TABLE I

Sample	A	B	C	D	E
η_{sp}/c, c = 1.00% in 0.50 *N* NaCl, 30°	0.328	0.358	0.370	0.200	0.380
S, %	16.17	16.74	14.28	14.27	15.99
N (Kjeldahl), %	3.75	3.44	3.70	3.12	3.15
N (Van Slyke), %	1.02	0.25	1.09	0.19	0.17
Moles of $-SO_3Na$ group per repeating unit					
Total	1.68	1.80	1.32	1.32	1.64
on OH	0.95	0.87	0.61	0.38	0.69
on NH_2	0.73	0.93	0.71	0.94	0.95
Activity, I.U./mg.	13	59	11	57	57

that, other conditions being equal, the contribution of sulfamic acid groups (above a certain limit) to the anticoagulant activity of polysaccharide polysulfate esters is far greater than that of sulfate ester groups.

That there is no simple relationship, *per se*, between anticoagulant activity and acute toxicity is amply demonstrated by other of our sulfated chitosan products, shown in Table II.

TABLE II

Sample	F	G	H	I
Activity, I.U./mg.	20	57	63	74
LD_{50}, i.v. in mice, mg. of kg.	3250 ±250	1500 ±500	1700 ±300	1250 ±250

In consideration of reports that the U.S.P. method does not give a reliable measure of *in vivo* activity with polysaccharide polysulfate synthetic anticoagulants,[6,7,8] our products which have been assayed by the U.S.P. method are currently being evaluated for activity by *in vivo* methods.

(6) E. G. Snyder (to Wyeth Inc.), U. S. Patent 2,508,433, May 23, 1950.

(7) C. N. Mangieri, R. Engelberg and L. O. Randall, *J. Pharmacol. Exptl. Therap.*, **102**, 156 (1951).

(8) H. E. Stavely, P. J. Baker, Jr., and H. G. Payne, *Federation Proc.*, **11**, 488 (1952).

WARNER-CHILCOTT RESEARCH LABORATORIES JOHN DOCZI
113 WEST 18TH STREET ALEX FISCHMAN
NEW YORK 11, NEW YORK JOHN A. KING

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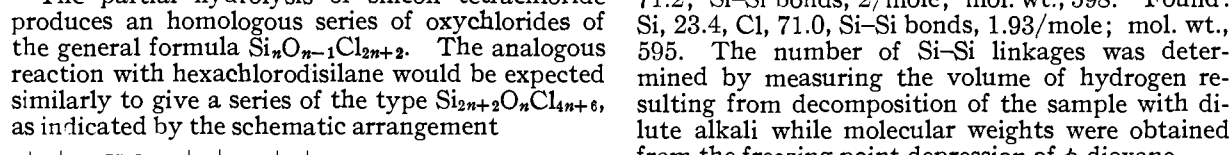
THE PARTIAL HYDROLYSIS OF HEXACHLORODISILANE

Sir:

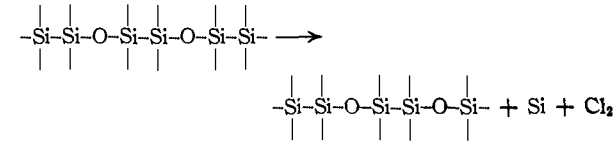
Part of the current research program of these laboratories involves a comparative study of the partial hydrolysis, ammonolysis and thiohydrolysis of silicon halides. Using the method of Schumb and Stevens,[1] we have succeeded in partially hydrolyzing hexachlorodisilane, obtaining the first member of what is believed may be a new series of silicon oxychlorides.

The partial hydrolysis of silicon tetrachloride produces an homologous series of oxychlorides of the general formula $Si_nO_{n-1}Cl_{2n+2}$. The analogous reaction with hexachlorodisilane would be expected similarly to give a series of the type $Si_{2n+2}O_nCl_{4n+6}$, as indicated by the schematic arrangement



(1) W. C. Schumb and A. J. Stevens, THIS JOURNAL, **72**, 3178 (1950).

The first member of this series, Si_4OCl_{10}, has been isolated and identified. Evidence also has been obtained for the existence of higher members, which, however, appear to undergo thermal decomposition during fractional distillation. This decomposition is probably the result of the thermal instability of long chains containing Si–Si linkages and has prevented the isolation of higher members. Analyses and estimated molecular weights of higher boiling fractions, however, are in the expected region. One of these fractions appears to be a decomposition product of the second member of the series referred to above, by such a process as the following



The hexachlorodisilane was added to dry ether in a three-necked round-bottom flask fitted with a slip-seal stirrer. The solution was cooled to −78° in a solid carbon dioxide–trichloroethylene bath and, with constant stirring, a measured amount of water was added dropwise from a buret. The mixture remained in the cold-bath for two hours and was then allowed to come to room temperature. The ether and unreacted hexachlorodisilane were removed by fractional distillation. The partial hydrolysis was then repeated with the recovered hexachlorodisilane. In a series of six such reactions, a total of 230 g. of Si_2Cl_6 was partially hydrolyzed.

The higher boiling residues remaining after the removal of the ether and unreacted hexachlorodisilane were combined and fractionated under reduced pressure. During the fractionation the contents of the distillation pot darkened gradually and a considerable quantity of black residue remained after removal of all liquid material. All fractions were clear liquids, increasing in viscosity with increasing temperature. While the first two fractions exhibited relatively narrow boiling point ranges, no well defined holds were observed at higher temperatures.

The first fraction, b.p. 120–123° (13 mm.), weighed about 12 g. *Anal.* Calcd. for Si_4OCl_{10}: Si, 23.3; Cl, 73.4; Si–Si bonds, 2/mole; mol. wt., 483. Found: Si, 23.2; Cl, 73.4; Si–Si bonds, 1.96/mole; mol. wt., 475. The second fraction, b.p. 140–143° (14 mm.), weighed about 5 g. *Anal.* Calcd. for $Si_5O_2Cl_{12}$: Si, 23.5; Cl, 71.2; Si–Si bonds, 2/mole; mol. wt., 598. Found: Si, 23.4, Cl, 71.0, Si–Si bonds, 1.93/mole; mol. wt., 595. The number of Si–Si linkages was determined by measuring the volume of hydrogen resulting from decomposition of the sample with dilute alkali while molecular weights were obtained from the freezing point depression of *p*-dioxane.

DEPARTMENT OF CHEMISTRY
MASSACHUSETTS INST. OF TECH. W. C. SCHUMB
CAMBRIDGE, MASSACHUSETTS R. A. LEFEVER

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