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Abstract

Full Text

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THE REACTION OF ETHYLENE OXIDE WITH SILICON TETRACHLORIDE AND ETHYLTRICHLOROSILANE

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The reaction of α-oxides of the ethylene series with silicon tetrachloride and alkyl(aryl)chlorosilanes has been partially described in the literature ([1–5]). In these cases, products of complete reaction of ethylene oxide were usually obtained both with silicon tetrachloride and with its substituted derivatives. The reactions, as is known, proceed according to the schemes:

$$1)\ SiCl_4 + 4\,\underset{\diagdown\,\diagup}{\overset{CH_2-CH_2}{O}} \rightarrow Si(OCH_2\cdot CH_2Cl)_4$$

$$2)\ RSiCl_3 + 3\,\underset{\diagdown\,\diagup}{\overset{CH_2-CH_2}{O}} \rightarrow RSi(OCH_2\cdot CH_2Cl)_3$$

Meanwhile, it is evidently possible to obtain not only products of complete reaction, but also a series of substances formed as a result of partial (incomplete) reaction of silicon tetrachloride and its substituted derivatives with ethylene oxide. This may be represented by the following equations for silicon tetrachloride:

$$1)\ SiCl_4 + \underset{\diagdown\,\diagup}{\overset{CH_2-CH_2}{O}} \rightarrow SiCl_3OCH_2\cdot CH_2Cl$$

$$2)\ SiCl_4 + 2\,\underset{\diagdown\,\diagup}{\overset{CH_2-CH_2}{O}} \rightarrow SiCl_2(OCH_2\cdot CH_2Cl)_2$$

$$3)\ SiCl_4 + 3\,\underset{\diagdown\,\diagup}{\overset{CH_2-CH_2}{O}} \rightarrow SiCl(OCH_2\cdot CH_2Cl)_3$$

$$4)\ SiCl_4 + 4\,\underset{\diagdown\,\diagup}{\overset{CH_2-CH_2}{O}} \rightarrow Si(OCH_2\cdot CH_2Cl)_4$$

The reaction of ethyltrichlorosilane with ethylene oxide may be expressed by the equations:

$$1)\ C_2H_5SiCl_3 + \underset{\diagdown\,O\,\diagup}{CH_2{-}CH_2} \rightarrow C_2H_5SiCl_2OCH_2{\cdot}CH_2Cl$$

$$2)\ C_2H_5SiCl_3 + 2\,\underset{\diagdown\,O\,\diagup}{CH_2{-}CH_2} \rightarrow C_2H_5SiCl(OCH_2{\cdot}CH_2Cl)_2$$

$$3)\ C_2H_5SiCl_3 + 3\,\underset{\diagdown\,O\,\diagup}{CH_2{-}CH_2} \rightarrow C_2H_5Si(OCH_2{\cdot}CH_2Cl)_3$$

We have found conditions for the preparation and isolation of products of incomplete reaction of silicon tetrachloride and ethyltrichlorosilane with ethylene oxide. These products have not yet been described in the literature.

Table 1

Name of substance	Formula	Yield of 2-chloroethoxysilanes, %	B.p., °C (mm Hg)	D_{20}^{20}	C, % found	C, % calc.	H, % found	H, % calc.	Si, % found	Si, % calc.	Cl, % found	Cl, % calc.
2-Chloroethoxydichlorosilane	$C_2H_5SiCl_2OCH_2CH_2Cl$	41.0	173–174 (760)	1.2360	23.20	23.14	4.44	4.33	13.43	13.50	50.94	51.32
2-Chloroethoxydichlorosilane	$C_2H_5SiCl_2OCH_2CH_2Cl$	41.0	173–174 (760)	1.2360	23.40	23.14	4.53	4.33	13.80	13.50	50.61	51.32
Di-2-chloroethoxychlorosilane	$C_2H_5SiCl(OCH_2{\cdot}CH_2Cl)_2$	40.0	86 (3)	1.3622	28.60	28.64	5.19	5.15	11.14	11.13	42.99	42.31
Di-2-chloroethoxychlorosilane	$C_2H_5SiCl(OCH_2{\cdot}CH_2Cl)_2$	40.0	86 (3)	1.3622	28.81	28.64	5.28	5.15	11.36	11.13	43.17	42.31
Tri-2-chloroethoxysilane	$C_2H_5Si(OCH_2CH_2Cl)_3$	40.0	122–123 (3)	1.3631	32.48	32.49	5.76	5.76	9.41	9.48	36.52	35.88

