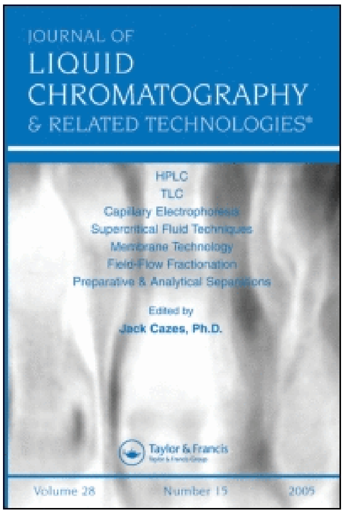

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EFFICIENT SOLID PHASE EXTRACTION PROCEDURES FOR ORGANO SILOXANES AND SILANOLS FROM AQUEOUS AND BIOLOGICAL MATRICES

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ABSTRACT

Efficient solid phase extraction (SPE) procedures were established to extract a variety of organosiloxanes and organosilanols at low concentrations from aqueous and biological matrices. The organosilicon materials used in SPE experiments were all ^{14}C-labeled. Use of ^{14}C- labeled materials made it very convenient to determine the SPE process efficiency.

For non-polar siloxanes of lower molecular weight, viz., ^{14}C-octa-methyl-cyclotetrasiloxane (D_4), ^{14}C-decamethylcyclopentasiloxane (D_5), and ^{14}C-hexamethyldisiloxane (MM), the recoveries from aqueous solutions, using C18 SPE cartridges, were 91 (± 2), 98.6 (± 0.5), and 96.2 (± 2), % respectively. For polymeric siloxane fluids of 350 and 1000 cSt viscosities with number average molecular weights, Mn of ~9800 and 17,000 respectively, C4 sorbent was preferred. The recoveries for these polymeric siloxanes were 89.3 (± 1.7), and 96 (± 0), % respectively.

For polar silanols, styryl-divinylbenzene based polymeric-sorbent ENV+® worked exceptionally well. Using ENV+ sorbent, samples of dimethylsilanediol $Me_2Si(OH)_2$, methylsilanetriol $MeSi(OH)_3$, were recovered from aqueous medium with excellent efficiencies (99 and 94 %, respectively). The ENV+ cartridge was also applied to recover very efficiently the metabolites (i.e. hydroxy and hydroxymethyl functional siloxanes) of D_4, D_5, and MM present in rat urine. The recoveries were >90% in all cases. Recoveries of metabolites of MM and D_5 from plasma were ~80 and 66% respectively.

The concentrations of the analytes ranged from parts per billion to parts per million. For successful retention of the analytes to the sorbent, it was found to be necessary to place solid NaCl to the top of the SPE cartridge bed, or treat the aqueous matrices with NaCl prior to sample load. HPLC analyses of samples performed following the SPE process showed that the organosilicon materials, including the very reactive silanols, remained unchanged.

INTRODUCTION

Cyclic and linear polydimethylsiloxane (PDMS) comprising of $-Me_2Si-O-$ units are produced in hundreds of millions of pounds a year.[1] They are used in a wide variety of consumer and industrial applications. Among them, the volatile cyclic siloxanes, octamethylcyclotetrasiloxane (D_4) (Abbreviations used are: MM, hexamethyldisiloxane; D_4 = octamethylcyclotetrasiloxane; D_5= deca-methylcyclopentasiloxane; The abbreviations are based on the General Electric's siloxane notation [D.T. Hurd, *J. Am. Chem. Soc.*, **68**, 364, 1946] which is as follows: $M=Me_3SiO_{1/2}$; $D= -Me_2SiO_{2/2}$) and decamethyl-cyclopentasiloxane (D_5) have found use as ingredients in a wide range of personal care products and carrier fluid applications.[2] The applications of siloxanes stem from a variety of favorable physical characteristics such as adequate evaporation rate, low surface tension, lack of odor, a high degree of compatibility with many formulation ingredients and solubility characteristics. Typical examples of applications include moisturizing creams, lotions, bath oils, colognes, shaving products, and perfumes. The cyclic materials are also used as lubricants and penetrating oils. The majority of the cyclic siloxanes is used as site limited intermediates in the manufacturing of polydimethylsiloxane (PDMS) polymers.

