Registry No.—Boc-L-Ala, 15761-38-3; DCC, 538-75-0; EEDQ, 16357-59-8; Woodward's reagent K, 4156-16-5; ethyl chloroformate, 541-41-3.

References and Notes

(1) This study was supported by grants from the U. S. Public Health Service (NIH AI-07515 and AM-12473).
(2) The preliminary study was made in collaboration with Dr. J. T. Sheehan of The Squibb Institute for Medical Research; cf. M. Bodanszky in "Peptides, 1971", H. Nesvedba, Ed., North-Holland Publishing Co., Amsterdam, 1973, p 81.
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Use of (2,3-Dihydro-2-oxo-1*H*-1,4-benzodiazepin-3-yl)-phosphonic Acid Esters as Novel "Wittig Reagents"

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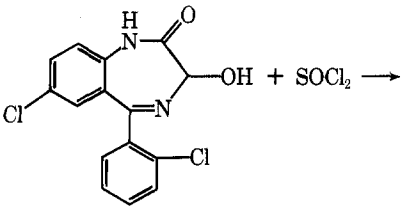
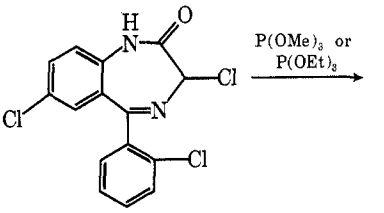
Because of our interest in the 1,4-benzodiazepine field, we sought a convenient method for the preparation of various useful lorazepam (**1**)[1] derivatives having functional substituents at the 3 position. One of our first thoughts was to prepare the 3-ketone and 3-methylene derivatives of **1** and use these groups as reactive intermediates. Only one paper[2] has described the preparation of any 1*H*-1,4-benzodiazepine-2,3-diones, and these preparations required the use of ruthenium tetroxide, which on any large preparative scale would be prohibitively expensive (5 g/$195.00). The preparation of 3-methylene-2*H*-1,4-benzodiazepin-2(3*H*)-ones has not been described. Instead of using this oxidation approach for the 3-keto type compounds, we decided to try making a "Wittig–Horner" type reagent from the benzodiazepine itself and using this reagent for the preparation of our desired intermediates. We found that **1** was easily converted to its corresponding 3-chloro derivative (**2**) with $SOCl_2$.[3] Condensation of **2** with $P(OMe)_3$ and $P(OEt)_3$ gave respectively **3** and **4**, by an Arbuzov–Perkow reaction.[4-6] Both **3** and **4** were methylated on the amide nitrogen by sodium hydroxide and dimethyl sulfate, giving respectively **5** and **6**. The acidic 3 carbon adjacent to the phosphorus was not methylated, at least on **3**, as evidenced by the $P-H_3$ coupling of **3** which is still present in the product **5**. Presumably this was also true in methylation of **4** to **6**, because **6** behaved like a Wittig reagent and the exchangeable NH of **4** disappeared. During one attempt to methylate the nitrogen of **3** with sodium hydride and methyl iodide in DMF, only **7** was isolated. Apparently the sodium iodide formed from the methylation on nitrogen caused an anionic demethylation of one of the phosphate OMe groups.[7]

The phosphonate carbanion of **6** was prepared in 1,2-dimethoxyethane with sodium hydride,[8] and reaction with gaseous formaldehyde readily gave **8**. Surprisingly, in spite of the apparent stability of phosphonate carbanions to oxygen,[4] reaction of the sodium salt of **6** with oxygen readily gave **9**.



1



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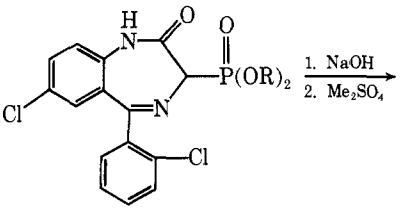
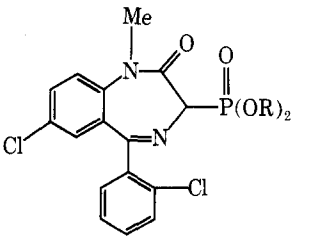
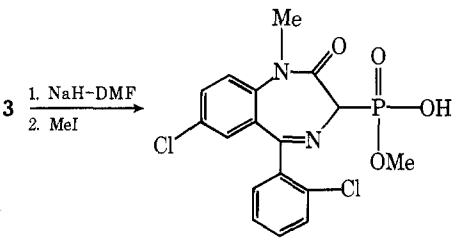


