tween the composition of Solid Solution A and Na_2CO_3.

TABLE III

THE SYSTEM Na_2CO_3–Na_2SO_4–NaOH–H_2O AT 100°

Soln.	Solution composition, weight per cent.			Wet residue composition, weight per cent.			Solid phase identification
	Na_2CO_3	Na_2SO_4	NaOH	Na_2CO_3	Na_2SO_4	NaOH[a]	
			Univariant Line A				
1	28.5	3.2	0	49.8	37.3	0	$Na_2CO_3 \cdot H_2O$, S.S.
2	28.0	3.1	0.9	53.5	24.5	1.0	$Na_2CO_3 \cdot H_2O$, S.S.
3	26.4	2.9	2.5	54.6	22.0	2.5	$Na_2CO_3 \cdot H_2O$, S.S.
4	25.4	2.8	2.6	61.6	23.8	0	$Na_2CO_3 \cdot H_2O$, S.S.
5	23.3	2.6	4.2	..	..	..	$Na_2CO_3 \cdot H_2O$, S.S.
6[b]	23.8	2.5	3.8	62.6	18.4	0.9	$Na_2CO_3 \cdot H_2O$, S.S.
7	21.5	2.3	5.4	56.9	16.8	2.3	$Na_2CO_3 \cdot H_2O$, S.S.
8	17.9	1.9	8.6	67.1	13.6	0.6	$Na_2CO_3 \cdot H_2O$, S.S.
9	17.4	2.0	8.7	61.5	15.3	1.6	$Na_2CO_3 \cdot H_2O$, S.S.
10	14.0	1.7	12.0	68.7	12.6	0	$Na_2CO_3 \cdot H_2O$, S.S.
11	10.8	1.2	15.4	..	..	..	$Na_2CO_3 \cdot H_2O$, S.S.
12	8.0	0.8	18.9	55.2	8.3	0.2	$Na_2CO_3 \cdot H_2O$, S.S.
13	5.0	0.6	23.2	50.9	7.1	9.2	$Na_2CO_3 \cdot H_2O$, S.S.
14	3.2	0.5	26.9	51.9	6.8	11.0	$Na_2CO_3 \cdot H_2O$, S.S.
			Univariant Line B				
1	4.0	26.6	0	19.3	70.5	0	Na_2SO_4, S.S.
2	3.4	24.5	1.5	18.3	71.8	1.8	Na_2SO_4, S.S.
3	3.0	21.8	3.4	..	..	..	....
4[c]	2.9	21.9	3.3	15.2	84.8	0	Na_2SO_4, S.S.
5	2.4	19.6	5.1	15.3	78.8	0	Na_2SO_4, S.S.
6	2.2	17.3	7.0	12.9	79.7	0.4	Na_2SO_4, S.S.
7	2.5	11.7	11.8	..	..	..	Na_2SO_4, S.S.
8	2.1	7.5	16.3	12.0	73.5	0.2	Na_2SO_4, S.S.
9	2.3	4.7	21.4	12.9	71.9	2.6	Na_2SO_4, S.S.
10	1.3	2.0	30.4	11.0	65.6	8.0	Na_2SO_4, S.S.

[a] NaOH calculated by difference. [b] Solution 5 given 68 hours agitation time, all others 20 hours. [c] Solution 3 given 68 hours agitation time, all others 20 hours.

Similarly along line "B" the extrapolated tie lines intersected the Na_2SO_4–Na_2CO_3 axis at points between Na_2SO_4 and Solid Solution B. No attempts was made to study completely the composition of the solid phase in the area of $x Na_2SO_4 \cdot Na_2CO_3$ which covers the major portion of the diagram, since it was felt that an adequate estimate of this composition could be made by utilizing the data illustrated in Fig. 3.

Acknowledgment.—The authors wish to express their appreciation to Albert Kauffman and Howard Jaffee of the Petrographic Department at this station for doing the microscopic work, and to Richard A. Brown, Chemical Engineer, in charge of the Alkaline Alumina Project, and John E. Conley, Chief of the Chemical Engineering Unit, for valuable suggestions and criticisms during the investigation.

Summary

1. The major portion of the 4-component system Na_2CO_3–Na_2SO_4–NaOH–H_2O at 100° has been determined, involving the determination of parts of the three related 3-component systems, namely: (1) Na_2CO_3–NaOH–H_2O (2) Na_2SO_4–NaOH–H_2O and (3) Na_2CO_3–Na_2SO_4–H_2O at the same temperature. Ordinary chemical methods were used in solution and solid phase analyses and the petrographic microscope was employed to identify the solid phases.

