0.08 mm) afforded 1.040 g (93%) of dodecanal bromo ketal as a colorless oil: infrared peaks (film) at 8.75 and 8.90 μ; nmr $\delta_{TMS}^{CDCl_3}$ 4.99 (HCOO, multiplet), 4.58–3.17 [OCH(CH_2Br)CH_2O, complex], and 0.89 ppm (CH_3, triplet, J = 4.0 Hz); mass spectrum calcd for $C_{15}H_{29}O_2Br$ 320.1350; found 320.1342.

Deketalization.—A solution of 162 mg (0.505 mmol) of dodecanal bromo ketal and 500 mg of zinc dust in 10 ml of methanol was heated at reflux under argon for 12 hr. The zinc was removed by filtration, and the product was isolated by ether extraction and evaporatively distilled (bp 80°, 0.07 mm), yielding 83 mg (89%) of dodecanal, identical with an authentic sample. The zinc was activated by brief treatment with acetic acid followed by washing with methanol.

The efficiency of introduction and removal of the bromomethylethylene ketal group, its stability toward many synthetic reagents, and the selectivity with which it can be removed[6] all indicate a real utility in synthesis.[7]

Registry No.—IV, 4704-77-2; V, 98-53-3; V bromo ketal, 37447-43-1; VI, 22348-95-4; VI bromo ketal, 37447-45-3; VII, 112-54-9; VII bromo ketal, 37447-47-5.

(6) For controlled potential electrolytic removal of such groups, see M. F. Semmelhack and G. E. Heinsohn, *J. Amer. Chem. Soc.*, **94**, 5140 (1972).

(7) This research was financially assisted in part by the National Institutes of Health and the National Science Foundation.

Acid Hydrolysis Products of DDD and DDT Precursors[1]

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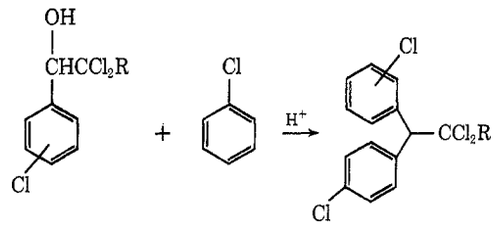
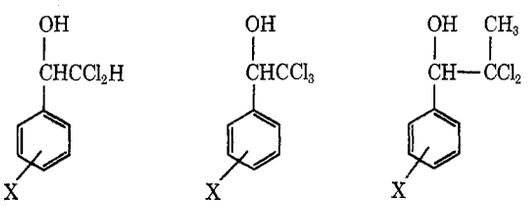
Received September 8, 1972

The widespread use of 2,2-di(*p*-chlorophenyl)-1,1,1-trichloroethane (DDT) as a pesticide as well as the more limited use of 2-(*o*-chlorophenyl)-2-(*p*-chlorophenyl)-1,1-dichloroethane (*o,p'*-DDD) as the only clinically approved agent for the treatment of adrenocortical carcinoma has prompted numerous chemical investigations in these classes of compounds.[3-13] Studies in our laboratories have been devoted to (1) preparing derivatives of *o,p'*-DDD which lack the serious toxicity of this drug and (2) studying the chemistry of the precursors used in the synthesis of these derivatives.

The conventional and most direct method for the preparation of DDD derivatives involves the acid-catalyzed condensation of 1-(chlorophenyl)-2,2-di-



chloroethanols with chlorobenzene.[3,8] This method is commonly employed, since usable amounts of DDD's are obtained from readily available starting materials. However, the yields are generally rather poor (*ca.* 30–50%).[3,8] An investigation of the products and by-products in this reaction was, therefore, undertaken to aid in delineating the scope and limitations of this reaction. Moreover, ramifications of this work can be extended to the synthesis and hydrolysis of DDT and its precursors. This paper reports the products formed when phenyldichloroethanols **1**, phenyltrichloroethanols **2**, and phenyldichloropropanols **3** are subjected to concentrated sulfuric acid,[14] conditions normally employed for the synthesis of DDT[3] and DDD.[8]



