

SUPPORTS

Uniport®-HP High Performance Silanized Support

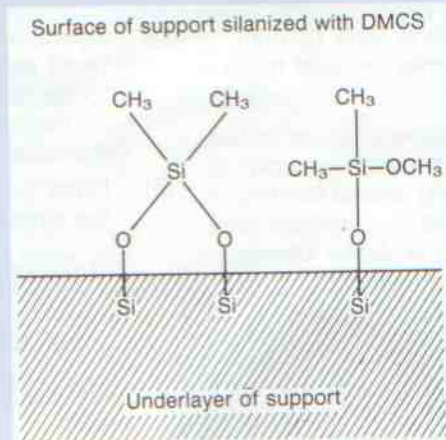
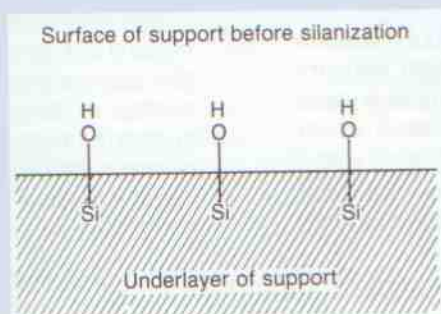
Now that high sensitivity detectors for gas chromatography like FID, FPD, ECD and PID have become so commonplace, high precision in trace analysis is coming to be required. At the same time, analytical procedures are improving, and the range of substances which can be analyzed is widening to include high boiling compounds, highly polar compounds and compounds that decompose easily. When these compounds are analyzed by gas chromatography, they have to be eluted quickly, and it is therefore necessary to use a packing with a low liquid phase load of about 1-5%.

Reducing the amount of liquid phase, however, is almost certain to increase adsorption on the support and increase its tendency to decompose the sample, mainly due to the presence of metal oxides in the support and silanol groups on its surface. These two factors can be nullified by acid treatment to remove the metal oxides, followed by silanization.

Silanization is a process whereby the silanol groups on the surface of the support are substituted by inactive alkylsilyl groups. Various methods of doing this exist, but pre-treatment and post-treatment are also important. In one method, for example, toluene solvent is used; in another, the vapor of

the silanizing agent is used. There are many pre-treatment and post-treatment problems, e.g. removal of fines¹⁾, acid washing²⁾, and removing the hydrogen chloride which is a by-product of silanization, and if these conditions are not optimized, a good silanized support will not be obtained. GL Sciences has overcome these problems to bring you the best silanized support possible — Uniport HP.

Uniport-HP is a support which has been deactivated to the ultimate limit. Use it for analyses requiring a high no. of theoretical plates, symmetrical peaks, and freedom from sample adsorption or decomposition.

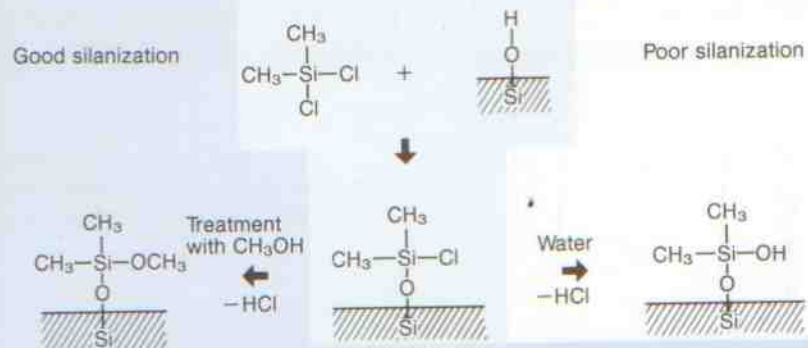


The Silanization Process

Acid washing is an essential part of the silanization process, and was so far carried out by a variety of techniques ranging from immersion of the support in dilute or concentrated hydrochloric acid to heating under reflux with acid (References 2, 3, and 4). Since Uniport

HP has been thoroughly acid washed, it has excellent properties as a silanized material. Silanization is usually carried out by refluxing or leaving to stand with a 5% solution of DMCS in toluene or benzene (References 1, 4); however, there is no actual basis for using a 5% concentration, and some references (5) recommend 0.1%.