2. In this system for the concentration range studied, three fields were encountered which are $Na_2CO_3 \cdot H_2O$, solid solution of the varying composition, (1.4–2.2)$Na_2SO_4 \cdot Na_2CO_3$ and Na_2SO_4.

3. The results of this study are presented conveniently on a ternary diagram with lines of constant total dissolved solids.

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[CONTRIBUTION FROM THE RESEARCH LABORATORY OF THE GENERAL ELECTRIC COMPANY]

Dipole Moments of Linear and Cyclic Polymethylpolysiloxanes

BY ROBERT O. SAUER AND DARWIN J. MEAD

In connection with an extended study in this Laboratory of the physical properties[1] of polysiloxanes we have had occasion to investigate the dielectric behavior of the pure liquid polymethylpolysiloxanes reported recently by Patnode and Wilcock.[2] We sought to gain an insight into the structure of the polysiloxanes, and we hoped, in particular, to obtain at least an approximate value for the silicon–oxygen–silicon bond angle from the dipole moments computed by means of Onsager's equation.[3] Upon evaluation of the accepted physical constants appearing in this equation it reduces to

$$\mu^2 = 0.0482 \times 10^{-36}\left[V \cdot \frac{2\epsilon + n^2}{\epsilon(n^2 + 2)^2}(\epsilon - n^2)\right] \text{ (e. s. u.)} \qquad (1)$$

at 20.0°, in which

μ = dipole moment (gas phase) of the molecule
V = the molar volume
ϵ = dielectric constant of the pure liquid substance, and
n = the refractive index

Although, according to Onsager, the refractive index should include the contribution of the atomic polarization as well as that of the electron polarization we have set $n = n^{20}{}_D$. Equation 1 may be condensed to the form

$$\mu = 0.220 \times 10^{-18}\sqrt{P^*} \text{ (e. s. u.)} \qquad (2)$$

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[1] The properties so far discussed include: (a) crystal habit and configuration [Burkhard, Decker and Harker, THIS JOURNAL, **67**, 2174 (1945)]; (b) molar volume and viscosity [Hurd, *ibid.*, **68**, 364 (1946)]; (c) vapor pressure and viscosity relations [Wilcock, *ibid.* **68**, 691 (1946)]; (d) molar and group refraction [Sauer, *ibid.*, **68**, 954 (1946)].

[2] Patnode and Wilcock, THIS JOURNAL, **68**, 358 (1946).

[3] Onsager, THIS JOURNAL, **58**, 1490 (1936). Böttcher [*Physica*, **6**, 59 (1939)] has analyzed a large amount of experimental work in the light of the Onsager formula. In general, results are obtained which check well with those found by the usual methods.

in which P^* represents the quantity in the brackets.

Our attention has been called to the fact that the oxygen bond angle in cristobalite has been determined[4] and found to be in the neighborhood of 150°. There exists the definite possibility that the oxygen bond angle in the polymethylpolysiloxanes might similarly have a value in the range 150 ± 10°. Since such a value would be somewhat at variance with the accepted valence geometry of the oxygen atom as shown by the simpler compounds, for example, water, oxygen fluoride and dimethyl ether, it seemed of some interest to interpret our dielectric constant data from the structural point of view.

We also wished to examine the dipole moments of the linear compounds $(CH_3)_3Si[OSi(CH_3)_2]_qCH_3$ as a function of q, the number of oxygen atoms per molecule. Of interest in this connection is the apparently well-established equation

$$\mu = C\sqrt{q} \qquad (3)$$

in which q is the number of equal dipoles per molecule and C is a constant usually associated with the value of the individual group moments. Equation 3 has been found generally valid for the α,ω-dihydroxyalkanes[5] (alkylene glycols), the α,ω-dibromoalkanes[5] (alkylene dibromides), a number of cellulosic materials[6] and peptides,[7] the polymeric internal esters of ω-oxyundecanoic acid,[8] and for certain of the vinyl type polymers, such as polychloroprene[6] and polyvinyl acetate.[6,9]