1a, X = *o*-Cl; **b**, X = *m*-Cl; **c**, X = *p*-Cl

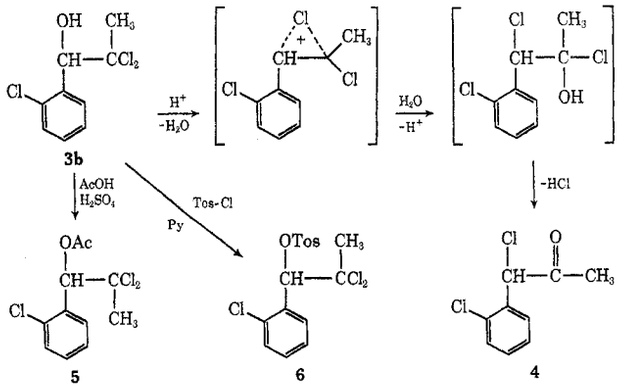
2a, X = H; **b**, X = *o*-Cl

3a, X = H; **b**, X = *o*-Cl; **c**, X = *p*-Cl

Glc–mass spectral analysis and infrared and nuclear magnetic resonance spectroscopy were employed for characterization of the products.

Results and Discussion

Unexpected results were obtained in the acid hydrolysis of the phenyldichloropropanols **3**. Treatment of 2,2-dichloro-1-(*o*-chlorophenyl)propanol (**3b**) with concentrated sulfuric acid at 40–45° for 3 hr afforded, exclusively, 1-(*o*-chlorophenyl)-1-chloropropanone (**4**). One possible means for such a conversion can be explained as proceeding through a chloronium ion as shown below.



(1) This investigation was supported by the U. S. Public Health Service under Research Grants No. CA-49340 and CA-08349 from the National Cancer Institute.

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(10) R. E. Counsell, V. V. Ranade, L. K. Lala, and B. H. Hong, *J. Med. Chem.*, **11**, 380 (1968).

(11) R. E. Counsell, V. V. Ranade, P. Pocha, R. E. Willette, and W. Diguilo, *J. Pharm. Sci.*, **57**, 1657 (1968).

(12) A. B. Sakla, W. Tadros, and A. A. A. Helmy, *J. Chem. Soc. C*, 1044 (1969).

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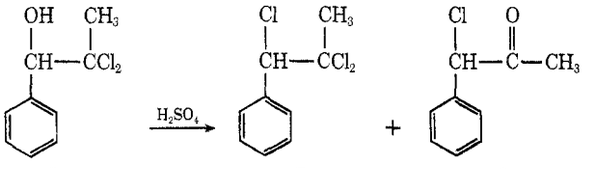
(14) A recent report by P. B. Blumbergs and M. P. LaMontagne, *J. Org. Chem.*, **37**, 1248 (1972), has shown that phenyldichloromethylcarbinols on hydrolysis with potassium carbonate afford α-hydroxyaldehydes.

By following this rearrangement using an acidified nmr sample, it was shown that the small amount of water formed by the protonation of the alcohol in acid is sufficient for the conversion to the ketone **4** even before aqueous work-up conditions are employed. The rearrangement occurs so rapidly, in fact, that attempted condensations with chlorobenzene in sulfuric acid failed completely and only **4** was formed. Normal esterification methods were, on the other hand, completely successful using acetic acid–sulfuric acid and *p*-toluenesulfonyl chloride–pyridine. The structures of the esters **5** and **6** were confirmed by their mass spectra, which showed distinct loss of Cl_2CCH_3.

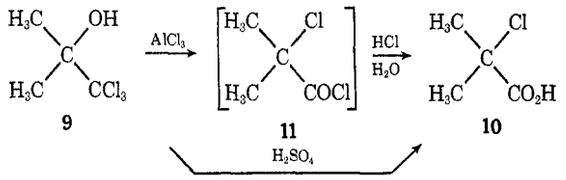
When 2,2-dichloro-1-phenylpropanol (**3a**) was treated under identical conditions, two compounds were formed. Separation of these compounds by preparative glc afforded materials with virtually identical nmr spectra. The nmr spectrum of the less volatile component showed a five-proton multiplet at τ 2.48 assigned to aromatic protons, a single-proton singlet at 4.70 assigned to a benzylic proton, and a three-proton singlet at 7.74 assigned to a methyl group. Its infrared spectrum lacked both OH and C=O stretching bands. Mass spectrometry showed molecular ions at *m/e* 222, 224, and 226 in the correct ratio for three chlorine atoms. The structure of this compound was assigned as 1-phenyl-1,2,2-trichloropropane (**7**). The nmr spectrum of the more volatile component showed a five-proton singlet at τ 2.55 assigned to aromatic protons, a single-proton singlet at 4.64 assigned to a benzylic proton, and a three-proton singlet at 7.80 assigned to protons. Its infrared spectrum showed a very intense C=O stretching band at 1715 cm^{-1}. The mass spectrum of this component showed molecular ions at *m/e* 168 and 170 in the correct ratio for one chlorine atom. This information was consistent for 1-chloro-1-phenyl-2-propanone (**8**).



