

INVESTIGATION OF ELECTROCHEMISTRY OF HIGH ENERGY COMPOUNDS IN ORGANIC ELECTROLYTES

BIBLIOGRAPHY ON PROPYLENE CARBONATE,
γ-BUTYROLACTONE, AND RELATED SUBJECTS

SUPPLEMENT TO THIRD PROGRESS REPORT

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by

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BIBLIOGRAPHY ON PROPYLENE CARBONATE AND γ -BUTYROLACTONE

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Preface

In the investigation of new high energy electrochemical systems the cyclic esters such as propylene carbonate and γ -butyrolactone have been demonstrated to possess exceptional characteristics. This stems from their chemical, physical, and electrochemical properties. With respect to electrochemical properties, it has been observed that the decomposition voltages for solutions of lithium perchlorate in these solvents are in the 5-6 volt range.

The employment of these solvents in electrochemical systems requires a rather complete knowledge of the chemistry of the compounds. For example, it is intended to use organic depolarizers at cathodes in certain cells. The question arises as to possible condensations, couplings, etc. that these organic depolarizers can undergo with the cyclic esters. Further, it has been observed that aluminium chloride forms highly conducting solutions in these solvents. However, aluminium chloride also catalyzes certain condensations and decompositions of the cyclic esters. It must be known how these catalytic reactions affect the stability of the electrolytic solutions.

Much work has been done and reported in the literature on questions of this type and the purpose of this bibliography is to organize the references in the open literature so that all this previous work will be available. It is expected that this bibliography will furnish a good starting point for future investigations on these solvent systems.

Whereas, the bibliography is fairly extensive, many references in the categories set out below were not included because they did not appear pertinent to the topic of interest, namely, the investigation of the electro-

chemistry of high energy compounds in organic electrolytes. On the other hand, some references were included because they appeared to discuss analogous reactions of interest.

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1. 370-480 mg. in 250 ml. of water.

2. Take 10 ml. and dilute to 100 ml. with water.

3. Take 5 ml. and add 10 ml. hydroxylamine-hydrochloride/sodium hydroxide reagent (25 ml. 3N ~~hydroxylamine-hydrochloride~~ with two drops indicator containing 100 mg. each of phenolphthalein and thymolphthalein in 100 ml. 95% ethanol, 3N sodium hydroxide added to turn purple, then 10-20 drops of indicator added).

4. Heat 20 min. at 40°, cool and shake with 10 ml. ferric alum reagent (25 g. alum in 125 ml. 3.5N nitric acid up to 250 ml. with water), then let stand 10 min.

5. Absorbancy at 505 millimicrons.

% by weight = (11093 A + 77.5)/mg. sample.

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Ber. deut. chem. Ges. 75B, 121-3 (1942). C. A. 37, 2717 (1943).
Synthesis of substituted butyrolactones.

Ladd, E. C. and Harvey, M. P.
 γ -Lactones.
U. S. 2,569,064, Sept. 25, 1951. C. A. 46, 5077 (1952).
Synthesis of substituted butyrolactones.

Linville, R. G. and Elderfield, R. C.
Synthesis of β -substituted $\Delta^{\alpha,\beta}$ -butenolidenes from omega-acetoxy-methyl ketones.
J. Org. Chem. 6, 270-2 (1951). C. A. 35, 403 (1941).
Synthesis of some substituted butyrolactones.

Mc Elvain, S. M. and Laughton, P. M.
Piperidene derivatives. XXIV. 1-methyl-4-phenyl-3-piperidone and related compounds.
J. Am. Chem. Soc. 73, 448-52 (1951). C. A. 46, 982-3 (1952).
C. A. 45, 1592 (1951).
Synthesis of some substituted butyrolactones.

Mc Graw, W. J.,
 α -Acyl, α -(α' -hydroxyalkyl), and α -alkylidene lactones.
U. S. 2,624,723, Jan. 6, 1953. C. A. 47, 11232-3 (1953).
Synthesis of several butyrolactones.

Michael, F. and Jung, F.

Hydroxytetronic acid.

Ber. deut. chem. Ges. 67B, 1660-4 (1934). C. A. 29, 456-7 (1935).

C. A. 27, 5723 (1933).

Formation of γ -butyrolactone derivatives.

Mumm, O. and Brodersen, K.

Course of reduction of pyridinecarboxylic acids to nitrogen-free products.

Ber. deut. chem. Ges. 56B, 2295-2301 (1923). C. A. 18, 1128 (1924).

Production of several butyrolactones.

Nemec, J. W.

Substituted butyrolactones.

U. S. 2,839,538, June 17, 1958. C. A. 52, 17114 (1958).

Synthesis.

Obata, S.

γ -Butyrolactone derivatives. I. Synthesis of γ - and α , γ -substituted butyrolactones. II. Synthesis of β , γ - and α , β , γ -substituted butyrolactones. III. Synthesis of γ , γ -substituted butyrolactones. IV. Relation between chemical structure and odor of butyrolactone derivatives.

J. Pharmacol. Soc. Japan 75, 1295-1306 (1953). C. A. 49, 175-6 (1955).

Initial reactions are with zinc metal.

Patel, N. Z. and Nargund, K. S.

Synthetic anthelmintics. XII, XIII. 4-alkoxy-2-tolyl- γ -butyrolactones, 2-alkoxy-5-tolyl- γ -butyrolactones.

J. Univ. Bombay, Sect. A, 15, Pt. 3, Sci. No. 20, 31-41 (1946).

C. A. 41, 6200-01 (1947).

Synthesis.

α -Amino- γ -butyrolactones.