Among the linear siloxanes, the rigorously purified linear siloxane, MM (Dow Corning® OS-10, purity > 99.9%) is one of the many ozone-safe volatile methylsiloxanes (VMS) that is exempt from federal VOC (volatile organic content) regulations and, hence, being accepted as an alternative for other organic solvents. Another important industrial use of MM is as a chain terminating agent in siloxane polymerizations. The utility of non-volatile, higher molecular weight, polymeric PDMS is also quite impressive. The latter finds extensive applications in textiles, automotive, construction, and electrical industries.

Due to the widespread use, polydimethylsiloxanes have been subject to numerous toxicological and environmental fate and effect studies.[3-7] In soil, PDMS degrades primarily to the water soluble dimethylsilanediol. In a biological system (e.g. laboratory animals), however, a demethylated silanol, viz, methylsilanetriol is also formed in addition as a major urinary metabolite.[8] These studies invariably require, as part of sample preparation for subsequent analysis, extraction and concentration of parent siloxanes or their degradation products from matrices containing water.

Among a variety of sample preparation techniques to concentrate analytes of interest from aqueous matrices into an organic medium, solid-phase extraction is gaining importance as a viable technique. This technique has been known for about 20 years. It has been the subject of numerous articles[9-11] and book reviews.[12-13] SPE is increasingly being used as an alternative to liquid-liquid extraction. Earlier,[14] we have developed a liquid-liquid extraction methodology using tetrahydrofuran (THF) as the extractant to extract cyclic siloxanes, such as octamethylcyclotetrasiloxane (D_4), and their biotransformation products (metabolites) from aqueous and a variety of biological matrices. The THF extract made it convenient to carry out subsequent analysis for structural elucidation. However, SPE procedure was evaluated for several potential advantages that the technique can offer. The present paper describes in detail the successful development of an SPE procedure to recover a variety of siloxanes from aqueous and biological matrices.

EXPERIMENTAL

Instrumentation/Reagents

Radioactivity measurements were made using a Liquid Scintillation Counter (Tri-Carb 2500 TR Liquid Scintillation analyzer from Packard Instruments). The oxidation of all the residual samples was processed using a Biological Oxidizer (Model, OX500, R. J.Harvey Instrument Corporation). HPLC analyses were performed with a Hewlett Packard 1050 Liquid Chromatograph equipped with an HP Autosampler (model #79855A), and a Radiomatic detector (model #515 TR) from Packard Instrument Co. The detector was installed with a 500 µL Flow Liquid Cell. HPLC conditions in the water/acetonitrile mobile phase were as follows: 100% water, 0-20min; 100% water to 100% acetonitrile, 20-40 min; 100% acetonitrile, 40-50 min; 100% acetonitrile to 100% water, 50-60 min. A C18 Ultima column (4.6 x 250 mm and 5 micron from Alltech) was used as the stationary phase; Ultima-Flo M liquid scintillation cocktail was used in the flow cell. The ratio of column effluent to scintillation cocktail was 1:3.

^{14}C-labeled siloxanes, ^{14}C-D_4, ^{14}C-D_5, ^{14}C-MM, and ^{14}C- $Me_2Si(OH)_2$, ^{14}C-350 cSt PDMS and ^{14}C-1000 cSt PDMS were synthesized following the published procedure.[15] The labeling was at random. The specific activities of the labeled materials are as follows: ^{14}C-D_4, 33.2 mCi/mmol; ^{14}C-D_5, 30.6 mCi/mmol; ^{14}C-MM, 24.4 mCi/mmol; ^{14}C-$Me_2Si(OH)_2$, 2.5 µCi/mL; ^{14}C-350 cSt PDMS, 0.52 mCi/g; ^{14}C-1000 cSt PDMS, 0.302 mCi/g. ^{14}C-$MeSi(OH)_3$, 7µCi/mL. ^{14}C-$MeSi(OH)_3$ was received as a gift from General Electric Corporation. [Specific activity = 2.4 mCi/mmol).

Reagent grade tetrahydrofuran, hexane, methanol, acetonitrile, were purchased from Aldrich and were used without further purification. Deionized water was generated using Ultrapure Water Systems (Millipore). Liquid scintillation cocktails, Hionic-Fluor, Ultima-Gold were obtained from Packard Instrument Co.