Experimental

Starting Materials.—Our samples of the compounds $(CH_3)_3Si[OSi(CH_3)_2]_qCH_3$, $q = 2, 3, 4$ and 5, and $[(CH_3)_2SiO]_q$, $q = 4, 5, 6, 7$ and 8, were kindly provided by Dr. Winton Patnode of this Laboratory and were portions of the same materials used by Hurd[1b] for his density and viscosity studies. The dielectric constants of these compounds were measured as received, and the molar volumes found by Hurd were used in our calculations. The hexamethyldisiloxane was prepared by hydrolyzing trimethylchlorosilane of about 98% purity (as shown by chlorine analyses) without solvent, distilling the water-insoluble layer from phosphoric anhydride, and carefully refractionating the distillate in a column of about 15 theoretical plates. The densities, d^{20}_4 (vac.), and refractive indices, $n^{20}D$, of the two samples used in our work were: (A) 0.7638, 1.3772; (B) 0.7637, 1.3774.

The methyl silicone oil (0–74) was prepared by "equilibrating" hexamethyldisiloxane with the hydrolyzate of pure dimethyldichlorosilane according to the procedure of Patnode and Wilcock.[2] After washing thoroughly with water to remove all traces of sulfuric acid and after drying the sample for our dielectric measurements the following constants were obtained: d^{20}_4 (vac.) 0.9650, $n^{20}D$ 1.4036. The value of the specific refraction computed from these data ($R_D = 0.2532$) indicates[10] an average molecular weight corresponding to the formula $(CH_3)_3Si[OSi(CH_3)_2]_{66}CH_3$ (methyl-to-silicon ratio, 2.030). Our experience with such "equilibration" products indicates that there are substantially no cyclopolysiloxanes present.

Measurement of the Dielectric Constant.—Dielectric constants at 20.0° and 60 cycles were measured with the Schering bridge.[11,12] The (guarded) cell was of the platinum-glass type described by Mead and Fuoss.[13] The dielectric losses were very low in all cases (tan $\delta < 0.0003$). The results of our measurements and the calculated values of P^*, P^*/q, and μ are given in Table I.

TABLE I

Compound $(CH_3)_3Si[OSi(CH_3)_2]_qCH_3$	V^{20} (mls.)	$n^{20}D$	ϵ'^{20a}_{60}	P^* (mls.)	P^*/q	$\mu \times 10^{18}$
$q = 1$, sample A	212.5	1.3772	2.17_8	11.3	11.3	0.74
sample B	212.5	1.3774	2.17	11.0	11.0	.73
$q = 2$	288.4	1.3848	2.30_2	20.4	10.2	.99
$q = 3$	363.9	1.3895	2.39_4	30.6	10.2	1.22
$q = 4$	439.4	1.3925	2.45_8	40.8	10.2	1.40
$q = 5$	515.0	1.3948	2.50_4	51.4	10.3	1.58
Oil 0–74, $q = 66$	78.4^b	1.4036	2.72	..	10.2	..
$[(CH_3)_2SiO]_q$						
$q = 4$	310.2	1.3968	2.39_4	24.8	6.2	1.09
$q = 5$	386.4	1.3982	2.50_0	37.5	7.5	1.35
$q = 6$	459.9	1.4015	2.58_7	50.3	8.4	1.56
$q = 7$	533.3	1.4040	2.67_8	65.4	9.3	1.78
$q = 8$	605.5	1.4060	2.74_1	79.6	10.0	1.96

[a] $\epsilon'' \leq 0.0009$. [b] This quantity has been computed as the average value per oxygen atom.

Theory and Discussion of Results

A glance at Table I indicates that P^*/q, and consequently $\mu/\sqrt{q}$, is approximately constant; hexamethyldisiloxane is the only member of the group of linear polymethylpolysiloxanes for which a significant deviation exists. Empirically, we then have the two relationships:

$$P^* = 10.2q \text{ (mls./mole)} \qquad (4)$$

and

$$\mu = 0.70\sqrt{q} \text{ (Debye units)} \qquad (5)$$

Hence, equation 3 is valid for these linear siloxanes. This validity is readily explained by the assumption of a large (145° or larger) valence angle for oxygen as demonstrated by the following discussion.

In molecules of the type formula $(CH_3)_3Si-[OSi(CH_3)_2]_qCH_3$ the total moment μ of the molecule can be thought of as being the resultant of certain vector dipoles of magnitude m (total number, $2q$) in the direction of and associated with *each* silicon–oxygen bond, each dipole itself being the resultant of several bond moments. The magnitude of μ will then be a function of q, of the oxygen bond angle α, of the silicon bond angle β (except for $q = 1$), and of the scalar value of m. Assuming free rotation around each silicon–oxygen bond and the tetrahedral angle for silicon, *i.e.*, $\cos \beta = -1/3$, one may express the value of μ in terms of (a) the equal scalar moments m associated with each silicon–oxygen bond, (b) the

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(4) Nieuwenkamp, *Z. Krist.*, **92**, 82 (1935); **96**, 454 (1937).