3a **7** **8**

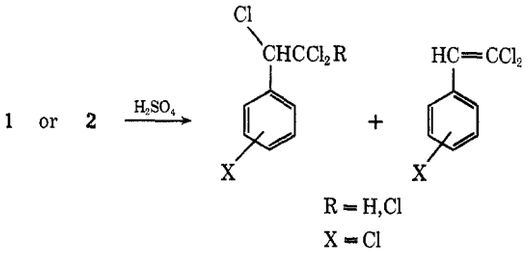
During the course of this work, a literature search revealed that Kundiger and Pledger[15] had observed a related product resulting from attempted Friedel–Crafts condensations with 1,1,1-trichloro-2-methyl-2-propanol (**9**) (Chloretone). In the presence of aluminum chloride, **9** was shown to afford only α-chloroisobutyric acid (**10**) after a work-up procedure employing aqueous hydrochloric acid. These workers assumed that **10** was formed *via* α-chloroisobutyroyl chloride (**11**). Under the conditions of this reaction it would be impossible, however, to postulate what the true intermediate species would be and by what means it was formed, since several sources of chloride ion were present. Moreover, our further examination of this reaction showed that concentrated sulfuric acid afforded the same α-chloro acid **10** in quantitative yield. Under these conditions, the only source of chloride ion was 1,1,1-trichloro-2-methyl-2-propanol (**9**).

(15) D. G. Kundiger and H. Pledger, Jr., *J. Amer. Chem. Soc.*, **78**, 6098 (1956).



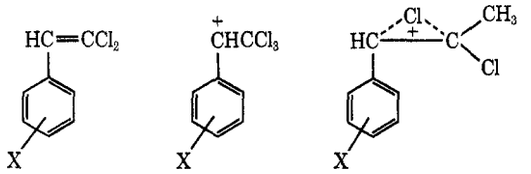
9 **11** **10**

In contrast to the above results, acid hydrolysis of **1** and **2** afforded no oxidative rearrangement carbonyl products. In general, the two types of products which predominated were those formed by the replacement of the hydroxyl group by a halogen and/or the acid-catalyzed elimination of the elements of HOX.[16]



Summary

From the products obtained, it appears as though three different intermediates are involved in the acid-catalyzed syntheses of DDD and DDT. Schriesheim[17] has pointed out that phenyldichloroethanols **1** probably proceed through the olefin **12** *via* elimination of water. This olefin is the true electrophile in the reaction with chlorobenzene. On the other hand, phenyltrichloroethanols **2** form stable carbonium ions **13** when treated with acid.[18] The carbonium ions **13** are the reacting species in the condensation with chlorobenzene. Phenyldichloropropanols **3** rearrange in acid *via* chloronium ion **14** before condensation products form. In virtually every case, chloride ion resulting from liberated HCl or from disproportionation reactions reacts with the alcohol to afford benzylic chlorinated derivatives. A summary of the hydrolysis products is listed in Tables I, II, and III.



12 **13** **14**

Experimental Section

General.—Melting points were obtained with a Fisher-Johns melting point apparatus and are uncorrected. Boiling points are uncorrected. The nuclear magnetic resonance (nmr) spectra were recorded on a Varian A-60A spectrometer in $CDCl_3$ with tetramethylsilane as an internal standard. Infrared spectra were determined on a Perkin-Elmer Model 337 spectrophotometer. Glc–mass spectral analyses were performed on a Loenco Model 160 gas chromatograph using a 6 ft × 0.125 in. stainless steel column packed with 3% OV-17 on 100/120 mesh Chromo-

(16) J. W. Cornforth, R. H. Cornforth, and K. K. Mathew, *J. Chem. Soc.*, 112 (1959).

