$PhCONHCH_2OCH_3$ (9) showed them to be identical (R_f 0.3 with Et_2O).

In a separate experiment, a solution of 6 (30 mg, 0.12 mmol) in 1 mL of CD_2Cl_2 in an NMR tube was treated with O_3 as described for 5. After the mixture was warmed to room temperature, immediate ^1H NMR analysis indicated 66% conversion to sulfoxide 10: ^1H NMR (CD_2Cl_2) δ 4.40 (dd, 1 H, J_{gem} = 12 Hz, J_{CH-NH} = 6 Hz), 4.84 (dd, 1 H, J_{gem} = 12 Hz, J_{CH-NH} = 6 Hz), 7.3–8.1 (m, 11 H, including NH). After the sample was warmed to 40 °C for 10 min, ^1H NMR analysis indicated that no sulfoxide 10 remained; the decomposition products were not analyzed further.

$NaIO_4$ Oxidation of 6: Preparation of *N*-[(Phenylsulfonyl)methyl]benzamide (11). To a stirred solution of 6 (130 mg, 0.535 mmol) in 20 mL of CH_3OH was added a solution of $NaIO_4$ (1.145 g, 5.35 mmol) in 5 mL of H_2O by Pasteur pipet at room temperature. The reaction mixture was stirred at room temperature for a total of 60 h. Over the course of the reaction, additional $NaIO_4$ (3.45 g, 6.1 mmol) was added periodically until silica gel TLC showed no starting sulfide 6 and only a trace of sulfoxide 10. The white precipitate was suction filtered off with a fritted-glass funnel, and then the volatiles were removed on a rotary evaporator. The white solid residue was extracted with 2 × 40 mL of Et_2O, and then the combined Et_2O solution was dried over anhydrous Na_2SO_4. Filtration followed by evaporation provided crude product, which was purified by preparative silica gel TLC with 40:60 (v:v) EtOAc/Et_2O to provide 60 mg (40%) of sulfone 11. ^1H NMR analysis of the remainder of the preparative plate cut as a single sample revealed minor products as follows: 4 (5%), 9 (8%), and 10 (8%). Recrystallization of chromatographically purified 11 with Et_2O/EtOAc furnished white needles of 11: mp 127–129 °C (lit.[14] mp 129–131 °C); ^1H NMR ($CDCl_3$) δ 4.91 (d, 2 H, J = 7 Hz), 6.85 (br m, 1 H), 7.4–7.9 (m, 10 H); IR ($CDCl_3$) 1680 (s), 1511 (s), 1490 (s), 1322 (s, sulfone), 1148 (s) cm^{-1}. Anal. Calcd for $C_{14}H_{13}NO_3S$: C, 61.07; H, 4.76; N, 5.09; S, 11.65. Found: C, 60.85; H, 4.66; N, 4.92; S, 11.63.

Thermolysis of 11. A solution of 11 (5 mg, 0.018 mmol) in 1 mL of CH_3OH was refluxed for 45 h with periodic monitoring by silica gel TLC. After 45 h, the solvent was removed with a rotary evaporator leaving 4.5 mg of residue. Silica gel TLC and ^1H NMR analyses of the residue showed that it was exclusively unchanged 11.

Acknowledgment. We acknowledge the financial assistance of the University of Oregon, the National Institutes of Health Biomedical Research Support Grant 2 S07 RR07080, the Medical Research Foundation of Oregon, and the American Heart Association, Oregon Chapter. The General Electric QE-300 NMR spectrometer was purchased with funds provided by PHS Grant RR 02336 and NSF Grant CHE 8411177.

Registry No. 4, 6282-02-6; 5, 110682-74-1; 6, 58379-67-2; 7, 110682-75-2; 8, 81793-17-1; 9, 13156-28-0; 10, 110682-76-3; 11, 76965-50-9; $PhCONH_2$, 55-21-0; HCHO, 50-00-0; PhSeH, 645-96-5; PhSH, 108-98-5.