Water is one factor that interferes with satisfactory silanization, and it is therefore excluded completely from the whole system when Uniport HP is treated. If HMDS (References 2, 4, 6, 7) or TMCS (References 2, 7) is used, water no longer has an effect, but these reagents suffer from the disadvantage that their reactivity is very low.



- 1) Supina, W.R., R.S. Henly and R.F. Kruppa, J. Am. Oil Chemists' Soc., 43 202A (1966)
- 2) Ottenstein, D.M., J. Gas Chromatog., 6 129 (1968)
- 3) Horning, E.C., EA. Moscatelli and C.C. Sweeley, Chem. & Ind., 751 (1959)
- 4) Smith, E.D., J.M. Oathout and T. Cook, J. Chromatog. Sci., 8 291 (1970)
- 5) Holmes, W.L. and E. Stack, Biochim Biophys. Acta, 56 163 (1962)
- 6) Perrett, R.H. and J.H. Purnell, J. Chromatog., 7 455 (1962)
- 7) Purnell, J.H., Gas Chromatography, P237 John Wiley & sons, (1962)

Examination of Intertness of Silanized Supports

Although conventional silanized supports have certain disadvantages, there has so far been no method of fairly evaluating their performance.

GL Sciences has however devised some methods which are easily carried out using a gas chromatograph and FID.

The butyl acetate test consists of subjecting the support to a mixture of methane and butyl acetate. Good supports give no separation, and this provides an indication of the degree of silanization.

The endrin test determines how much of the hydrogen chloride produced in the silanization has remained. Endrin decomposes completely in the presence of only several tens of ppm of HCl, and so this may be said to be a very suitable test.

N.B. GL Sciences gave a lecture on test methods for silanized supports at the 37th Spring Annual Meeting of the Japan Chemical Institute (See Abstracts of the Japan Chemical Institute 1, 3106, (1978).

Butyl Acetate Test

(1. Methane, 2. Butyl Acetate)

Endrin Test

(1. Dieldrin 2. Endrin
3. 4. Endrin Decomposition product)

Analysis of methyl esters of bile acids

1. Methyl lithocolate
2. Methyl deoxy cholate
3. Methyl chenodeoxy cholate
4. Methyl cholate

Butyl Acetate Test

- (1) A 3 mm x 2 m glass column (on column method) is packed with a support which has not been coated with a liquid phase, and is attached to the gas chromatograph. Carrier gas (use nitrogen), air and hydrogen are then adjusted to suitable flowrates, and the FID switched on.
- (2) Column and injection temperatures are set to 150°C, and 5 μL of methane are injected. Adjust flowrate such that methane is eluted in 2 min.
- (3) Inject 20 μL of the head space gas in a bottle of butyl acetate, then compare the height of the peak with that of 5 μL of methane. With a poorly silanized support butyl acetate sticks to the column, its peak is small, and it is eluted later due to tailing.
- (4) Inject the amount of butyl acetate head space gas which appears to be necessary to give a peak of the same height as 5 μL of methane.
- (5) Inject 5 μL methane, together with the amount of butyl acetate head

space gas which appears to be necessary to give a peak of the same height as the methane.

- (6) With good supports, the methane and butyl acetate are not separated. With poor supports, they are separated and exhibit tailing.