(17) A. Schriesheim, "Friedel–Crafts and Related Reactions," Vol. II, G. A. Olah, Ed., Interscience, New York, N. Y., 1964, p 480.

(18) D. Bethell and V. Gold, "Carbonium Ions. An Introduction," Academic Press, New York, N. Y., 1967, p 117.

TABLE I
HYDROLYSIS OF PHENYLDICHLOROETHANOLS 1a–c

Alcohol	Major products	Other products
1a	o-$ClC_6H_4CH{=}CCl_2$ *m/e* 206 (3 Cl)	Starting material
1b	m-$ClC_6H_4CHClCHCl_2$ *m/e* 242 (4 Cl) + m-$ClC_6H_4CH{=}CCl_2$ *m/e* 206 (3 Cl)	
1c	p-$ClC_6H_4CHClCHCl_2$ *m/e* 242 (4 Cl) + p-$ClC_6H_4CH{=}CCl_2$ *m/e* 206 (3 Cl)	Starting material

TABLE II
HYDROLYSIS OF PHENYLTRICHLOROETHANOLS 2a–b

Alcohol	Major products	Other products
2a	$C_6H_5CHClCCl_3$ *m/e* 242 (4 Cl)	Unidentified solids
2b	o-$ClC_6H_4CHClCCl_3$ *m/e* 276 (5 Cl) + o-$ClC_6H_4CH{=}CCl_2$ *m/e* 206 (3 Cl) + o-$ClC_6H_4CCl{=}CCl_2$ *m/e* 240 (4 Cl)	o-ClC_6H_4CHO *m/e* 139 (1 Cl)

TABLE III
HYDROLYSIS OF PHENYLDICHLOROPROPANOLS 3a–c

Alcohol	Major products	Other products
3a	$C_6H_5CHClC(=O)CH_3$ *m/e* 168 (1 Cl) + $C_6H_5CHClCCl_2CH_3$ *m/e* 222 (3 Cl)	
3b	o-$ClC_6H_4CHClC(=O)CH_3$ *m/e* 202 (2 Cl)	
3c	p-$ClC_6H_4CHClC(=O)CH_3$ *m/e* 202 (2 Cl) + p-$ClC_6H_4CHClCCl_2CH_3$ *m/e* 256 (4 Cl)	p-$ClC_6H_4CO_2H$ *m/e* 156 (1 Cl)

sorb G and interfaced with a Du Pont Model 21-490 mass spectrometer. Microanalyses were performed by Spang Microanalytical Laboratories, Ann Arbor, Mich., and Midwest Microlab, Indianapolis, Ind.

Materials. Preparation of Phenyldichloroethanols (1a–c).—These compounds were prepared as previously described by Counsell and Willette.[8]

Preparation of Phenyltrichloroethanols (2a–b).—These compounds were prepared by the method of Bergmann, *et al.*[19]

Preparation of Phenyldichloropropanols (3a–c).—The general method is illustrated by the preparation of 2,2-dichloro-1-(*o*-chlorophenyl)propanol (**3b**).

Dry chlorine gas was bubbled slowly through a solution of *o*-chloropropiophenone[20] (19.7 g, 0.117 mol) in glacial acetic acid (150 ml) for 2 hr at 110°. The solution was flushed with nitrogen for 15 min and anhydrous sodium acetate (25.0 g) was added. Chlorine gas was again bubbled through the solution at 110° for an additional 5 hr. The cooled reaction mixture was poured into ice water (2 l.) saturated with NaCl and containing $NaHSO_3$ (5.0 g) and extracted with ether. The ethereal extract was dried (Na_2SO_4), concentrated, and distilled, giving 22.0 g (79%) of α,α-dichloro-*o*-chloropropiophenone: bp 58–59° (0.75 mm); ir (film) 1720 cm^{-1} (C=O); nmr ($CDCl_3$) τ 2.50 (m, 4, ArH) and 7.67 (m, 3, CH_3).

Anal. Calcd for $C_9H_7Cl_3O$: C, 45.51; H, 2.97. Found: C, 45.40; H, 2.88.