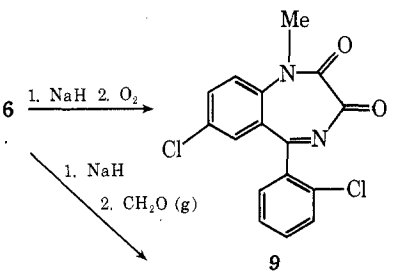
3, R = Me
4, R = Et



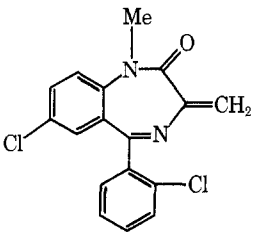
5, R = Me
6, R = Et



7

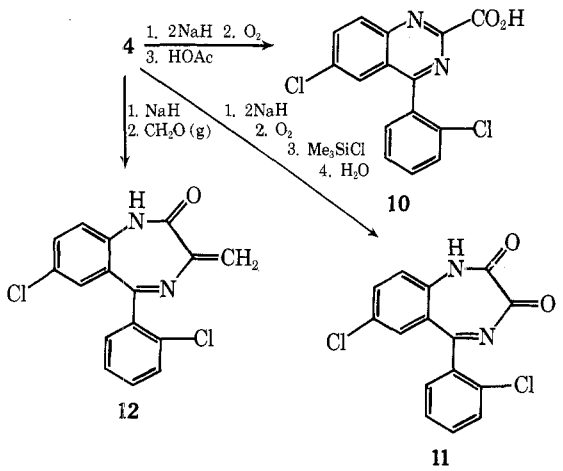


9



8

In order to prepare the carbanion of 4 it was necessary to use 2 mol of sodium hydride, and in the subsequent oxygenation and acid work-up only 10 was formed, by a known rearrangement.[2] In subsequent oxygenations, the intermediate salt of 11 was neutralized with Me_3SiCl and the resulting silylated amide was hydrolyzed under neutral conditions. Analogous to the reactions of 6, the carbanion of 4 gave 11 on oxygenation and 12 when condensed with gaseous formaldehyde.



Experimental Section

Melting points were taken in capillary tubes in an oil bath and are uncorrected. Solvents were removed in vacuo on a Büchi Rotavapor R. Anhydrous sodium sulfate was used for all solution drying. Spectra were obtained under the supervision of Mr. Bruce Hofmann. Ir spectra were determined in KBr pellets using a Perkin-Elmer Model 21 spectrophotometer. NMR spectra were determined with a Varian Model A-60 or a Jeolco Model C-60HL NMR spectrometer using TMS in DMSO-d_6. Analyses were carried out on a Perkin-Elmer Model 240 elemental analyzer.

[7-Chloro-5-(*o*-chlorophenyl)-2,3-dihydro-2-oxo-1*H*-1,4-benzodiazepin-3-yl]phosphonic Acid Dimethyl Ester (3). 7-Chloro-5-(*o*-chlorophenyl)-1,3-dihydro-3-hydroxy-2*H*-1,4-benzodiazepin-2-one (1, 32.1 g, 0.1 mol) and 150 ml of $SOCl_2$ were refluxed on a steam bath for 1.25 hr using a $CaCl_2$ tube. The solution was concentrated using a vacuum pump and a rotary evaporator. The residue was scrubbed twice with toluene and 125 ml of $P(OMe)_3$ was added. The mixture was heated on a steam bath for 2 hr, refluxed for 1.5 hr, and warmed on the steam bath overnight under N_2. The mixture was filtered and the cake was washed well with toluene, giving 42.5 g of crude 3 (mp 242° dec), which on crystallization (MeCN) gave 30 g (73%) of 3: mp 248° dec; ir 5.94 μ (C=O); NMR (DMSO-d_6) δ 3.78 (d, 3, J = 11 Hz, CH_3), 3.82 (d, 3, J = 11 Hz, CH_3), 4.50 (d, 1, J = 11 Hz, 3-CH), 7.01 (d, 1, J = 1.5 Hz, 6-CH), 7.2–7.85 (m, 6, aromatic), 11.05 (d, 1, J = 5 Hz, exchangeable NH).