C18 and C4 SPE cartridges were purchased from Diazem Corporation, Midland, MI. ENV+ cartridge was purchased from Jones Chromatography, Lakewood, Colorado. Amberlite® XAD-2 and XAD-4 (non-ionic polystyrene adsorbents), as well as, Amberlite® XAD-7 (non-ionic polyacrylate adsorbent) were all purchased from Aldrich. SPE procedures were carried out either using RapidTrace™ Automated SPE Workstation purchased from Zymark® Corporation (Hopkinton, MA), or the PrepTorr™ vacuum manifold purchased from Fisher Scientific. With RapidTrace™ Automated SPE Workstation, materials were forced through the cartridge by application of pressure. With PrepTorr™ vacuum manifold, on the other hand, the materials were passed through by application of vacuum. The PrepTorr™ was connected to the vacuum pump, Model MB 41 purchased from Parker Hannifin Corporation (Metal Bellows Division).

Preparation of Standards in Aqueous Solution

All the ^{14}C-labeled siloxanes of certain specific activities were sufficiently diluted with milli-Q water to obtain aqueous solutions of radioactivity of approximately 3000 dpm/mL suitable for SPE analysis. Since the non-polar siloxanes (D_4, D_5, MM, and 350 and 1000 cSt fluids) have very poor water solubilities,[16] addition of a 10 ppm surfactant (Tween 20 or Tween 80) solutions were necessary to obtain a homogeneous solution. It is important that the surfactant amount is kept at minimum since excess surfactant caused the analytes to elute during the sample loading step. For illustration, the preparative aspect on the aqueous solution of ^{14}C-D_4 solution is given. Similar procedure was used to prepare other siloxanes except that the polar silanols [$Me_2Si(OH)_2$ and $MeSi(OH)_3$] do not require addition of the surfactant. ^{14}C-D_4 (39 µL ; specific activity 33.2 mCi/mmol) was dissolved in 500 µL of absolute ethanol (solution 1). Solution 1 (100 µL) was dissolved in 5 mL of methanol (solution 2).

Table 1

Average Radioactivity and Concentrations of Samples Used for SPE

Sample	Avg. Radioactivity dpm/mL	Aqueous Concentration
^{14}C-D$_4$	2633	11 ppb
^{14}C-D$_5$	4435	24 ppb
^{14}C-MM	2319	7 ppb
^{14}C-Me$_2$Si(OH)$_2$	3142	57 ppb
^{14}C-MeS$_i$(OH)$_3$	2446	102 ppb
^{14}C-350 cSt PDMS	4378	3.8 ppm
^{14}C-1000 cSt PDMS	3409	5.1 ppm

Solution 2 (125 μL) was placed in a 50 mL volumetric flask and one mL of a 10 ppm solution of Tween 80 in water was added. The flask was then made up to the mark with milli-Q water (solution 3). Solution 3 (500 μL) was further diluted to 50 mL in a volumetric flask with milli-Q water (solution 4). One mL aliquots of the solution 4 was used for SPE analysis. The average radioactivity of the solution, determined using a liquid scintillation counter, was 2633 dpm/mL. The average radioactivity of other standards and the respective concentration calculated from the radioactivity is given in Table 1.

SPE Procedures

SPE procedures were performed using both the manual vacuum manifold and the automated SPE Rapid Trace Work Station (Zymark). However, all the data (Tables 2-6) presented in this paper were generated using the manual vacuum manifold.

Extraction of MeSi(OH)$_3$, Me$_2$Si(OH)$_2$, and Metabolites of D$_4$, D$_5$,, and MM in urine

Sodium chloride (1g) was placed on top of the sorbent contained in a 500mg, ENV+ cartridge. The sorbent was conditioned using 6 mL methanol. The flow rate of methanol was 3 mL/min. This was followed by washing with 6 mL of milli-Q water (3 mL/min). The cartridge was then loaded with 1 mL aliquots of the aqueous solution of sample. The sample eluate (1mL/min) was collected into a 7 mL glass vial. The sorbent was air dried for 0.5 min. The analyte was eluted in three installments, each with 1mL aliquots of THF (1 mL/min) and the extracts were collected in separate 7 mL vials. Weights of the sample eluate and the THF extracts were recorded.