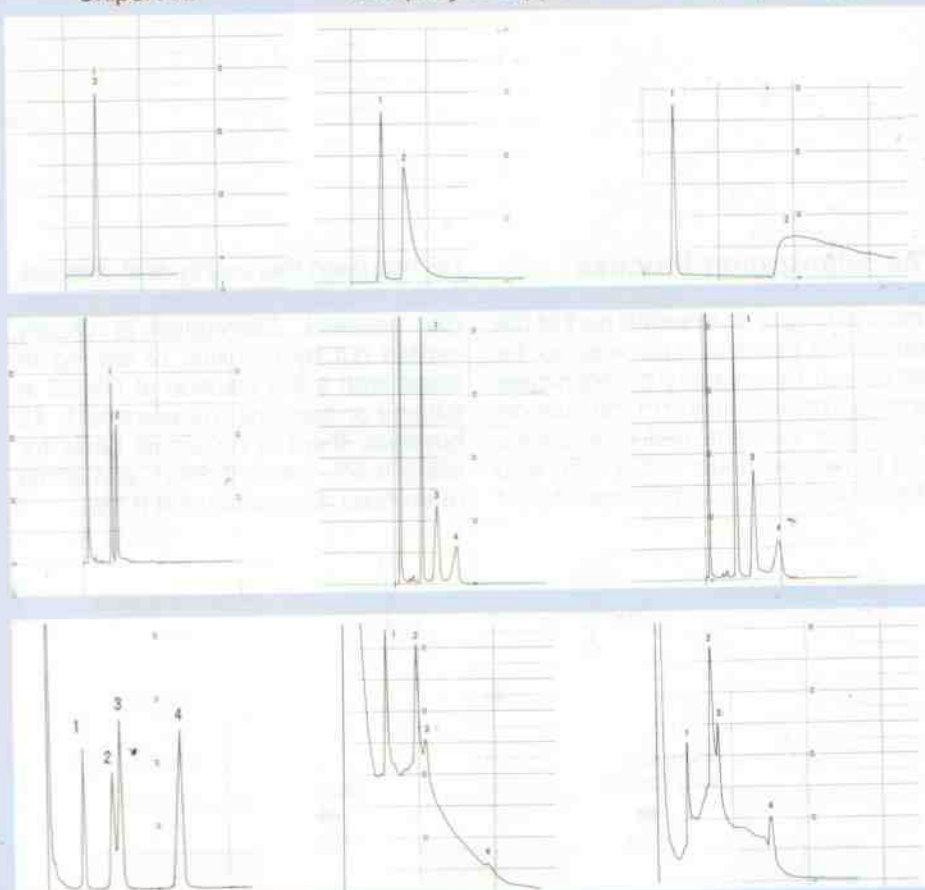
Endrin Test

- (1) The support is loaded with 2 wt% OV-17, packed into a 3 mm x 2 m glass column, and then conditioned at 320°C for 1-2 hours.
- (2) Column and injection temperatures are set to 270°C, and the FID is switched on. (Endrin analyses are carried out at 240°C or below actually).
- (3) Inject 0.4 μL of a mixed solution of Dieldrin and Endrin (25 mg/mL of each in toluene), and adjust flowrate of carrier gas such that Dieldrin is eluted in 2 min.
- (4) Inject 0.4 μL of test solution as in (3), and measure ratio of peak heights of Endrin/Dieldrin. With supports containing hydrogen chloride, the peak due to Endrin is either very small, or decomposes completely to give 2 peaks.

Uniport HP

Company A support

Company B support



SUPPORTS

Uniport®

Uniport supports were developed by the Research Division of GL Sciences. Unlike some supports of foreign manufacturer or other Japanese-made supports, only raw materials of the highest purity are used. They therefore have low reactivity to samples with little tailing, and permit stationary phase loads to be considerably reduced.

These all-round supports are ideal for samples liable to decompose, high boiling samples, and samples which easily cause tailing.

The Uniport Series of supports have different characteristics to enable you to choose the right one for your needs.

Uniport B

A diatomaceous earth prepared for gas chromatography. Surface metals have been carefully eliminated by a special process so as to give a support without any catalytic activity that is simple to use.

Uniport HP

Uniport HP which has been subjected to a special silanization. This is the most inert silanized support on the market today.

As hydrogen chloride generated in the silanization process has been completely eliminated, this support is ideal for ECD application.