(5) Smyth and Walls, THIS JOURNAL, **53**, 527, 2115 (1931); **54**, 2261 (1932).

(6) Sakurada and Lee, *Z. physik. Chem.*, **43B**, 245 (1939).

(7) Wyman and McMeekin, THIS JOURNAL, **55**, 908 (1933).

(8) Bridgman, THIS JOURNAL, **60**, 530 (1938).

(9) Although not specifically stated in the paper by Svirbely and Lander [THIS JOURNAL, **67**, 2189 (1945)] it appears that the dipole moment of Carbowax 4000 (9.91 D) is compatible with equation 3; the value of C for this substance ($q = 86$) is 1.07 D whereas 1.14 D is the accepted value of the moment of ethyl ether [Fuchs, *Z. Physik*, **63**, 824 (1930); Stuart, *ibid.*, **51**, 490 (1928)].

(10) Sauer, THIS JOURNAL, **68**, 954 (1946).

(11) Hague, "Alternating Current Bridge Methods," Pittman, London, 1932, pp. 241ff.

(12) Fuoss, THIS JOURNAL, **59**, 1703 (1937).

(13) Mead and Fuoss, *ibid.*, **61**, 2048 (1939).

oxygen bond angle α, and (c) the number of oxygen atoms in the siloxane chain (q). Thus, by applying the analytical method described by Eyring[14] the following relation is obtained:

$$\mu^2/q = 2m^2\,(1 + \cos\alpha)\left\{\frac{2}{3 + \cos\alpha} + \frac{3}{q}\,\frac{1 + \cos\alpha}{(3 + \cos\alpha)^2}\left[1 - \left(\frac{\cos\alpha}{-3}\right)^q\right]\right\} \qquad (6)^{14a}$$

The term $2m^2(1 + \cos\alpha)$ is seen to be the square of the moment associated with an isolated siloxane (Si–O–Si) linkage, *i.e.*, μ_1^2. Further, it is readily apparent that as q approaches infinity the second term in the brackets of equation 6 vanishes. Thus, we may write

$$\left(\frac{\mu^2}{q}\right)_{q \to \infty} = 4m^2\,\frac{1 + \cos\alpha}{3 + \cos\alpha} = \mu_1^2 \cdot \frac{2}{3 + \cos\alpha} \qquad (7)$$

in which it is seen qualitatively that if $\cos\alpha$ is close but not quite equal to -1, equation 3 will be valid for the higher members of this polysiloxane series since the term $2/(3 + \cos\alpha)$ will be close to unity. On the other hand if $\alpha = 110°$ the coefficient of μ_1^2 in equation 7 would be 0.75, and the constant C in equation 3 would then be equal to 0.867 μ_1 rather than μ_1.

Substituting the results on hexamethyldisiloxane and on methyl silicone oil 0-74 in equation 7 we find that $2/(3 + \cos\alpha) = 0.91$. This result indicates that α is considerably larger than the tetrahedral angle.

In order to compute the silicon–oxygen–silicon bond angle in hexamethyldisiloxane from the dipole moment it is necessary to estimate the amount of ionic character in the silicon–oxygen bond. We have used the empirical equation

$$\text{Amount of ionic character} = 0.16\,(x_0 - x_{\text{Si}}) + 0.035\,(x_0 - x_{\text{Si}})^2 \qquad (8)$$

suggested by Hannay and Smyth[15] which, for $x_0 - x_{\text{Si}} = 1.7$, yields 0.37 as the amount of ionic character rather than the 0.50 estimated previously.[16] The silicon–oxygen bond moment may then be taken as 2.8 debye units (using 1.60 Å. as the internuclear separation) directed toward the silicon atom. To obtain m, the net moment associated with and directed along each silicon-oxygen bond in hexamethyldisiloxane, one must subtract an estimated 1.0 D for the carbon–silicon bond moment from and add 0.4 D (the resultant of the nine carbon–hydrogen bond moments) to the silicon–oxygen bond moment proper. The estimated value for m then becomes 2.2 D. Two such vectors forming an angle of 161° with each other give a resultant moment of 0.73 debye unit.[17]

The oxygen bond angle α may also be computed using the data on silicone oil 0-74 and equation 7, thus

$$4m^2\,\frac{1 + \cos\alpha}{3 + \cos\alpha} = 0.49$$

Assuming $m = 2.2\ D$ the value of α calculated from this relation is 161.5°.