Table 2

Effect of SPE Conditions on the Recovery of D_4 (~8 ppb) from Water

Run #	Conditioning Solvent	Wash Step	Eluting Solvent	Percent Recovery
SPE Without NaCl in the C-18 Cartridge (Single Determination)				
1	Acetonitrile	Water	THF	76.8
2	Methanol	Water	THF	73.7
3	THF	Water	THF	71.5
4	Acetonitrile	Water	THF	86.9
SPE With NaCl in the C-18 Cartridge (Single Determination)				
5	Acetonitrile	Air dry	THF	96.8
6	Acetonitrile	Air dry	Acetone	95.5
7	Acetonitrile	Air dry	Methylene chloride	34.0
8	Acetonitrile	Air dry	Hexane	5.0
9	Acetonitrile	Hexane	THF	90.5
10*	Acetonitrile	Air dry	THF	87.7

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* Sample was diluted with two additional volumes of water.

Table 3

Sample	Sample Conc.	Extracted dpm	Residue dpm	% Recovery	Mean & Std. Dev.
$^{14}C-D_4$	11 ppb	2595	152	94	
		2226	210	91	
		2417	301	89	91.0 ± 2
$^{14}C-D_5$	24 ppb	4179	84	98	
		4952	57	99	
		4006	26	99	98.6 ± 0.5
^{14}C-MM	7 ppb	2363	152	94	
		2276	63	97	
		2031	72	97	96.0 ± 2

Table 4

Recovery of High Molecular Weight PDMS of Viscosity 350 and 1000 cSt from Aqueous Solution Using C4 Cartridge and Hexane Elution

Sample	Sample Conc.	Extracted dpm	Residue dpm	% Recovery	Mean & Std. Dev.
^{14}C-350 cSt PDMS	3.8 ppm	3931	410	91	
		3907	592	87	
		3854	441	90	89.3 ± 1.7
^{14}C-1000 cSt PDMS	5.1 ppm	3304	152	96	
		3252	140	96	
		3233	147	96	96.0 ± 0

Table 5

Recovery of Silanol Standards from Aqueous Solutions Using ENV + and THF Elution

Sample	Sample Conc.	Extracted dpm	Residue dpm	% Recovery	Mean & Std. Dev.
^{14}C-$Me_2Si(OH)_2$	57 ppb	3143	9	99	
		3120	15	99	
		3125	13	99	99.0 ± 0
^{14}C-$MeSi(OH)_3$	102 ppb	2409	82	92	
		2236	233	91	
		2241	137	94	94.0 ± 0

Extraction of D_4, D_5, and MM

The procedure is similar to that described above with several variations. For easy perusal, the procedural steps can be summarized as follows:

Cartridge: 6 mL size cartridge containing 1000 mg C-18

Step 1: Place 1 g NaCl in the cartridge.

Table 6

Recovery of Urinary Metabolites of D_4, D_5, and MM from Rat Urine using ENV^+ = and THF Elution

Sample	Sample Conc.*	Extracted dpm	Residue dpm	% Recovery	Mean & Std. Dev.
Metabolites of $^{14}C-D_4$	9 ppb	2307	68	97	
		2289	240	91	
		2284	262	90	93 ± 3
Metabolites of $^{14}C-D_5$	10 ppb	3166	343	90	
		3568	186	95	
		3444	143	96	94 ± 3
Metabolites of ^{14}C-MM	10 ppb	2849	5	99	
		3509	229	94	
		3523	199	95	96 ± 2

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* Concentration for the mixtures of components are based on the total radioactivity as parent compound equivalents.

Step 2: Condition column with 6 mL acetonitrile (1mL/min).

Step 3: Dry column with air for 30 sec.

Step 4: Load 1mL of aqueous sample onto column (1 mL/min).

Step 5: Dry column with air for 40 sec.

Step 6: Elute analyte three times, each with 1 mL THF (1 mL/min).

SPE Procedure for 350 cSt and 1000 cSt Polydimethylsiloxanes

The procedure is similar to that described for D_4, D_5 and MM above with the following variations: cartridge: 3 mL size cartridge containing 200 mg C4; In step 5, the sorbent was rinsed with 6 mL methanol at a flow rate of 2 mL/min instead of air drying for 40 sec. In step 6, the analytes were eluted three times, each with 2 mL hexane at a flow rate of 1 mL/min.