Uniport HPS

Uniport HPS specially treated to make it suitable for weakly basic compounds and pesticides.

Uniport R

Ideal for the analysis of highly polar substances such as lower alcohols and amines. As this support deteriorates when kept at high temperature for long periods, it should not be used above 250°C.

Uniport C

Firebrick type support made from specially treated diatomaceous earth.

Uniport CS

Uniport C which has been especially silanized.

Uniport A

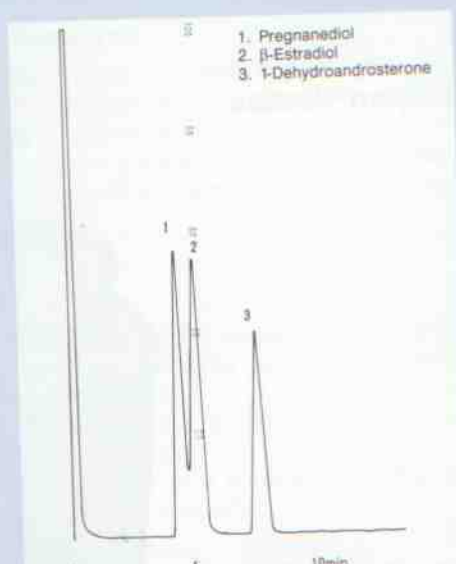
Diatomaceous support with weakly basic surface for neutral or weakly basic samples.

Uniport AB

Diatomaceous support with strongly basic surface for strongly basic samples such as amines.

Uniport S

Diatomaceous support with acidic surface for samples showing acidity or weak acidity such as cresol.



Silicone OV-17 2% Uniport HP 80/100
Glass 3mm I.D. x 2m 280°C N₂ 50mL/min. FID

Physical properties of Uniport

	Uniport	
	B	C
Density (g/mL)	2.36	2.15
Free Fall Density (g/mL)	0.18	0.38
Packing Density (g/mL)	0.20	0.47
Pore Size (μ m)	>5	<5
Specific Surface Area (m ² /g)	1	4
Coating Range (%)	1-25	5-30
Color	White	Pink

Chemical composition of Uniport

	Uniport	
	B	C
SiO ₂	90.0	89.2
Al ₂ O ₃	3.6	4.1
Fe ₂ O ₃	1.4	1.5
TiO ₃	0.2	0.2
CaO	0.4	0.5
MgO	0.5	0.5
Na ₂ O+K ₂ O	3.2	1.0

SUPPORTS

Uniport® Series



Supports	Mesh	100mL		500mL		1L	
		Cat. No.	Price	Cat. No.	Price	Cat. No.	Price
Uniport HP	60/80	1001-31306		1001-31356		1001-31376	
	80/100	1001-31308		1001-31358		1001-31378	
	100/120	1001-31310		1001-31360		1001-31380	
Uniport HPS	60/80	1001-31406		1001-31456		1001-31476	
	80/100	1001-31408		1001-31458		1001-31478	
	100/120	1001-31410		1001-31460		1001-31480	
Uniport R	60/80	1001-31006		1001-31056		1001-31076	
	80/100	1001-31008		1001-31058		1001-31078	
	100/120	1001-31010		1001-31060		1001-31080	
Uniport B	30/60	1001-31603		1001-31653		1001-31673	
	60/80	1001-31606		1001-31656		1001-31676	
	80/100	1001-31608		1001-31658		1001-31678	
	100/120	1001-31610		1001-31660		1001-31680	
Uniport C	30/60	1001-31103		1001-31153		1001-31173	
	60/80	1001-31106		1001-31156		1001-31176	
	80/100	1001-31108		1001-31158		1001-31178	
	100/120	1001-31110		1001-31160		1001-31180	
Uniport CS	30/60	1001-31203		1001-31253		1001-31273	
	60/80	1001-31206		1001-31256		1001-31276	
	80/100	1001-31208		1001-31258		1001-31278	
	100/120	1001-31210		1001-31260		1001-31280	
Uniport A	60/80	1001-31706		1001-31756		1001-31776	
	80/100	1001-31708		1001-31758		1001-31778	
Uniport AB	60/80	1001-31806		1001-31856		1001-31876	
	80/100	1001-31808		1001-31858		1001-31878	
Uniport S	60/80	1001-31906		1001-31956		1001-31976	
	80/100	1001-31908		1001-31958		1001-31978	