Anal. Calcd for $C_{17}H_{15}Cl_2N_2O_4P$: C, 49.42; H, 3.66; N, 6.78; Cl, 17.16. Found: C, 49.45; H, 3.76; N, 6.85; Cl, 17.17.

[7-Chloro-5-(*o*-chlorophenyl)-2,3-dihydro-2-oxo-1*H*-1,4-benzodiazepin-3-yl]phosphonic Acid Diethyl Ester (4). A solution of 1 (6.42 g, 0.02 mol) in 50 ml of $SOCl_2$ was refluxed for 1 hr on a steam bath using a $CaCl_2$ tube. The solution was concentrated using a vacuum pump and a rotary evaporator. The residue was scrubbed twice with toluene, 45 ml of $P(OEt)_3$ was added, and the mixture was heated on the steam bath overnight under N_2. The resulting solution was concentrated and the residue was crystallized (MeCN), giving 6.5 g (74%) of 4: mp 172–174°; ir 5.92 μ (C=O); NMR (DMSO-d_6) δ 1.25 (t, 6, J = 7.5 Hz, CH_3), 3.92–4.64 (m, 4, CH_2), 4.42 (d, 1, J = 12 Hz, 3-CH), 7.02 (d, 1, J = 2 Hz, 6-CH), 7.24–7.85 (m, 6, aromatic), 11.02 (d, 1, J = 5 Hz, exchangeable NH).

Anal. Calcd for $C_{19}H_{19}Cl_2N_2O_4P$: C, 51.71; H, 4.34; N, 6.45; Cl, 16.07. Found: C, 52.06; H, 4.35; N, 6.81; Cl, 15.96.

[7-Chloro-5-(*o*-chlorophenyl)-2,3-dihydro-1-methyl-2-oxo-1*H*-1,4-benzodiazepin-3-yl]phosphonic Acid Dimethyl Ester (5). Addition of 10 ml of 1 *N* NaOH to a mixture of 4.14 g (0.01 mol) of 3 in 50 ml of THF caused the solid to dissolve, giving a red solution. Addition of 0.944 ml (0.01 mol) of Me_2SO_4 was carried out over 1 min, and the solution was stirred for 3 hr at room temperature. The solution was concentrated, H_2O was added, and the solution was extracted twice with CH_2Cl_2. The organic layer was washed successively with H_2O and brine, dried, and concentrated. The product (3.89 g) was crystallized (toluene and EtOAc–hexane), giving 2.6 g (61%) of 5: mp 185–187°; ir 6.00 μ (C=O); NMR (DMSO-d_6) δ 3.4 (s, 3, NCH_3), 3.75 (d, 3, J = 11 Hz, OCH_3), 3.85 (d, 3, J = 11 Hz, OCH_3), 4.49 (d, 1, J = 11.25 Hz, 3-CH), 7.02 (s, 1, 6-CH), 7.45–7.8 (m, 6, aromatic).

Anal. Calcd for $C_{18}H_{17}Cl_2N_2O_4P$: C, 50.60; H, 4.01; N, 6.56; Cl, 16.60. Found: C, 50.61; H, 3.89; N, 6.48; Cl, 16.76.

[7-Chloro-5-(*o*-chlorophenyl)-2,3-dihydro-1-methyl-2-oxo-1*H*-1,4-benzodiazepin-3-yl]phosphonic Acid Diethyl Ester (6). The title compound (6) was prepared using the same method as for preparation of 5, but starting with 4.41 g (0.01 mol) of 4. The crude product (3.4 g) was crystallized (EtOAc–hexane), giving 2.5 g (55%) of 6: mp 163–166°; ir 5.94 μ (C=O); NMR (DMSO-d_6) δ 1.29 (t, 6, J = 7.5 Hz, CH_2CH_3), 3.42 (s, 3, NCH_3), 3.9–4.59 (m, 5, CH_2CH_3 and 3-CH), 7.04 (s, 1, 6-CH), 7.41–7.84 (m, 6, aromatic).

Anal. Calcd for $C_{20}H_{21}Cl_2N_2O_4P$: C, 52.76; H, 4.65; N, 6.16; Cl, 15.58. Found: C, 52.80; H, 4.82; N, 6.31; Cl, 15.73.