In Table II we have listed the calculated values of the dipole moments (in debye units) of the linear molecules using equation 5 (column 2) and equation 6 (column 3); in the latter computation we have used $m = 2.2$ D and $\alpha = 161.5°$. The values so obtained compare favorably with those found (last column).

TABLE II

Values q	μ(eq. 5)	μ(eq. 6)	μ(found)
1	0.70	0.71	0.74, 0.73
2	0.99	0.99	0.99
3	1.22	1.22	1.21
4	1.40	1.40	1.40
5	1.58	1.57	1.56

Because of the assumptions involved in our calculations of m it does not appear justifiable to describe the silicon-oxygen-silicon bond angle in the linear polysiloxanes more precisely than 160 ± 15°.

For the cyclic polysiloxanes we note from Table I that the values of P^*/q approach the values found for the linear molecules. This fact appears to confirm the rather obvious deduction that the contribution per dipole in a long chain to the mean square moment should be identical with that observed per dipole in a very large ring.

Acknowledgment.—The authors gratefully acknowledge the experimental assistance given by Messrs. S. I. Reynolds and E. G. Chace and wish to express their appreciation for the help and encouragement given us by Dr. David Harker who originally suggested to us the possibility that the oxygen bond angle in the polymethylpolysiloxanes might be appreciably larger than the tetrahedral angle.

Summary

1. Dielectric constants at 20° and 60 cycles are reported for a series of linear polymethylpolysiloxanes: $(CH_3)_3Si[OSi(CH_3)_2]_qCH_3$, in which q has the values 1, 2, 3, 4, and 5, and for a series of cyclic polymethylpolysiloxanes: $[(CH_3)_2SiO]_q$ in which q has the values 4, 5, 6, 7 and 8. By using Onsager's equation the dipole moments of these compounds have been computed.

2. The dipole moments of the linear compounds agree satisfactorily with the simple empirical relationship: $\mu = 0.70\sqrt{q}$. For the cyclic

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[14] Eyring, *Phys. Rev.*, **39**, 746 (1932). Compare also Fuoss and Kirkwood, THIS JOURNAL, **63**, 385 (1941).

[14a] Our original expression was of the form

$$\mu^2/q = 2m^2\,(1 + \cos\alpha)\left\{1 - \frac{1 + \cos}{3}\left[\frac{3}{\cos\alpha} + \sum_{a=1}^{a=q}\frac{q + 1 - a}{q}\left(\frac{\cos\alpha}{-3}\right)^{a-2}\right]\right\}$$

The Referee has indicated, however, that this may be more neatly expressed in the closed form of equation 6.

[15] Hannay and Smyth, THIS JOURNAL, **68**, 171 (1946).

[16] Pauling, "The Nature of the Chemical Bond," 2d ed., Cornell University Press, Ithaca, N. Y., p. 74.

[17] Note added in proof: Since the acceptance of this paper for publication we have seen the recently available work of Malatesta and Pinzotti [*Gazz. chim. ital.*, **73**, 143 (1944)] who measured the dipole moment of hexaethyldisiloxane in carbon tetrachloride at 24.2°. These authors report $\mu = 0.63 \pm 0.01$ for this analogous compound.

compounds the dipole moments of the larger rings appear to approach the values predicted by this equation.

3. From the dipole moment of hexamethyldisiloxane (0.74 *D.*) the silicon-oxygen-silicon bond angle has been calculated; its value appears to be 160 ± 15°.

SCHENECTADY, N. Y. RECEIVED[18] MARCH 11, 1946

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[18] Original manuscript received September 18, 1945.

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY OF THE POLYTECHNIC INSTITUTE OF BROOKLYN]

Preparation of Ethylenesulfonic Acid—Salts and Esters

BY WILLET F. WHITMORE AND EDWARD F. LANDAU[1,2]

The preparation of ethylenesulfonic acid has been previously described. Kohler[3,4,5,6] prepared the compound by decomposition of ethanedisulfonyl chloride. Ethylenesulfonic acid has also been prepared by dehydrohalogenation of 2-haloethanesulfonic acid,[4] and by removal of sulfuric acid from ethionic acid.[5]

In the course of this investigation, the preparation of ethylenesulfonic acid by the above methods has been reinvestigated, and improved methods for the isolation of its salts in a pure form have been found.

