcyclotetrasiloxane in Higb Purity

Commercial polymer grade octaphenylcyclotetrasiloxane normally contains small amounts of other diphenyl cyclics and linears. Although these materials present few problems in the

production of low molecular weight silicone fluids, they frequently manifest themselves as haze or color in high molecular weight polymers used as stationary phases. A high purity grade of this reagent which eliminates these problems is now available.

SIO6705.1
Octaphenylcyelotetrasiloxane, 98% 500g/$340.00

Vector Phase Reagents for Deactivation of Silica Allowed for Commercial Use

Phenylsilazane reagents are used for the deactivation of glass columns in a method called "persilylation." Similar applications include endcapping of bonded phases. The reagents are used by multiple injections into capillary columns or by admixture with bonded phases and heating to ~120°C. Until recently the use of these materials in commercial applications was not allowed. Gelest has applied to the EPA and has been allowed LVE status for the following reagents, thus satisfying TSCA inventory requirements.

SID4586.0
Diphenyltetramethyldisilazane 25g/$108.00

SIT7553.0
Tetraphenyldimethylsilazane 25g/$96.00

7. K. Grob *et al.*, High Resol. Chrom. & Col. Chrom., *3*, 197, 1980

Search www.Gelest.com the Interactive Web Site

Gelest, Inc. offers over 500 chemicals and polymers directed toward surface modification. Our interactive web-site allows you to analyze listings of products by application or by functionality. Our advanced search function allows you to draw a chemical structure for a product search. Every Gelest material has technical data and reference notations on the search results. You can contact Gelest through e-mail or by telephone for inquiries regarding custom synthesis.

Phase News™ is published by Gelest Inc. Gelest is a leading supplier of materials for the preparation of bonded and stationary phases. With over 500 chemicals and polymers directed toward surface modification, Gelest is able to participate at all levels of research, development and commercialization.

Gelest Inc.
612 William Leight Drive
Tullytown, PA 19007
Phone: (215) 547-1015
FAX: (215) 547-2484
Web: www.gelest.com