Synthesis of Phospholipids Suitable for Covalent Binding to Surfaces

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Received March 17, 1987

Phospholipids are an important class of organic compounds and find extensive applicability in a variety of fields.[1] Their most important area of utility is in the study of biomembrane structure and function.[2-5] Synthetic as well as naturally occurring phospholipids are used as membrane model systems for investigating lipid–protein,[6-8] lipid–steroid,[9] lipid–receptor,[2] and other similar biochemical interactions that occur within the biological milieu. During the past decade, extensive work has been carried out to determine the effects on the nature and extent of these interactions of altering the head-group structure,[10] length of the acyl chain,[5] substituting the ester function by ether linkages,[11] and introducing polymerizable moieties into the lipid system.[12,13]

In our work[14,15] we have been concerned with the development of chemical sensors based on the use of the lipid membrane as a chemoreceptive transducer for bimolecular interactions. Present technology involves deposition of lipid layers on polymer and other surfaces by the Langmuir–Blodgett technique. However, it was found that lipid films prepared by this method lack durability and were also subject to undesirable perturbation which imposed limitations on their use as membranes for extended periods of time. Covalent binding of lipids to substrates offers an attractive route to overcome these problems. This type of attachment can be accomplished through the introduction of a reactive functional group such as a chlorosilyl moiety into the alkyl chains of the lipid skeleton and reacting it with the surface hydroxyl groups. The reactivity of simpler chlorosilanes with silicon and other metallic surfaces containing a thin oxide layer has been extensively examined previously,[16-18] and the chlorosilylated lipid is expected to behave analogously. The present paper describes the synthesis of two such [(chlorosilyl)oxy] acyl-substituted phosphatidylcholines (12 and 21) suitable for covalent attachment to a silica surface. Previous reports on the covalent binding of lipids[19] and lipid precursors[20] to silica surfaces made use of photoactivable heterobifunctional cross-linking agents and a prelinked alkylamino function carrying a reactive acid chloride moiety respectively.

The synthetic route to 3-hexanoyl-2-[9-((chlorodimethylsilyl)oxy)nonanoyl]-*sn*-glycerophosphatidylcholine (12) has been outlined in Scheme I. The methyl ester of 9-hydroxynonanoic acid (4) could be isolated in only 35% yield starting from nonanedioic acid monomethyl ester (5),

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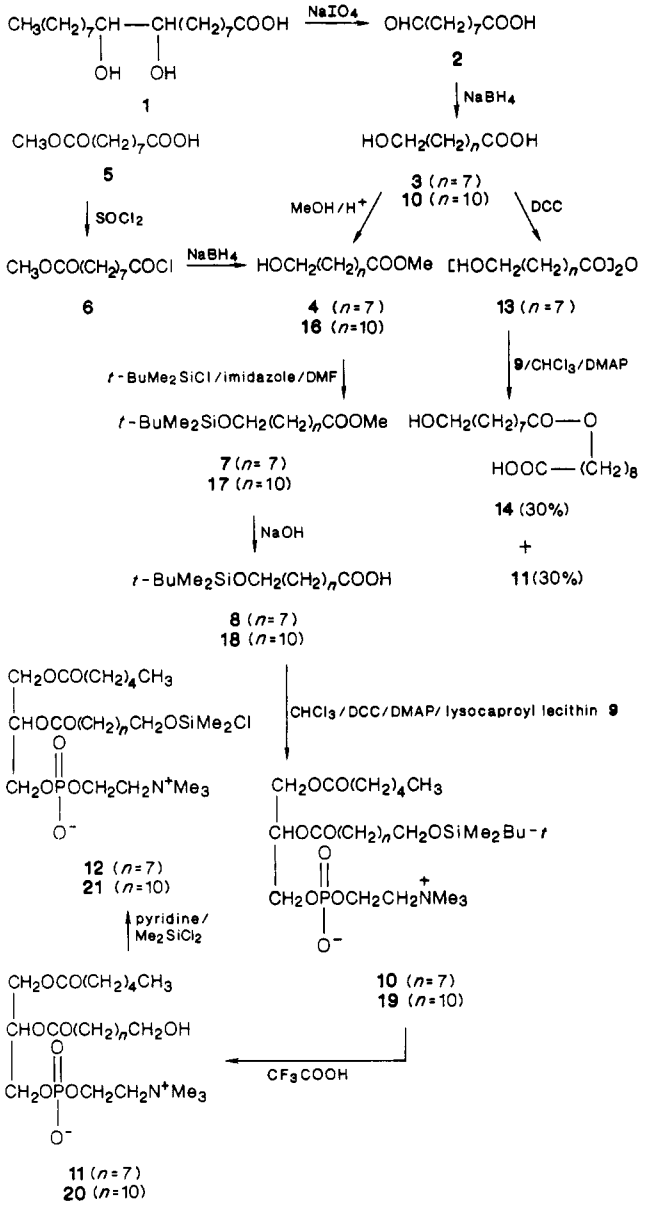
Scheme I. Synthesis of 3-Hexanoyl-2-[9-((chlorodimethylsilyl)oxy)nonanoyl]-*sn*-glycerophosphatidylcholine (12) and Its 2-[12-((Chlorodimethylsilyl)oxy)dodecanoyl] Analogue (21)



contrary to a previous report[21] claiming 71% yield for **4** from **5**. Trans-esterification seems to occur during the reduction of the acid chloride **6** as nonanedioic acid and its dimethyl ester were isolated as the byproducts. However, **4** could be obtained in good yield by the reduction of 9-oxononanoic acid (**2**) with $NaBH_4$ in dioxane. The oxo acid **2** was made by the oxidative cleavage of 9,10-dihydroxyoctadecanoic acid (**1**) with periodic acid following an earlier procedure.[22]