[7-Chloro-5-(*o*-chlorophenyl)-2,3-dihydro-1-methyl-2-oxo-1*H*-1,4-benzodiazepin-3-yl]phosphonic Acid Methyl Ester (7). To 0.421 g (0.01 mol) of hexane-washed 57% NaH was added 20 ml of DMF, followed dropwise by 4.13 g (0.01 mol) of 3 in 40 ml of warm DMF. After the evolution of H_2 ceased, 1.4 ml (0.022 mol) of MeI was added slowly and the solution was stirred overnight at room temperature. The solution was concentrated to dryness, H_2O was added, and the solution was washed with Et_2O. The aqueous layer was acidified to pH 1.5, extracted three times with CH_2Cl_2, washed with brine, dried, and concentrated to dryness. Crystallization (MeCN) gave 1.49 g (36%) of 7: mp 171° dec; ir 5.97 μ (C=O); NMR (DMSO-d_6) δ 3.39 (s, 3, NCH_3), 3.74 (d, 3, J = 10.5 Hz, OCH_3), 4.18 (d, 1, J = 11 Hz, 3-CH), 7.06 (s, 1, 6-CH), 7.44–7.96 (m, 6, aromatic), 8.89–9.39 (broad s, 1, POH).

Anal. Calcd for $C_{17}H_{15}Cl_2N_2O_4P$: C, 49.42; H, 3.66; N, 6.78; Cl, 17.15. Found: C, 49.08; H, 3.77; N, 6.79; Cl, 17.21.

7-Chloro-5-(*o*-chlorophenyl)-1,3-dihydro-1-methyl-3-methylene-2*H*-1,4-benzodiazepin-2-one (8). A solution of 18.2 g (0.04 mol) of 6 in 100 ml of dry $(MeOCH_2)_2$ was added to a mixture of 1.70 g (0.04 mol) of hexane-washed 57% NaH in 40 ml of dry $(MeOCH_2)_2$ and the mixture was stirred at 20–30° until 970 ml (0.04 mol) of H_2 was evolved. Gaseous CH_2O, evolved from the pyrolysis (190°) of two separate batches of paraformaldehyde (1.44 g, 0.048 mol each), was passed into the solution in a stream of N_2, the first at 20° and the second at 35°. The solution was stirred for 1 hr at 25–35°, refluxed for 0.25 hr, concentrated to dryness, and, after the addition of H_2O–EtOAc, extracted with EtOAc. The organic layer was washed successively with H_2O and brine, dried, and concentrated, giving 12.8 g (mp 147–150°) of crude 8. Crystallization (MeCN) gave 6.87 g (52%) of 8: mp 163–165°; ir 6.02 μ (C=O); NMR (DMSO-d_6) δ 3.44 (s, 3, NCH_3), 5.05 (s, 1, C=CHH), 5.16 (s, 1, C=CHH), 7.0 (s, 1, 6-CH), 7.44–7.95 (m, 6, aromatic).

Anal. Calcd for $C_{17}H_{12}Cl_2N_2O$: C, 61.64; H, 3.65; N, 8.46; Cl, 21.41. Found: C, 61.80; H, 3.69; N, 8.48; Cl, 21.37.

7-Chloro-5-(*o*-chlorophenyl)-1-methyl-1*H*-1,4-benzodiazepine-2,3-dione (9). A warm solution of 13.66 g (0.03 mol) of 6 in 75 ml of warm, dry $(MeOCH_2)_2$ was added to a mixture of 1.27 g (0.03 mol) of hexane-washed 57% NaH in 30 ml of $(MeOCH_2)_2$ and the mixture was stirred at ca. 30° until H_2 ceased to be evolved (ca. 1 hr). The solution was cooled to 10–15° and O_2 was passed in through a sintered tube for ca. 1 hr. The mixture was filtered through Celite and the filtrate was poured into 1.5 l. of H_2O and extracted with ether. The solid which crystallized from the mixture was collected. Crystallization (EtOAc) gave 2.08 g (21%) of 9: mp 204–206°; ir 5.82, 5.96 μ (C=O); NMR (DMSO-d_6) δ 3.61 (s, 3, CH_3), 7.35 (s, 1, 6-CH), 7.62–8.08 (m, 6, aromatic).