Determination of Percent Recovery (SPE Efficiency)

Aliquots (100-200 μL) of the sample eluate were mixed with ~6.5 mL of the scintillation cocktail Hionic-Fluor and the radioactivity measured. Taking into account the dilution factor, the total dpm (radioactivity) of the sample eluate was calculated. In a similar fashion the total radioactivity of solvent extracts was obtained. The residual radioactivity that remained on the sorbent un-extracted was determined as follows: The entire sorbent was removed from the cartridge, oxidized using a Biological Oxidizer, the $^{14}CO_2$ generated collected in a Carbon-14 cocktail and the activity measured. The percent recoveries were then calculated as follows:

$$\%\ \text{Recovery} = \frac{\text{Total dpm extracted}}{\text{Total dpm of the sample}} \times 100$$

Total dpm of the sample = total dpm extracted + total dpm of the sample eluate + dpm of the residual analyte on the sorbent.

RESULTS AND DISCUSSION

SPE Results on Non-Polar Siloxanes

Initial work was focused on developing an SPE procedure for the recovery of D_4 from aqueous medium. For non-polar material such as D_4, sorbent such as C2, C4, and C18 are expected to perform well. But C18 which has the least polar surface of the three sorbents was expected to be the best choice and therefore chosen for SPE experiments.

Developing an SPE procedure is as much an art as it is science. In addition to choosing an appropriate sorbent, several other critical parameters must also be optimized to obtain best recoveries of the analytes. These include: flow rates for samples, flow rates for elution solvents, the nature of the conditioning solvent, elution solvents, etc. Lower flow rates are in general better, especially, for the sample loading step, since they provide ample time for the interaction of the analytes with the sorbents. We determined the flow rates of 1 and 3 mL/min, respectively, for the sample load and solvent elution steps to be optimal. At these fixed flow rates, the effects of other changes such as conditioning solvent, elution solvent, etc., on the recoveries were studied and results are summarized in Table 2. The experiments were performed manually using vacuum manifold.

D_4 concentrations in water were at 7- 8 ppb level in all but run # 10. One mL sample aliquots were used in all but run #10 in which the concentration was ~ 2.5 ppb. It was apparent that conditioning with acetonitrile followed by air-drying, and elution with either THF or acetone (run # 5 and 6) resulted in best recovery. At these conditions, D_4 can be recovered with efficiencies of about 95%. SPE was also performed with a diluted sample (run #10). Here, the aqueous D_4 sample was diluted with 2 additional volumes of water and the same amount D_4 now present in a total of 3 mL was loaded. The intent here was to demonstrate the feasibility to recover smaller amounts of D_4 (or other silicones) present in large volumes of water. The recovery (87.7%) at this lower concentration of 2.5 ppb D_4 was still in the acceptable level. The recoveries for larger sample sizes may be improved further, if needed, by using SPE cartridges of higher loading capacity.

Runs 1-4 were carried out using the commercial cartridges as such. Runs 5-10 were, however, performed after placing a gram of solid NaCl on top of the sorbent in the cartridge. The reason for adding NaCl was to further decrease the solubility of D_4 in water (i.e. essentially attempting to salt out D_4) and, thus, release D_4 from the aqueous matrix to interact with the sorbent. This concept appears to be working as seen from the results in runs 5 and 6. It was interesting to note that CH_2Cl_2 and hexane which are excellent solvents for D_4 did not elute D_4 from the sorbent. This may be due to the fact that the sorbent following the sample load and air drying step still retained at its surface sufficient amount of moisture to decrease the ability of these hydrophobic solvents to penetrate and dissolve D_4. With hydrophilic solvents such as acetone or THF therefore, the D_4 elution improved.

In the initial attempts (data in Table 2) to determine the recovery, no attempt was made to determine how much of D_4 was still being retained on to the sorbent following solvent elution. The recoveries here were calculated simply based on the total radioactivity recovered (i.e. radioactivity of the sample eluate + that of three extracts). To better assess the recovery, in subsequent experiments, at the completion of the analyte elution step, the sorbent was collected from the cartridge, subjected to combustion in the oxidizer and then the radioactivity measured using the liquid scintillation counter. Taking into account the residual radioactivities in the sorbents, the percent recovery was then calculated. The recovery values obtained in this manner are shown in Table 3. In a similar fashion, the SPE was also performed on other non-polar siloxanes viz., D_5 and MM and the results are included in Table 3. The efficiencies for all the three siloxanes tested were >90%.