The hydroxyl group in **4** was protected by silylation with *tert*-butyldimethylsilyl chloride and hydrolysis of the resulting silyl ester **7** with alkali afforded the silylated acid **8**. Treatment of **8** with lysocaproyllecithin **9** in the presence of DCC and (dimethylamino)pyridine (DMAP) under the same conditions utilized by Eibl[5] resulted in the phospholipid **10**. The silyl protective group in **10** was removed with trifluoroacetic acid to give the hydroxyacyl lipid **11**, which was resilylated with dichlorodimethylsilane to yield the desired product **12**. Lipids **10–12** were all characterized by proton and ^{13}C NMR respectively.

The synthesis of **11** directly from the hydroxy acid **3** was attempted by converting the latter into the anhydride **13** and condensation with the lysocaproyllecithin **9**. The acylated lipid **11** could be obtained in only 30% yield since condensation between the free acid liberated from the anhydride with excess anhydride occurred resulting in **14**, characterized by its proton NMR spectrum.

The synthesis of 3-hexanoyl-2-[12-((chlorodimethylsilyl)oxy)dodecanoyl]-*sn*-glycerophosphatidylcholine (**21**) was carried out by an analogous procedure to that of **12**, as outlined in Scheme I. The lipids **19–21** were characterized by their proton and ^{13}C NMR spectra.

Preliminary analysis of silicon wafers coated with **12** and **21** (see Experimental Section for details) by photoelectron spectroscopy (XPS) indicated that attachment of the lipid to the surface occurred to the extent of at least 30%. The details of XPS studies will be reported separately.

Experimental Section

Lysocaproyllecithin **9** was obtained from Avanti Polar Lipids Inc., Birmingham, AL. 12-Hydroxydodecanoic acid (**15**), 9,10-dihydroxystearic acid (**1**), nonanedioic acid monomethyl ester (**5**), and *tert*-butyldimethylsilyl chloride were supplied by Aldrich. The silicon wafers were supplied by the Avrel Company, St. Charles, MO 63303. Chloroform was freshly distilled over P_2O_5 under nitrogen. Dimethyl formamide, dioxane, and methanol were freshly distilled over molecular sieves before use.

Proton NMR spectra were recorded on a Varian T-60 instrument and ^{13}C NMR spectra on a Varian XL-200 spectrometer in $CDCl_3$ solution. HPLC analyses were performed on a Varian Vista 5500 liquid chromatograph using a reverse-phase C-18-μ-Bondapak column with chloroform as solvent and a flow rate of 1 mL per min. All new compounds reported gave satisfactory carbon, hydrogen, and nitrogen analyses.

Reduction of 9-Oxononanoic Acid (2) with Sodium Borohydride. A solution of 1.7 g (0.01 mol) of the oxo acid **2**[23] in dioxane (10 mL) was treated with 0.2 g (0.05 mol) of sodium borohydride in small portions with cooling in ice–water. The mixture was then stirred for 10 min and poured over crushed ice. The product was extracted with 100 mL of ether and the organic layer dried over anhydrous $MgSO_4$ and concentrated on a rotary evaporator to yield a white solid in 80% yield. Recrystallization from alcohol gave pure 9-hydroxynonanoic acid, mp 53 °C (lit.[21] mp 54 °C).

Esterification of 9-Hydroxynonanoic Acid (3). A solution of 1.1 g (6.3 mmol) of the hydroxy acid in 20 mL of methanol was refluxed for 4 h after the addition of two drops of concentrated H_2SO_4. The mixture was poured on ice and the precipitate extracted with ether. The usual workup gave 1.1 g (96%) of the methyl ester **4**, bp 135–136 °C (3mm).

The same ester **4** was also prepared by the reduction of the monoacid chloride **6** adopting Dale's procedure.[21] However, the yield of **4** was only 35%, azelaic acid and its dimethyl ester being formed in a combined yield of 40%.