Anal. Calcd for $C_{16}H_{10}Cl_2N_2O_2$: C, 57.68; H, 3.03; N, 8.41; Cl, 21.28. Found: C, 57.56; H, 2.90; N, 8.22; Cl, 21.32.

The ether extract was washed successively with H_2O and brine, dried, and concentrated, giving an additional 1.8 g (18%) of 9, mp 204–207°.

6-Chloro-4-(*o*-chlorophenyl)-2-quinazolinecarboxylic Acid (10). A solution of 13.24 g (0.03 mol) of 4 in 75 ml of DMF was added to a mixture of 2.53 g (0.06 mol) of hexane-washed 57% NaH in 30 ml of DMF and the mixture was stirred for 2 hr at 5–10°. Dry O_2 was passed into the resulting solution through a sintered tube for 0.5 hr while the temperature was raised to 25°. The solution was poured into 2 l. of water and filtered, and 25 ml of HOAc was added. The residue was extracted with EtOAc, washed successively

with H_2O and brine, and dried. Concentration and crystallization (MeCN) gave 3.2 g (33%) of **10**: mp 218–219° dec; ir 5.83 μ (C=O); NMR (DMSO-d_6) δ 7.5–7.85 (m, 5, aromatic), 8.18–8.35 (m, 3, aromatic plus exchangeable CO_2H).

Anal. Calcd for $C_{15}H_8Cl_2N_2O_2$: C, 56.45; H, 2.52; N, 8.78; Cl, 22.22. Found: C, 56.55; H, 2.77; N, 8.89; Cl, 22.41.

7-Chloro-5-(*o*-chlorophenyl)-1*H*-1,4-benzodiazepine-2,3-dione (11). A solution of 13.24 g (0.03 mol) of **4** in 60 ml of DMF was added to a mixture of 2.54 g (0.06 mol) of hexane-washed 57% NaH in 30 ml of DMF and the mixture was stirred for 1 hr at 20°. Dry O_2 was passed into the solution through a sintered tube at 20–30° for 1.5 hr, 3.8 ml (0.03 mol) of Me_3SiCl was added, and the solution was stirred for 0.25 hr. The mixture was concentrated to dryness at 40° and water and EtOAc were added. The residue was extracted twice with EtOAc. The organic layer was washed successively with H_2O and brine and dried, giving 5.73 g (mp 250° dec) of crude **11** after concentration. Crystallization (MeCN) gave 4.1 g (43%) of **11**: mp 258° dec; ir 5.77, 6.00 μ (C=O); NMR (DMSO-d_6) δ 7.18 (d, 1, J = 2 Hz, 6-CH), 7.32–7.91 (m, 7, aromatic and exchangeable NH).

Anal. Calcd for $C_{15}H_8Cl_2N_2O_2$: C, 56.45; H, 2.52; N, 8.78; Cl, 22.22. Found: C, 56.55; H, 2.62; N, 9.15; Cl, 22.53.

7-Chloro-5-(*o*-chlorophenyl)-1,3-dihydro-3-methylene-2*H*-1,4-benzodiazepin-2-one (12). A solution of 4.41 g (0.01 mol) of **4** in 75 ml of dry $(MeOCH_2)_2$ was added to a mixture of 0.85 g (0.02 mol) of hexane-washed 57% NaH in 10 ml of dry $(MeOCH_2)_2$ and the mixture was stirred at 30–40° until H_2 ceased to be evolved. Gaseous CH_2O from the pyrolysis (190°) of 1 g (0.033 mol) of paraformaldehyde was passed into the solution at 35–42° in a stream of N_2. The solution was stirred for 1 hr at room temperature, refluxed for 0.5 hr, concentrated to dryness, and, after the addition of H_2O–EtOAc, extracted three times with EtOAc. The organic layer was washed successively with H_2O and brine, dried, and concentrated, giving 3 g of crude **12**. The solid was chromatographed on 100 g of silica gel, starting with $CHCl_3$. The desired product was removed with 10% v/v Et_2O in $CHCl_3$ and was crystallized (MeCN), giving 2.0 g (63%) of **12**: mp 200–202° dec; NMR (DMSO-d_6) δ 5.18 (s, 1, C=CHH), 5.39 (s, 1, C=CHH), 6.96 (d, 1, J = 1.5 Hz, 6-CH), 7.25–7.91 (m, 6, aromatic), 11.12 (s, 1, NH).