Over a period of 30 min, $NaBH_4$ (4.75 g, 0.126 mol) was added portionwise to a solution of α,α-dichloro-*o*-chloropropiophenone (13.1 g, 0.055 mol) in methanol (150 ml) at ice-bath temperature. Stirring was continued for 1.5 hr in the cold and for an additional 2.5 hr at room temperature. The reaction mixture was poured into ice water (1.5 l.) containing NH_4Cl (50 g), saturated with NaCl, and extracted with ether. The ethereal extract was dried (Na_2SO_4), concentrated, and distilled, yielding 11.8 g (89%) of **3b**: bp 79–81° (0.2 mm); mp 68–69° (CCl_4–hexane); ir ($CHCl_3$) 3550 cm^{-1} (OH); nmr ($CDCl_3$) τ 2.45 (m, 4, ArH), 4.34 (d, 1, CH), 6.87 (d, 1, OH), and 7.87 (s, 3, CH_3).

Anal. Calcd for $C_9H_9Cl_3O$: C, 45.12; H, 3.78. Found: C, 45.26; H, 3.92.

2,2-Dichloro-1-phenylpropanol (3a) had bp 72–76° (0.3 mm) [lit.[21] bp 85–86° (1.0 mm)]; ir (film) 3475 cm^{-1} (OH); nmr ($CDCl_3$) τ 2.55 (m, 5, ArH), 5.00 (d, 1, CH), 6.88 (d, 1, OH), and 7.95 (s, 3, CH_3).

2,2-Dichloro-1-(*p*-chlorophenyl)propanol (3c) had bp 128–129° (1.0 mm); mp 59–60° [petroleum ether (bp 30–60°)]; ir (film) 3550 cm^{-1} (OH); nmr ($CDCl_3$) τ 2.55 (m, 4, ArH), 5.02 (d, 1, CH), 6.80 (d, 1, OH), and 7.99 (d, 3, CH_3).

Anal. Calcd for $C_9H_9Cl_3O$: C, 45.12; H, 3.78. Found: C, 45.35; H, 3.79.

Product Study.—A 1.0-g sample of an alcohol was treated at 40–45° with 5 ml of concentrated sulfuric acid. After 3 hr, the mixture was poured into ice water and extracted with three 100-ml portions of ether. The ethereal extract was dried over Na_2SO_4 and concentrated *in vacuo* to leave a yellowish residue. Glc–mass spectral analyses were performed on a Loenco Model 160 (flame ionization detector) gas chromatograph interfaced with a Du Pont Model 21-490 mass spectrometer. The results are tabulated in Tables I, II, and III.

Preparation of 1-(*o*-Chlorophenyl)-1-chloropropanone (4).—At 40–45° concentrated sulfuric acid (30 ml) was added dropwise with stirring to **3b** (12.8 g, 0.054 mol). After 4 hr, the mixture was poured into ice water (400 ml) and extracted with ether. The ethereal extract was washed with water and dried (Na_2SO_4). Evaporation of the solvent left a residue which was distilled, yielding 8.0 g (74%) of **4**: bp 82–85° (0.4 mm); ir (film) 1730 cm^{-1} (C=O); nmr ($CDCl_3$) τ 2.60 (m, 4, ArH), 4.08 (s, 1, CH), and 7.74 (s, 3, CH_3); mass spectrum *m/e* 202 (M^+), 167 (M^+ − 35), 159 (M^+ − 43), 125 (M^+ − 77), 103 (M^+ − 99), and 89 (M^+ − 113).

Preparation of α-Chloroisobutyric Acid (10).—A mixture of 1,1,1-trichloro-2-methyl-2-propanol[22] (5.0 g, 0.028 mol) and concentrated sulfuric acid (15 ml) was heated at 40–45° for 3 hr. The mixture was poured into ice water and extracted with ether.

(19) E. D. Bergmann, D. Ginsburg, and D. Lavie, *J. Amer. Chem. Soc.*, **72**, 5012 (1950).

(20) V. L. Szotyory and E. Hamburg, *J. Prakt. Chem.*, **23**, 208 (1963).