Flusin®

Supports

Flusin T
Flusin T6

Flusin F
Flusin GL
Flusin GH
Flusin CF

Flusin CF
Flusin P

Support

Flusin T

Flusin T

Flusin F

Flusin

Flusin

Flusin

Flusin

Flusin

Flusin

SUPPORTS

Flusin® Series

Supports	Type	Packing Density (g/mL)	Max. Temp. (°C)
Flusin T	① Screened Teflon®	0.52	150
Flusin T6	② Screened Teflon®	0.58	200
Flusin F	④ Crystal	1.37	400
Flusin GU	⑤ Glass Beads	1.50	400
Flusin GH	⑥ Silanized GU	1.50	400
Flusin CF	⑦ Diatomaceous coated with fluoro-resin	0.27	250
Flusin CFS	⑧ Silanized CF	0.30	250
Flusin P	⑨ Terephthalic Acid System	0.50	185

Features

① Mechanical strength is comparatively tough so that its handling is easy.
 ② Despite of high operating temperature, its mechanical strength is weak. Column loading/unloading need a practice.

④ ⑤ ⑥ Smaller in surface area. Suitable for samples which are easily decomposed.

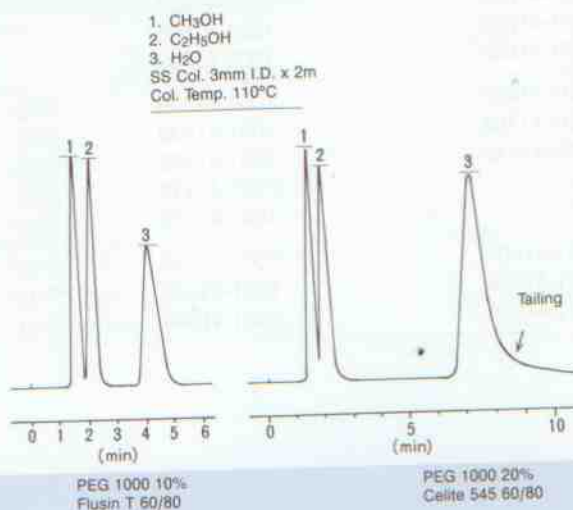
⑦ ⑧ Larger in surface area. Used as an inert support.

⑨ An inert acid support. Suitable for analysis of free acid.

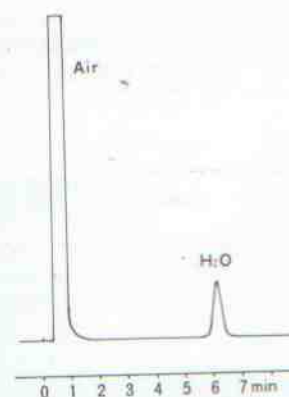
Supports	Mesh	Cat. No.	Price (100mL)	Cat. No.	Price (500mL)	Max. Liquid Phase Loading (%)
Flusin T	30/60	1001-32103				20%
	60/80	1001-32106				
	80/100	1001-32108				
Flusin T6	30/60	1001-32203				