Anal. Calcd for $C_{16}H_{10}Cl_2N_2O$: C, 60.59; H, 3.18; N, 8.81; Cl, 22.35. Found: C, 60.65; H, 3.46; N, 9.20; Cl, 22.43.

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Registry No.—**1**, 846-49-1; **3**, 54643-73-1; **4**, 54677-79-1; **5**, 54643-74-2; **6**, 54643-75-3; **7**, 54643-76-4; **8**, 54643-77-5; **9**, 54643-78-6; **10**, 54643-79-7; **11**, 54643-80-0; **12**, 54643-81-1.

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Preparation of Oxathiapentadecanes[1]

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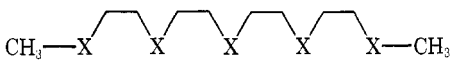
Our interest in the synthesis and complexation of cations by cyclic polyether sulfides[2–5] has led us to prepare a series of oxathiapentadecanes. These compounds are of interest because they are polydentate chelates with unique and unusual coordination properties. Such a series of related compounds may help determine how and where coordination to various cations takes place. Indeed, coordination with silver(I) and mercury(II) by these compounds appears to show definite structural features. These properties will be reported elsewhere.[5] This report deals only with the synthesis and properties of the oxathiapentadecanes.

Table I
Yield and Physical Properties of the Oxathiapentadecanes



Compd	Position of Heteroatoms 2	5	8	11	14	Yield, %	Bp, °C (0.1 mm) (mp, °C)
1	O	O	S	O	O	67	120–121 (0.1)
2	S	O	O	O	S	75	133–134 (0.1)
3	O	S	O	S	O	70	137–138 (0.1)
4	O	S	S	S	O	75	(56)
5	S	S	O	S	S	78	(36–37)[a]
6	S	S	S	S	S	89	(87–88)[b]

[a] Lit.[12] mp 37°. [b] Lit.[12] mp 88°.

The dimethyl ethers of the polyethylene glycols (often called glymes) have been prepared using the Williamson synthesis from the polyethylene glycols and alkyl halides or sulfates.[6–8] Chakhovskoy and coworkers have prepared certain glymes using alkyl tosylates which gave better yields than the halides.[9] The oxathiapentadecanes (see Table I) were prepared in a similar manner from the reaction of a mercaptan or sodium sulfide and an alkyl halide in basic media. These reactions are easier to perform than a Williamson synthesis using an alkoxide and an alkyl halide, since they require less severe conditions.[10,11] In addition, better yields are obtained. We tried to use compounds other than sulfur vesicants (blister-producing mustards) for these syntheses. Only one such compound was used (2-chloroethyl methyl sulfide in the preparation of **5**). In our synthesis of compound **1**, 1-(2-chloroethoxy)-2-methoxyethane was treated with sodium sulfide while compound **4** was prepared from bis(2-mercaptoethyl) sulfide and 2-bromoethyl methyl ether. The other compounds were prepared in a similar manner.

$$CH_3OCH_2CH_2OCH_2CH_2Cl + Na_2S \longrightarrow \mathbf{1}$$

$$HSCH_2CH_2SCH_2CH_2SH + 2BrCH_2CH_2OCH_3 \longrightarrow \mathbf{4}$$

Compounds **5** and **6** as well as other similar compounds have been prepared from the corresponding β-chloro sulfides (mustard compounds).[12–14] Meade and Moggridge[12] prepared **5** and **6** from the reaction of methyl mercaptan with 2,2′-(2-chloroethylthia)diethyl ether (**7**, X = O) and the corresponding sulfide (**7**, X = S), respectively. Williams and Woodward prepared similar compounds from **7** (X = S) using aromatic oxides and sulfides.[13] The bis(*n*-propoxyethylmercaptoethyl) ether (the di-*n*-propyl ether similar to **3**) was prepared[14] by treating the corresponding glycol with *n*-propyl alcohol in acid media.

$$X(CH_2CH_2SCH_2CH_2Cl)_2 + CH_3SH \xrightarrow{\text{base}} \mathbf{5}\ (X = O)\ \text{or}\ \mathbf{6}\ (X = S)$$

The nuclear magnetic resonance (NMR) spectra for the oxathiapentadecanes are similar to those observed for the