Silylation of 4 to 7. The methyl ester **4** (1 g, 5 mmol) and imidazole (0.4 g, 6 mmol) were dissolved in 5 mL of DMF, and a solution of 0.8 g of *t*-butyldimethylsilyl chloride in 5 mL of DMF was added dropwise. The mixture was stirred for 24 h at room temperature under nitrogen. The solvent and excess chlorosilane were removed under vacuum, and the residue was extracted with ether. Usual workup gave 1.3 g (85%) of **7**, bp 145–146 °C (0.2 mm).

9-[(*tert*-Butyldimethylsilyl)oxy]nonanoic Acid (8). The above silyloxy ester **7** (4 mmol) was stirred at room temperature with 2 mL of 10% aqueous NaOH in 10 mL of 1:1 THF–methanol for 24 h. The mixture was cooled in ice, acidified with 6 M HCl, and ether extracted. Usual workup gave **8** as a viscous oil in almost quantitative yield.

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3-Hexanoyl-2-[9-((*tert*-butyldimethylsilyl)oxy)nonanoyl]-*sn*-glycerophosphatidylcholine (10). A mixture of 0.2 g (0.6 mmol) of lysocaproyllecithin **9**, 0.2 g (0.66 mmol) of 9-[(*tert*-butyldimethylsilyl)oxy]nonanoic acid (**8**), 0.15 g (0.75 mmol) of dicyclohexylcarbodiimide, and 50 mg (0.4 mmol) of (dimethylamino)pyridine were stirred in 100 mL of chloroform at room temperature under nitrogen for 48 h. The mixture was filtered to remove dicyclohexylurea and concentrated on a rotary evaporator. The residue in a small volume of chloroform was passed through a column of Rexyne I-300. Initially, the column was eluted with chloroform to remove excess **8** and then with 1:1 chloroform–methanol to recover the title product **10**. Further purification of **10** was effected by HPLC. The lipid **10**, recovered in 60% yield, had a retention time of 5.6 min.

3-Hexanoyl-2-(9-hydroxynonanyl)-*sn*-glycerophosphatidylcholine (11). The above silyloxy lipid **10** (0.3 g, 0.5 mmol) was treated in dioxane solution with trifluoroacetic acid (0.6 mL) and1 mL of water. After being stirred at room temperature for 1 h, the product was extracted with chloroform. Usual workup gave the desilylated lipid **11** in about 90% yield.

The preparation of **11** was also carried out by directly condensing lysocaproyllecithin **9** (0.6 mmol) with 0.22 g (0.66 mmol) of the hydroxy acid anhydride **13** in the presence of (dimethylamino)pyridine (0.05 g, 0.4 mmol) in chloroform solution at room temperature. The anhydride **13** was earlier made by treating the acid **3** with DCC in dichloromethane solution at room temperature for 5 h. The product **11** could be obtained in only 30% yield after chromatography over silica gel. A solid, mp 89 °C, was also recovered in an earlier fraction eluted with chloroform in ~35% yield and was characterized by its ^{1}H NMR as **14**.

3-Hexanoyl-2-[9-((chlorodimethylsilyl)oxy)nonanoyl]-*sn*-glycerophosphatidylcholine (12). The lipid **11** (0.2 g, 0.5 mmol) was dissolved in chloroform and stirred at room temperature under nitrogen with 0.8 g of dichlorodimethylsilane and 0.5 g of pyridine for 24 h. The mixture was filtered to remove pyridine hydrochloride and concentrated in vacuum to yield 0.2 g of residue (65%), which was purified by HPLC. The product **12** had a retention time of 4.2 min: ^{1}H NMR ($CDCl_3$, ppm from TMS) 0.21 (s, 6 H), 0.91 (t, 3 H), 1.03–1.63 (br s, 18 H), 2.26 (t, 3 H), 3.48 (br s, 9 H), 3.67–4.25 (br m, 11 H); ^{13}C NMR ($CDCl_3$, ppm from TMS) 1.66 ($SiCH_3$), 13.55 (CH_3), 21.85, 24.10, 30.73, 33.71 (11 C, aliphatic), 54.17 ($N(CH_3)_3$), 60.48 (CH_2O), 60.53 (CH_2O), 63.75 (CH_2OSi), 65.47 (CH_2NMe_3), 70.43 (*C*HO), 70.55 ($OCH_2CH_2NMe_3$), 172.56 and 173.32 (*C*=O).