Name of substance	Formula	Yield of 2-chloroethoxysilanes, %	B.p., °C (mm Hg)	D_{20}^{20}	C, % found	C, % calc.	H, % found	H, % calc.	Si, % found	Si, % calc.	Cl, % found	Cl, % calc.
Tri-2-chloroethoxysilane	$C_2H_5Si(OCH_2CH_2Cl)_3$	80.0	122–123 (3)	1.4863	32.29	32.49	5.82	5.76	9.67	9.48	36.79	35.88
2-Chloroethoxytrichlorosilane	$SiCl_3OCH_2CH_2Cl$	60.4	153–154 (760)	1.4376	11.36	11.12	1.98	1.90	13.10	13.03	65.57	65.92
2-Chloroethoxytrichlorosilane	$SiCl_3OCH_2CH_2Cl$	60.4	153–154 (760)	1.4376	11.41	11.12	2.02	1.90	13.14	13.03	65.62	65.92
Di-2-chloroethoxydichlorosilane	$SiCl_2(OCH_2CH_2Cl)_2$	50.5	226–227 (760)	1.4668	18.86	18.62	3.15	3.05	10.82	10.68	54.56	54.98
Di-2-chloroethoxydichlorosilane	$SiCl_2(OCH_2CH_2Cl)_2$	50.5	226–227 (760)	1.4668	18.70	18.62	3.17	3.05	11.10	10.68	54.34	54.98
Tri-2-chloroethoxychlorosilane	$SiCl(OCH_2CH_2Cl)_3$	47.0	123 (3)	1.5373	23.98	23.75	4.02	3.97	9.33	9.4	46.77	46.99
Tri-2-chloroethoxychlorosilane	$SiCl(OCH_2CH_2Cl)_3$	47.0	123 (3)	1.5373	24.02	23.75	4.03	3.97	9.28	9.4	46.46	46.99
Tetra-2-chloroethoxysilane	$Si(OCH_2CH_2Cl)_4$	80.0	159 (3)	1.5434	27.91	28.04	4.61	4.61	8.16	8.09	41.62	41.01
Tetra-2-chloroethoxysilane	$Si(OCH_2CH_2Cl)_4$	80.0	159 (3)	1.5437	27.53	28.04	4.58	4.61	7.98	8.09	41.83	41.01

Experimental Part

We shall consider the conditions for obtaining the products of the interaction of ethylene oxide with silicon tetrachloride and ethyltrichlorosilane using the preparation of 2-chloroethoxyethyldichlorosilane as an example.

To 32.6 g (0.2 g-mol) of cooled (ice) ethyltrichlorosilane, b.p. 98–99° (760 mm)

and D_{20}^{20} 1.2380, liquid ethylene oxide was added in portions, with stirring, until an increase in weight of 8.8 g (0.2 g-mol) was obtained. If the reaction did not begin at once, the reaction vessel was removed from the ice (the onset of the reaction can be observed from the rise in the temperature of the reaction mixture).

After completion of the reaction, the reaction mass was subjected to distillation. 15.8 g (44.13% of theory) of 2-chloroethoxyethyldichlorosilane was isolated, b.p. 173–174° (760 mm), D_{20}^{20} 1.2365.

Found, %: C 23.40; 23.20; H 4.44; 4.53; Si 13.43; 13.80; Cl 50.92; 51.32
$C_4H_9SiOCl_3$. Calculated, %: C 23.14; H 4.33; Si 13.50; Cl 51.32

The remaining products were obtained by an analogous procedure. Some of their physicochemical properties are given in Table 1.

The products of the incomplete interaction of ethylene oxide with silicon tetrachloride and ethyltrichlorosilane are liquids with a sharp odor, fuming in air, and readily soluble in organic solvents.

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Note: Figure translations are in progress. See original paper for figures.