(21) M. Ballester and J. Riera, *An. Real Soc. Espan. Fis. Quim.*, **56**, 897 (1960); *Chem. Abstr.*, **55**, 19876 (1961).

(22) C. Weizmann, E. Bergmann, and M. Sulzbacker, *J. Amer. Chem. Soc.*, **70**, 1189 (1948).

The ethereal extract was washed with water before drying over Na_2SO_4. The solvent was concentrated under vacuum and the residue was distilled, yielding 3.4 g (100%) of **10**: bp 38–39° (0.2 mm) [lit.[15] bp 51–52° (1.0 mm)]; ir (CCl_4) 3500–3200 (OH) and 1710 cm^{-1} (C=O); nmr ($CDCl_3$) τ −2.36 (s, 1, CO_2H) and 9.16 (s, 6, 2 CH_3).

Preparation of 2,2-Dichloro-1-acetoxy-1-(*o*-chlorophenyl)propane (5).—A stirred solution of **3b** (2.0 g, 0.0084 mol) in glacial acetic acid (100 ml) and concentrated sulfuric acid (5 ml) was heated at 45° for 3 hr. The solution was poured into ice water (800 ml) and extracted with ether. The combined extracts were washed with 10% $NaHCO_3$ and water before drying over Na_2SO_4. Removal of the solvent left a residue which recrystallized from methanol to afford 1.5 g (64%) of **5** as colorless crystals: mp 86–87°; ir (KBr) 1745 cm^{-1} (C=O); nmr ($CDCl_3$) τ 2.43 (m, 4, ArH), 3.24 (s, 1, CH), 7.83 (s, 3, CH_3), and 7.85 (s, 3, CH_3); mass spectrum *m/e* 280 (M^+), 245 ($M^+ - 35$), 183 ($M^+ - 97 - Cl_2CCH_3$), and 141 ($M^+ - 139$).

Anal. Calcd for $C_{11}H_{11}Cl_3O_2$: C, 46.92; H, 3.94. Found: C, 47.12; H, 4.19.

Preparation of 2,2-Dichloro-1-tosyloxy-1-(*o*-chlorophenyl)propane (6).—A solution of **3b** (1.2 g, 0.005 mol) and *p*-toluenesulfonyl chloride (1.6 g, 0.008 mol) in pyridine (50 ml) was stirred for 5 days at room temperature. The solution was poured into ice-cold 10% HCl (500 ml) and the product was extracted into ether. The ethereal solution was washed with water and 5% KOH before drying over Na_2SO_4. Removal of the solvent afforded **6** as an oil which crystallized from hexane–petroleum ether (bp 30–60°) as colorless crystals (1.0 g, 68%): mp 88–89°; nmr ($CDCl_3$) τ 2.55 (m, 8, ArH), 3.75 (s, 1, CH), 7.64 (s, 3, CH_3), and 7.83 (s, 3, CH_3); mass spectrum *m/e* 392 (M^+), 295 ($M^+ - 97$- Cl_2CCH_3), 186 ($M^+ - 106$), 155 ($M^+ - 237$), 115 ($M^+ - 277$), and 91 ($M^+ - 301$).

Registry No.—**3b**, 35996-56-6; **3c**, 37610-56-3; **4**, 37610-57-4; **5**, 37610-58-5; **6**, 37610-59-6; DDD, 72-54-8; DDT, 50-29-3; *o*-chloropropiophenone, 6323-18-8; α,α-dichloro-*o*-chloropropiophenone, 35996-45-3.

Addition of Simple Siloxanes to β-Methallyl Chloride[1]

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Received June 26, 1972

The importance of silicone surfactants prompted this investigation of the catalyzed addition of small siloxane units to β-methallyl chloride. The resulting adducts may serve as intermediates for surfactant synthesis.

Three types of products were observed from the reaction of siloxanes **1a–c** with β-methallyl chloride:

$$\underset{\textbf{1a–c}}{\equiv SiH} + CH_2{=}C(CH_3)CH_2Cl \longrightarrow \underset{\textbf{2a–c}}{\equiv SiCH_2CH(CH_3)CH_2Cl} + \underset{\textbf{3a–c}}{\equiv SiCl} + \underset{\textbf{4b,c}}{\equiv SiCH_2C(CH_3){=}CH_2}$$

a, $Me_3SiOSiMe_2H$
b, $(Me_3SiO)_2SiMeH$
c, $(Me_3SiO)_3SiH$

(1) Reported in part at the 163rd National Meeting of the American Chemical Society, Boston, Mass., April 1972.