Synthesis of 3-Hexanoyl-2-[12-((chlorodimethylsilyl)oxy)dodecanoyl]-*sn*-glycerophosphatidylcholine (21). This compound was obtained by a procedure similar to **12**. Condensation of the lysolecithin **9** with 12-[(*tert*-butyldimethylsilyl)oxy]dodecanoic acid[23] according to the conditions utilized for making **10**, gave **19** in ~75% yield (HPLC retention time, 6.1 min). Product **19** was desilylated to **20** in 90% yield with trifluoroacetic acid and **20** was resilylated with dichlorodimethylsilane (for conditions, see preparation of **12**) to yield about 70% of **21** (HPLC retention time, 4.9 min): ^{1}H NMR ($CDCl_3$, ppm from TMS) 0.18 (s, 6 H), 0.87 (t, 3 H), 1.04–1.68 (br s, 24 H), 2.32 (t, 4 H), 3.51 (br s, 9 H), 3.60–4.09 (br m, 11 H); ^{13} NMR ($CDCl_3$, ppm from TMS) 1.59 ($SiCH_3$), 13.50 (CH_3), 21.90, 24.05, 29.87, 30.15, 33.18 (14 C, aliphatic) 54.29 (NCH_3), 60.48 (CH_2O), 60.60 (CH_2O), 62.55 (CH_2OSi), 65.41 (CH_2NMe_3), 70.35 (*C*HO), 70.63 ($OCH_2CH_2NMe_3$), 173.40, 174.71 (*C*=O).

Surface Attachment of 12 and 21. Oxidized silicon wafers, pretreated according to previously reported procedures,[24] were refluxed with the appropriate chlorosilyl derivative (0.1 g, 0.15 mmol) in dry chloroform (25 mL) and dry pyridine (0.2 mL) in a nitrogen atmosphere for 24 h. The solution was decanted off, and the wafers were washed several times with chloroform and methanol and finally vacuum-dried. For longivity, these lipid-treated wafers were stored under nitrogen in the refrigerator.

Registry No. **2**, 2553-17-5; **2**-ol, 3788-56-5; **4**, 34957-73-8; **7**, 110774-17-9; **8**, 110774-18-0; **9**, 58445-96-8; **10**, 110774-19-1; **11**, 110774-20-4; **12**, 110774-23-7; **13**, 110774-21-5; **14**, 110774-22-6; **18**, 77744-42-4; **19**, 110774-24-8; **20**, 110796-30-0; **21**, 110774-25-9.

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A Convenient Synthesis of Tetrakis(2-bromoethyl)methane[1]

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Received April 21, 1987

During our quest to prepare tetradirectional unimolecular cascades,[3] we needed convenient access to symmetrical tetrasubstituted methanes. The obvious derivatives of pentaerythrol such as its tetramesylate or tetrakis(bromomethyl)methane were inert to nucleophilic substitution under normal conditions.[4] It was surmised that the corresponding homologue would circumvent this problem, thus a required high-yield route to tetrakis(2-bromoethyl)methane (**1**) was devised and is herein reported.

Classically, **1** was prepared by the bromination of tetrakis(2-hydroxyethyl)methane,[5] which was derived from citric acid in eleven steps.[6] Our approach to the synthesis of tetrabromide **1** is shown in Scheme I.

Tetrahydro-4*H*-pyran-4-one (**3**), prepared in a two-step process from the readily available 3-chloropropionyl chloride,[7] was treated with excess ammonia in absolute ethanol and 2 mol of ethyl cyanoacetate to give the ammonium salt of the Guareschi imide (**4a**).[8] The free imide (**4b**) was easily obtained by acidification of the ammonium salt **4a** with concentrated hydrochloric acid. Hydrolysis/decarboxylation of **4a** to the *gem*-diacid **5a** by treatment with warm sulfuric acid or alkaline conditions caused considerable decomposition due to ring cleavage. Reaction of the salt of **4** with boiling concentrated hydrochloric acid smoothly led to the desired diacid in 65% from salt **4a**. Acid **5a** was esterified under normal Fischer conditions to give (72%) the diester, which was reduced with $LiAlH_4$ to generate (82%) diol **6**. The ^{13}C NMR showed two different α (δ 58.1 and 63.5) and β (δ 36.4 and 39.2) carbon atoms, which is supportive of the 4,4-disubstituted pyran **6**.

Treatment of diol **6** with KBr in concentrated H_2SO_4 or with a mixture of HBr (48%) and concentrated H_2SO_4 gave a mixture of **7** and **1** in poor isolated yield. The reaction of diol **6** with concentrated HCl gave (65%) spiro diether **7** as the major product. Subsequent treatment of this spiro diether with a mixture of HBr and H_2SO_4 gave the desired tetrabromide **1** via smooth ether cleavage. The direct conversion of diol **6** to tetrabromide **1** was accomplished (57%) by bromination with PBr_3 and HBr (48%). The reaction of **1** and potassium carbonate at 275 °C gave a trace amount of tetravinylmethane.[9]

Experimental Section

General Comments. Melting point data were obtained from

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