Kohler described ammonium ethylenesulfonate (m.p. 156°) as a yellow crystalline solid, having a limited solubility in hot 95% ethanol. In contrast to Kohler's description, it was found that by utilizing lower temperatures for the isolation of the ammonium salt, it could be obtained as a colorless crystalline material which was relatively soluble in hot ethanol. However, the melting point reported by Kohler could not be duplicated. It was also observed that exposure to heat (70–80°) caused a yellow discoloration of the salt and decreased its alcohol solubility.

The above-mentioned patents[4,5] described the preparation of sodium ethylenesulfonate, but do not give a method for its isolation. A procedure for the isolation of the purified salt is described below.

No crystalline derivative of ethylenesulfonic acid readily suitable for its characterization has been described in the literature. It was found that the S-benzyl thiouronium salt was easily prepared and had a sharp melting point. Three other aliphatic sulfonic acids were characterized by their S-benzyl thiouronium salts, and are described in Table I.

TABLE I

S-BENZYL THIOURONIUM SULFONATES $[C_6H_5CH_2SC(NH_2)_2][RSO_3]$

Sulfonic acid	M. p., °C.	Sulfur, % Calcd.	Sulfur, % Found	
$CH_2{=}CHSO_3H$	145–146	23.27	22.95	23.67
$BrCH_2CH_2SO_3H$	125–126	17.98	18.07	18.27
$ClCH_2CH_2SO_3H$	129–130	20.51	20.72	20.74
$(CH_2{-}SO_3H)_2$	201–202	24.42[a]	24.48	24.72

[a] Calcd. for $[(CH_2SO_3)_2][C_6H_5SC(NH_2)_2]_2$.

The esters of ethylenesulfonic acid have received scant attention by previous investigators. The ethyl ester has been prepared by esterification of ethylenesulfonic acid with ketene acetal,[7] and also from ethylene-sulfonyl chloride and ethanol. In the course of this investigation, a series of seven esters of ethylenesulfonic acid from 2-haloethanesulfonyl chloride was prepared by a method the chemistry of which is best illustrated by the following equation.

$$XCH_2CH_2SO_2Cl + ROH \xrightarrow{\text{pyridine}} CH_2{=}CHSO_2OR + HCl + HX$$

where X = Cl, Br; R = an alkyl radical

The pyridine serves both as a dehydrohalogenating agent and a halogen acid acceptor. The most satisfactory results were obtained by using only a slight excess of pyridine and performing the reaction in an inert solvent (methylene chloride). The esters so prepared were colorless liquids having physical properties as described in Table II.

Experimental

Salts of Ethylenesulfonic Acid

(a) Ammonium ethylenesulfonate was prepared by Kohler's procedure[3b] from 1,2-ethanedisulfonyl chloride. The following modifications were employed for its isolation:

The mixture of ammonium sulfonates (ammonium ethylenesulfonate and ammonium 1,2-ethanedisulfonate) was extracted with three 150-ml. portions of hot 95% ethanol. An alcohol insoluble residue of ammonium 1,2-ethanedisulfonate remained. The alcoholic extracts, pale yellow in color, were decolorized by treatment with 5 g. of Darco G-60 and concentrated by distillation under reduced pressure at 50°, to a volume of 200 ml. Upon cooling this solution to 5°, the ammonium ethylenesulfonate separated as a colorless crystalline solid. The yield was 63 g., 55% of the theoretical, based on 200 g. of 1,2-ethanedisulfonyl chloride.

Anal. Calcd. for $C_2H_7SO_3N$: S, 25.60; N, 11.2. Found: S, 25.72, 25.18; N, 10.9.

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(1) An abstract of a dissertation presented in May, 1945, to the Graduate Faculty of the Polytechnic Institute of Brooklyn by Edward F. Landau, in partial fulfillment of the requirements for the degree of Doctor of Philosophy.

(2) Present address: Celanese Corporation of America, Plastics Division, Newark, N. J.

(3) Kohler, *Am. Chem. J.*, **19**, 728 (1897); **20**, 680 (1898); **21**, 353 (1899).

(4) German Patent 678,730.

(5) German Patent 677,843.

(6) U. S. Patent 2,348,705.

(7) McElvain, Jelinek and Rorig, THIS JOURNAL, **67**, 1578 (1945).