(2) National Science Foundation Undergraduate Research Participant, 1971.

(3) (a) Chemistry Department, Drexel University; (b) Research Department, National Foam System, Inc.

adducts (**2a–c**), rearrangement products (**3a–c**), and methallylsiloxanes (**4b,c**).

β-Methallyl chloride, a siloxane, and catalyst were heated at reflux for an appropriate time. Products were identified and yields were determined by comparison with authentic samples using gas chromatography. These results are summarized in Table I.

TABLE I
SILOXANE ADDITION TO β-METHALLYL CHLORIDE

Siloxane	Catalyst	Reaction time, hr	Products (yield, %)
1a	H_2PtCl_6	2	**2a** (97)
1b	H_2PtCl_6	1	**2b** (34), **3b** (18), **4b** (9)
1b	Pt/C	72	**2b** (30), **3b** (18), **4b** (10)
1c	H_2PtCl_6	1.5	**2c** (14), **3c** (42), **4c** (23)
1a	Pd/C	Immediate	**3a** (96)
1b	Pd/C	100	**3b** (74)
1b[a]	$PdCl_2$	Immediate	**3b** (100)
1b	Ru/C	4	**3b** (60)
1c	Pd/C	48	**3c** (24)
1b	None	48	No conversion

[a] Reaction in the absence of β-methallyl chloride.

Structural proof of the adducts, **2a–c**, was based on their ir, nmr, mass spectra, and elemental analysis. The mass spectra of **2a–c** failed to show molecular ions, but did show predominant fragments at *m/e* 73 (Me_3Si^+) and $M - CH_2CH(CH_3)CH_2Cl$. The nmr spectra of **2a–c** are in full accord with the proposed structures and are summarized in Table II.

The structures of the chlorosilanes, **3a–c**, were based upon elemental analysis, mass spectra, nmr, and infrared spectra. The mass spectra of **3a–c** also failed to show molecular ions, but did have fragments at $M - CH_3$ and $M - Cl$. A predominant fragment corresponding to Me_3Si was observed for all the chlorosilanes. The nmr spectra of **3a–c** were similar to those of the corresponding siloxanes **1a–c**, and the infrared spectra of **3a–c** showed the absence of Si–H stretching at 2200 cm^{-1}.[4] Rapid hydrolysis of **3a–c** to the corresponding silanols with evolution of hydrogen chloride further supports the presence of the SiCl bond.

Methallylsiloxanes (**4b,c**) could not be purified by distillation, but were satisfactorily separated by preparative gas chromatography. Elemental analysis, ir, nmr, and mass spectra of these compounds were fully in accord with the proposed structure. Their ir spectra indicated an allylic structure[5] with absorption at 1635 and 1250 cm^{-1}. The nmr spectra are similar to those reported by Egorochkin, *et al.*, for analogous compounds.[6] The mass spectra of **4c** indicated a molecular ion at *m/e* 350 and predominant fragments at NCH_3 and $M - C_4H_7$ and for Me_3Si.

Two additional synthetic routes to siloxanes **2a–c** were explored. First, the reaction of **5**[7] with pyridine, followed by the addition of trimethylsilanol, gave a 20% yield of **2b**. The second route involved the cohydrolysis of **5** with trimethylchlorosilane in water to give a 32% yield of **2b**. The most convenient

(4) R. T. Conley, "Infrared Spectroscopy," Allyn and Bacon, Boston, Mass., 1966, Chapter 5.

(5) R. J. H. Voorhoeve, "Organohalosilanes: Precursors to Silicones," Elsevier, Amsterdam, 1967, p 372.

(6) A. N. Egorochkin, M. L. Khidekel, G. A. Razuvaev, G. G. Petukhov, and V. F. Mironov, *Izv. Akad. Nauk SSSR, Ser. Khim.*, 1521 (1964).

(7) J. W. Ryan, G. K. Menzie, and J. L. Speier, *J. Amer. Chem. Soc.*, **82**, 3601 (1960).