Flusin F	30/60	1001-32403		1001-32453		1%
	60/80	1001-32406		1001-32456		
	80/100	1001-32408		1001-32458		
	100/150	1001-32410		1001-32460		
Flusin GU	30/60	1001-32503		1001-32553		1%
	60/80	1001-32506		1001-32556		
	80/100	1001-32508		1001-32558		
Flusin GH	30/60	1001-32603		1001-32653		1%
	60/80	1001-32606		1001-32656		
	80/100	1001-32608		1001-32658		
Flusin CF	60/80	1001-32706		1001-32756		20%
	80/100	1001-32708		1001-32758		
Flusin CFS	60/80	1001-32806		1001-32856		20%
	80/100	1001-32808		1001-32858		
Flusin P	30/60	1001-32303		1001-32353		10%
	60/80	1001-32306		1001-32356		

Flusin T Celite545



H₂O in Air



PEG 1500 20% Flusin T 60/80
 SS 3mm I.D. x 2m 100°C He 50mL/min

SUPPORTS

Celite® 545

Celite is the trade name of the Manville Co. for a diatomaceous earth.

In Celite 545, a small amount of flux (Na_2CO_3) has been added to this diatomaceous earth and the product calci-

nated. It is then improved by various treatments so as to give a support of wide, general applicability.

This is a white support with a packing density of 0.28 g/mL and surface area 0.56 m²/mL.

Chemical Composition of Celite (%)

SiO ₂	89-90.5
Al ₂ O ₃	4-5.5
Fe ₂ O ₃	1.4-1.6
CaO	0.4-0.7
MgO	0.5-0.6

Supports	Mesh	Cat. No.	Price (100 mL)	Cat. No.	Price (500 mL)	Cat. No.	Price (1 L)
Celite 545 (heat treated)	30/60	1001-34103		1001-34153		1001-34173	
	60/80	1001-34106		1001-34156		1001-34176	
	80/100	1001-34108		1001-34158		1001-34178	
	100/120	1001-34110		1001-34160		1001-34180	
Celite 545 SK (acid washed)	30/60	1001-34203		1001-34253		1001-34273	
	60/80	1001-34206		1001-34256		1001-34276	
	80/100	1001-34208		1001-34258		1001-34278	
	100/120	1001-34210		1001-34260		1001-34280	
Celite 545 SK DMCS (DMCS-silanized)	30/60	1001-34303		1001-34353		1001-34373	
	60/80	1001-34306		1001-34356		1001-34376	
	80/100	1001-34308		1001-34358		1001-34378	
	100/120	1001-34310		1001-34360		1001-34380	
Celite 545 SK HMDS (HMDS-silanized)	30/60	1001-34403		1001-34453		1001-34473	
	60/80	1001-34406		1001-34456		1001-34476	
	80/100	1001-34408		1001-34458		1001-34478	
	100/120	1001-34410		1001-34460		1001-34480	

Gas Chrom®

Support	Type	Mesh	Cat. No.	Price (100 mL)	Cat. No.	Price (1/2 lb)
Gas Chrom A	Acid washed	60/80	1001-41106		1001-41136	
		80/100	1001-41108		1001-41138	
		100/120	1001-41110		1001-41140	
Gas Chrom P	Acid washed, Base washed	60/80	1001-41206		1001-41236	
		80/100	1001-41208		1001-41238	
		100/120	1001-41210		1001-41240	
Gas Chrom Z	Acid washed, DMCS treated	60/80	1001-41306		1001-41336	
		80/100	1001-41308		1001-41338	
		100/120	1001-41310		1001-41340	
Gas Chrom RZ	Acid washed, DMCS treated	45/60	1001-41604		1001-41634	
		60/80	1001-41606		1001-41636	
		80/100	1001-41608		1001-41638	
Gas Chrom Q	Acid washed, Base washed Silane treated	60/80	1001-41406		1001-41436	/50g
		80/100	1001-41408		1001-41438	/50g
		100/120	1001-41410		1001-41440	/50g
		120/140	—	—	1001-41412	/50g
		230/270	—	—	1001-41473	/50g
Gas Chrom QII	Acid washed, DMCS treated	60/80	1001-41506		1001-41536	/50g
		80/100	1001-41508		1001-41538	/50g
		100/120	1001-41510		1001-41540	/50g