Anal. Calcd. for $C_5H_9NO_5$: C, 41.14; sapn. equiv., 88.6. Found: C, 41.75; sapn. equiv., 86.8.

Polymerization of 2-Nitratoethyl Esters.—A 1-g. sample of 2-nitratoethyl methacrylate and 10 mg. of benzoyl peroxide on heating in a watch glass at 100° for twenty minutes gave a hard polymer. This compound also polymerized to a hard polymer after standing at 25° for a few days in the absence of a catalyst. 2-Nitratoethyl acrylate, 1 g., and 10 mg. of benzoyl peroxide on heating at 100° for one hour gave a soft, tacky polymer. The 2-nitratoethyl crotonate did not polymerize under these conditions.

Reaction with Alkali.—Mixtures of weighed samples of the 2-nitratoethyl esters (0.3 g.) and a measured excess of 0.1 *N* aqueous sodium hydroxide were shaken until they became homogeneous (5 minutes). Back-titration with standard hydrochloric acid gave for the equivalent weight for 2-nitratoethyl acrylate 164 (calcd. for one equivalent per molecular weight 161.2), for 2-nitratoethyl methacrylate 179 (calcd. 175.3), for 2-nitratoethyl crotonate 179 (calcd. 175.3) and for 2-nitratoethanol *ca.* 600 (calcd. 107.2).

The cleavage that occurred here was indicated to be that of the nitratoalkyl group from the unsaturated acid. A sample of 2.4 g. of 2-nitratoethyl crotonate was shaken with 2 g. of sodium hydroxide in 50 cc. of water until the mixture became homogeneous. Ethereal extracts of the solution on distillation gave 0.7 g. of 2-nitratoethanol, b. p. 95° (8 mm.), $n^{25}D$ 1.4366 and the saponification equivalent determined in the manner given below was 111 (calcd. 107).

Saponification equivalents for the three esters were determined in the following manner. A 0.3-g. sample was added to 25 ml. of 0.2 *N* alcoholic potassium hydroxide and the mixture refluxed for thirty minutes. Back-titration with standard acid gave the following equivalent weights: for 2-nitratoethyl acrylate, 80.5 (calcd. for two equivalents per molecular weight, 80.6), for 2-nitratoethyl methacrylate 88.4 (calcd. 88.5), for 2-nitratoethyl crotonate 86.8 (calcd. 88.5) and for 2-nitratoethanol 111 (calcd. 107).

A sample of 0.3 g. of 2-nitratoethyl crotonate was soluble on addition to 25 ml. of 0.2 *N* alcoholic potassium hydroxide. After standing for five minutes the solution was back-titrated with standard hydrochloric acid to give an equivalent weight of *ca.* 600. The experiment repeated with 2-nitratoethyl acrylate gave an equivalent weight of *ca.* 600.

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DEPARTMENT OF CHEMISTRY
DE PAUL UNIVERSITY
CHICAGO 14, ILLINOIS
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Some Properties of Pure γ-Butyrolactone

BY CLYDE MCKINLEY AND JOSEPH P. COPES

A highly purified sample of γ-butyrolactone has been prepared[1] and certain of its physical properties determined. Vapor pressures, using an isoteniscope, were determined and are presented in the table. Freezing point was determined using apparatus patterned after that in use by the National Bureau of Standards[2] and T_{f_0} was found to be −43.53°[3] by Mr. L. J. Lohr of this Laboratory. The refractive index, using an Abbe refractometer, was $n^{25}D$ 1.4348 and the density, using a pycnometer, was found as $d^{25}{}_4$ 1.1254.

P, mm.	Temp., °C.
50.1	119
79.4	132
125.9	145
199.6	159
316.3	174
501.2	189
708.0	201
760 (extrap.)	204 (extrap.)

GENERAL ANILINE AND FILM CORPORATION
CENTRAL RESEARCH LABORATORY
EASTON, PENNA.
RECEIVED JUNE 13, 1950

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[1] For method see PB 60902.

[2] Research paper 1937.

[3] Calculations as in Bureau of Standards Technical Paper No. 1596, Research paper 1676.

Dipositive Silver as an Intermediate in Coulometric Analysis

BY DALE J. MEIER AND ERNEST H. SWIFT

Electrolytically generated bromine has been shown to be a satisfactory intermediate oxidant in coulometric titrations of arsenic(III), antimony(III) and thiodiglycol.[1] At the concentration of bromine and bromide ion existing in these titrations the use of bromine as the intermediate is limited by equilibrium considerations to the determination of reductants with half-cell potentials (Red. = Ox. + e^-) more positive than approximately −0.78 v. This value is obtained by assuming that the final approximate concentrations of bromide and tribromide were 10^{-1} and 10^{-6} *M*, respectively, and that the final ratio of $M_{ox.}$ to $M_{red.}$ was 1000. The negative limit of the oxidation potential can be extended somewhat by the use of chlorine as the intermediate, and Farrington and Swift[2] have described the use of chlorine for the determination of arsenic(III). In an effort to further extend the permissible negative limit of the half-cell potential of the reductant being determined, and also to find non-halide intermediates, we have investigated dipositive silver as an intermediate oxidant for coulometric titrations. The Ag(I)–Ag(II) formal potential has been determined by Noyes and his co-workers to be −1.98 v. in perchloric acid solutions.[3,4]

It has long been known that higher oxidation states of silver can be obtained by anodic oxidation, and although solid compounds can be precipitated which contain silver in a tripositive state, Noyes and his co-workers[5] have shown in

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[1] R. Myers and E. H. Swift, THIS JOURNAL, **70**, 1047 (1948); R. A. Brown and E. H. Swift, *ibid.*, **71**, 2717 (1949); J. W. Sease, C. Niemann and E. H. Swift, *Ind. Eng. Chem., Anal. Ed.*, **19**, 197 (1947).

[2] P. S. Farrington and E. H. Swift, *Anal. Chem.*, **22**, 889 (1950).

[3] A. A. Noyes, D. DeVault, C. D. Coryell and T. J. Deahl, THIS JOURNAL, **59**, 1326 (1937).

[4] W. M. Latimer, "Oxidation Potentials," Prentice-Hall, Inc., New York, N. Y., 1938, p. 181.

[5] A. A. Noyes, J. L. Hoard and K. S. Pitzer, THIS JOURNAL, **57**, 1221 (1935); A. A. Noyes, K. S. Pitzer and C. L. Dunn, *ibid.*, **57**, 1229 (1935); A. A. Noyes and A. Kossiakoff, *ibid.*, **57**, 1238 (1935).