SPE procedure was then extended to include high molecular weight polymers, viz. polydimethylsiloxane fluids of 350 cSt (number average molecular weight, Mn = ~9800), and 1000 cSt (Mn = ~17000) viscosity. The data are summarized in Table 4. In the case of these high molecular weight siloxanes, it was surprising to find that THF was not very effective in eluting the siloxanes from the sorbent (data not included). With THF, numerous elutions were needed to elute the analytes completely. We found, however, that hexane elution worked exceptionally well. Even a single elution with 2 mL hexane recovered nearly 90% of the analyte from the cartridge. Since hexane is incompatible with an aqueous matrix, it became necessary, however, after sample loading, to remove any adsorbed water off the sorbent by methanol wash. Also, though the C18 cartridge worked well for 350 cSt PDMS, it did not perform well for 1000 cSt PDMS. Even with hexane the analyte elution was sluggish. This may be due to a stronger affinity of the latter fluid (1000 cSt) to the sorbent. When the non-polar interaction was decreased by the use of C4 cartridge in place of the C18, the desorption by hexane was found to be greatly facilitated.

SPE Results on Polar Siloxanes

Low molecular weight silanols and hydroxymethyl silicones are especially water soluble. Of these, the methylsilanetriol, $MeSi(OH)_3$ is extremely water soluble and hence finding a sorbent to which it can partition effectively from the aqueous matrix was difficult. It was noted, however, that Amberlite® XAD resins were quite successful in scavenging and accumulating trace levels of organic contaminants in environmental samples[17] especially from large volumes of aqueous matrices. These resins were shown to be capable of trapping organic compounds of varied polarities. But experiments with XAD resins (Amberlite® XAD-2 and XAD-4 non-ionic polystyrene adsorbents as well as Amberlite® XAD-7 non-ionic polyacrylate adsorbent) on standard solutions of $MeSi(OH)_3$ in water resulted only in very poor recoveries (<30%). However, a non-silica absorbent, ENV+ which is a styryl-divinylbenzene polymer was found to be quite efficient. Using this sorbent, the recoveries for $Me_2Si(OH)_2$ and $MeSi(OH)_3$ from pure mill-Q water were 99 and 94% respectively (Table 5).

In the case of $Me_2Si(OH)_2$, a good recovery was also achieved with a larger sample size (10 mL) at 0.7 ppb concentration. But when applied to $MeSi(OH)_3$ at this volume and concentration, the recovery dropped considerably (50%). To show that SPE process does not alter the structures, HPLC analysis was carried out before and after SPE for the most reactive of the two silanols, viz. $MeSi(OH)_3$. HPLC-radiochromatogram of the extract reconstituted in water (after removing THF) shown in Figure 1 attest to this fact. The presence of $MeSi(OH)_3$ is seen at retention time (3.3 min) which compares with that of the standard prior to SPE. The minor components eluting at 7.60 and 26.8 min are impurities present in the standard.

The ENV+ cartridge was then applied to rat urine samples containing metabolites of D_4, D_5 and MM. Urinary metabolites of D_4 and D_5 have hydroxy functions[8] and those of MM have, in addition, hydroxymethyl functionalities.[18] The metabolites were recovered quite efficiently from 1mL as well as 10 mL sample sizes (Table 6). For the purpose of illustration, the HPLC radiochromatogram obtained for the metabolites of D_4 following the SPE process is shown (Figure 2). The profile was identical to that obtained before urine was

