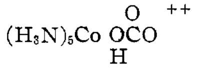
A conclusion about mechanism which applies to the results for $CO_3^=$ attached as a chelate, bound at one position, or not bound at all, is that there is no transfer of oxygen from the solvent to C at any stage on the release of CO_2. Thus it seems likely that the activated complex which in the case of $Co(NH_3)_5CO_3^+$ has the composition $Co(NH_3)_5CO_3H^{++}$, does not have the structure reasonable for this complex ion, but rather has the proton on the oxygen bridging cobalt and carbon



$$\overset{}{(H_3N)_5Co}\,\underset{H}{O}\overset{O}{C}O^{++}$$

Since new Co(III) bonds are established during the release of CO_2 from $Co(NH_3)_4CO_3^+$, it will be of interest to learn whether groups besides water (*e.g.*, Cl^-, $SO_4^=$) can enter during this rapid process. It was this possibility that attracted interest to the aquation of $Co(NH_3)_5CO_3^+$, but could not be realized in this reaction since oxygen is left attached to Co(III).

Acknowledgments.—This work was supported by the Office of Naval Research under contract N6-ori-02026. The funds for the purchase of the mass spectrometer used in the research were supplied by the Atomic Energy Commission under contract At(11-1)-92.

DEPARTMENT OF CHEMISTRY
UNIVERSITY OF CHICAGO
CHICAGO, ILL.

Some Esters of Unsaturated Acids

BY V. SATTA,[1] M. L. FEIN AND E. M. FILACHIONE

RECEIVED APRIL 6, 1953

In connection with a study of the preparation of co-polymers of acrylic esters, various esters of unsaturated acids (listed in Table I) were required. Though several of these esters are available commercially, their physical properties are either not found in the permanent chemical literature or these are insufficiently reported. *n*-Butyl maleate[2,3] and fumarate[2,3] and 2-chloroethyl maleate[4] and fumarate[5] have been most completely reported; for butyl fumarate, however, the previously reported constants and those of this paper are not in good agreement. The patent literature reports only the boiling point of 2-ethylhexyl maleate,[3] and the refractive index for *n*-butyl aconitate[6] and 2-ethylhexyl aconitate.[7] This paper reports the preparation, boiling point, refractive index and density of various esters of several unsaturated acids.

TABLE I

PROPERTIES OF SOME ESTERS OF UNSATURATED ACIDS

Ester	Yield, %	B.p., °C.	B.p., mm.	$n^{20}\text{D}$	d^{20}_4	Carbon, % Found	Carbon, % Calcd.	Hydrogen, % Found	Hydrogen, % Calcd.	Chlorine, % Found	Chlorine, % Calcd.	Sapn. equiv. Found	Sapn. equiv. Calcd.
2-Chloroethyl crotonate[a]		58–60	3.5	1.4599	1.1185					23.72	23.86		
n-Butyl maleate[a]		124–125	3.4	1.4451	0.9964								
n-Hexyl maleate	97	127	0.7	1.4499	.9583	68.20	67.57	10.48	9.92			142.1	142.2
2-Ethylhexyl maleate[a]		156	0.7	1.4548	.9437								
2-(2-Chloroethoxy)-ethyl maleate	86	197	1.0	1.4858	1.2738					21.70	21.54		
n-Butyl fumarate	80	117	2.5	1.4429	0.9775	63.12	63.13	8.77	8.77			113.9	114.1
2-Ethylhexyl fumarate	87	160	0.5	1.4570	0.9398	70.53	70.55	10.58	10.66			171.4	170.2
2-(2-Chloroethoxy)-ethyl fumarate	83	182	0.3	1.4863	1.2673					21.83	21.54		
n-Butyl chloromaleate	83	112	1.2	1.4558	1.0756	54.86	55.10	7.29	7.23	13.50	13.31		
2-Chloroethyl chloromaleate	75	138	1.2	1.4983	1.4047					38.44	38.60		
n-Butyl itaconate[a]		82	0.2	1.4450	0.9839								
n-Butyl aconitate[a]		155	.5	1.4553	1.0184								
2-Ethylhexyl aconitate[a]		170[b]	.07[b]	1.4620	0.9589								

[a] The commercially available esters were redistilled. [b] Distilled in an alembic-type still.

Experimental

Samples of 2-chloroethyl crotonate, butyl maleate, 2-ethylhexyl maleate, butyl itaconate, butyl aconitate and 2-ethylhexyl aconitate were available commercially; these were redistilled and the constants determined.

The other esters of Table I were prepared by the usual esterification procedure, glass equipment with ground glass joints being employed.[8] A mixture of acid or anhydride, an excess of the alcohol, sulfuric acid catalyst and entraining agent was refluxed, and water was continuously removed in a separating trap. Benzene or toluene was used as the entraining agent except in the preparation of the butyl esters, in which the excess of butyl alcohol served as the entraining agent. After esterification was complete, the catalyst was neutralized with sodium acetate, and the ester isolated by vacuum distillation. A 35-mm. by 50-cm. Vigreux or an alembic-type still,[9] mounted to permit agitation of the still pot contents, was used in the distillation. Center fractions were collected for determination of the various constants. Results are shown in Table I.

EASTERN REGIONAL RESEARCH LABORATORY[10]
PHILADELPHIA 18, PENNSYLVANIA

---

(1) Foreign trainee from Italy.

(2) G. H. Jeffery and A. I. Vogel, *J. Chem. Soc.*, 658 (1948).

(3) W. O. Kenyon and C. C. Unruh, U. S. Patent 2,448,531, September 7, 1948.

(4) M. E. Synerholm and A. Hartzell, *Contrib. Boyce Thompson Inst.*, **14**, 79 (1945); *C. A.*, **40**, 669 (1946).

(5) D. Vorländer, *Ann.*, **280**, 200 (1894).

(6) P. M. Kirk, U. S. Patent 2,375,563, May 8, 1945.

(7) E. R. Meincke, U. S. Patent 2,475,629, July 12, 1949.

(8) P. A. Shearer and A. M. Pardee, *Proc. S. Dakota Acad. Sci.*, **15**, 24 (1935); *C. A.*, **30**, 2550 (1936).

(9) W. P. Ratchford and C. E. Rehberg, *Anal. Chem.*, **21**, 1417 (1949).

(10) One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, United States Department of Agriculture. Article not copyrighted.

Sulfatoalkyl Amines

BY WILSON A. REEVES AND JOHN D. GUTHRIE

RECEIVED APRIL 13, 1953

Although a number of investigators have reported the preparation and use of 2-aminoethyl sulfuric acid[1,2] other aminoalkyl sulfates have received very little attention. Apparently, most interest in these sulfates has been for the production of

---

(1) Sigmund Fränkel and Martha Cornelius, *Ber.*, **51**, 1654 (1918).

(2) John D. Guthrie, *Textile Research J.*, **17**, 625 (1947).