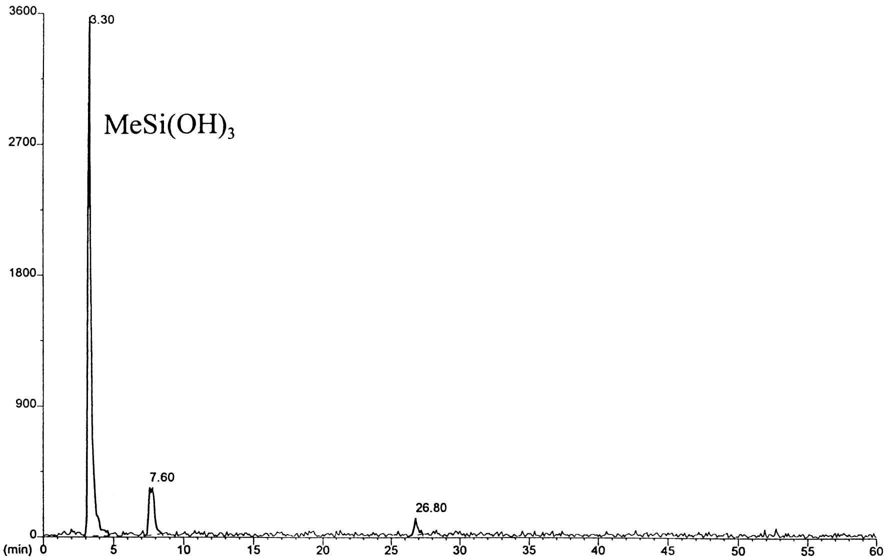

Figure 1. HPLC/Radiochromatogram of $MeSi(OH)_3$ following SPE.

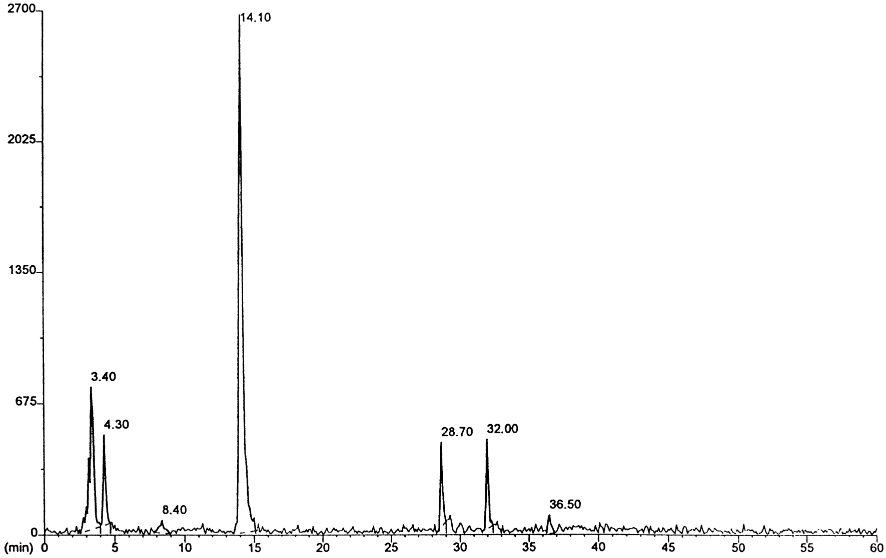

Figure 2. HPLC/Radiochromatogram of samples containing metabolites of D_4 following SPE. Structures of components at various retention times are as follows: 3.4 min, $MeSi(OH)_3$; 4.3 min, $HOMe_2SiOSi(OH)_3$; 14.10 min, $Me_2Si(OH)_2$; 28.7 min, $(HO)_2MeSiOSiMe_2OH$; 32.0 min, $HO(SiMe_2O)_2H$; 36.5 min, $HO(SiMe_2O)_3H$.

subjected to SPE process. Details concerning the structural assignments of the major component in the HPLC profile are presented elsewhere.[8]

Recovery of Metabolites of MM and D_5 from Plasma

From plasma, the recoveries for the metabolites of MM and D_5 were ~80 and 66%, respectively. The sample elute contained the remaining radioactivity. Thus the low recoveries can be attributed to the emulsified nature of the plasma samples. It is speculated that in order to improve the efficiency, the emulsion must be broken by protein precipitating agent before loading on to the cartridge.

CONCLUSION

SPE is shown to be a useful procedure to recover organosilanols and siloxanes of various types from aqueous matrices. Use of ^{14}C-labeled materials made it convenient to determine the percent recoveries. Using appropriate sorbents, functional and non-functional organosilicon materials can be recovered with >90% efficiency. Non-polar siloxanes require the use of C4 and/or C18 sorbents whereas the polar silanols require styryl-divinylbenzene sorbents. Irrespective of the nature of siloxanes, placing solid NaCl onto the sorbent to saturate the aqueous matrices was found to enhance the recoveries. HPLC analyses performed on samples following SPE process clearly established that the extraction methodology did not alter the structure of the original organosilicon materials.

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