Brit. 659,483, Oct. 24, 1951. C. A. 47, 3338, 606 (1953).

α -Hydroxy- β , β -dimethyl- γ -butyrolactone.

Swiss. 215,143, Sept. 1, 1941. C. A. 42, 3775 (1948).

Preparation.

Pattison, F. L. M. and Saunders, B. C.

Toxic fluorine compounds containing the carbon-to-fluorine link. VII. Evidence for the β -oxidation of omega-fluoro carboxylic acids in vivo.

J. Chem. Soc. 1949, 2745-9. C. A. 44, 3887 (1950).

Preparation of β , β -dimethyl- γ -butyrolactone.

Puetzer, B., Nield, C. H., and Barry, R. H.

Synthesis of a clavacin isomer and related compounds.

J. Am. Chem. Soc. 67, 832-7 (1945). C. A. 39, 2995-7 (1945).

disodium salt of (ortho-hydroxyphenyl-carbonyl)pyruvic acid ester = α -keto- β -(ortho-hydroxyphenyl-carbonyl)butyrolactone in the presence of water and formaldehyde.

Rambaud, R. and Ducher, S.

γ -Isocrotonolactone.

Bull. soc. chim. France 1956, 466-76. C. A. 50, 14535 (1956).
Synthesis of some substituted γ -butyrolactones as intermediates.

Rosenmund, K. W., Zymalkowski, F., and Engels, P.

Synthesis of γ -(hydroxyphenyl- and dihydroxyphenyl)butyrolactones.

Chem. Ber. 84, 711-18 (1951). C. A. 46, 3012-20 (1952).

Rothe, J. and Zimmer, H.

Substituted γ -lactones. III. General route to β -substituted- γ -butyrolactones.

J. Org. Chem. 24, 586-9 (1959). C. A. 54, 6541, 18421-2 (1960).

Salmon-Legagneur, W.

2, 2-diphenylbutanoloic isomers and their olides.

Compt. rend. 227, 437-9 (1948). C. A. 43, 3396-7 (1949).

Preparation of β , β -diphenyl- γ -butyrolactones.

Saotome, K. and Yamazaki, T.

γ -Alkylbutyrolactones.

Japan 26, 417-(1965), Nov. 16, Appl. July 27, 1963; 2 pp. C. A. 64, 9600 (1966).

Hexanol + ethyl acrylate + t-butyl peroxide 150-155° for 1 hr.;
 γ -amylbutyrolactone.

Schroeter, G.

ortho-phenol fatty acids and their derivatives.

Ger. 562,827, Dec. 21, 1928. C. A. 27, 1224-5 (1933).

Discusses the synthesis of several substituted γ -butyrolactones.

Schuster, C. and Simon, A.

Chlorobutyrolactones.

Ger. 810,025, Aug. 6, 1951 (Cl. 12o, 11). C. A. 47, 2769 (1953).

Schuster, C.

α -Amino- γ -butyrolactones.

Ger. 812,077, Aug. 27, 1951 (C. 12q, 6o₁). C. A. 47, 606 (1953).

Schuster, C.

α -Amino- γ -butyrolactones.

U. S. 2,605,268, July 29, 1952. C. A. 47, 3872, 606 (1953).

Soffer, M. D. and Hunt, M. C.

Methyl esterification of an easily lactonized hydroxy acid.

J. Am. Chem. Soc. 67, 692-3 (1945). C. A. 39, 2498 (1945).

Synthesis of a β -substituted γ -butyrolactone.

Sonne, W.

Substituted γ -butyrolactones.

Ger. 1,018,856, Nov. 7, 1957 (Cl. 12o). C. A. 54, 1364 (1960).

Preparation.

Stiller, E. J., Kerenztesy, J. C., and Finkerstein, J.
VI. Isolation and structure of the lactone moiety.
J. Am. Chem. Soc. 62, 1779-84 (1940). C. A. 34, 6224-5 (1940).
Isolation of a substituted butyrolactone.

Sudo, R., Akiyama, Y., Kato, T., and Ohta, M.
Studies of γ -butyrolactone derivatives. I. Synthesis of α -
amino- γ -butyrolactone.
J. Chem. Soc. Japan, Pure Chem. Sect. 74, 1009-11 (1953). C. A.
49, 6829 (1955).
Synthesis.

Tomita, M., Fushimi, J., and Shibakawa, M.
Synthesis of diphenyl ester butyrolactones.
J. Pharmacol. Soc. Japan 64, No. 11A, 65 (1944). C. A. 45,
5660 (1951).
Synthesis via the Friedel-Crafts reaction.

Trivedi, J. J. and Nargund, K. S.
Synthetic anthelmintics. II. γ -Substituted butyrolactones.
J. Univ. Bombay 10, Pt. 3, 99-101 (1941). C. A. 36, 3801 (1942).
Synthesis.

Trivedi, J. J. and Nargund, K. S.
Synthetic anthelmintics. V. γ -(para-alkoxyphenyl)butyrolactones.
J. Univ. Bombay 11, Pt. 3, 127-30 (1942). C. A. 37, 2005-6 (1943).
Preparation.

Van Tamelen, E. E. and Bach, S. R.
 α -Methylene- γ -phenyl- γ -butyrolactone.
J. Am. Chem. Soc. 77, 4683-4 (1955). C. A. 50, 6381 (1956).
Synthesis.

Vyas, V. A., Bokil, K. V., and Nargund, K. S.
Synthetic anthelmintics. I. α -Substituted- γ -butyrolactones.
J. Univ. Bombay 9, Pt. 3, 145-9 (1940). C. A. 35, 6574-5 (1941).
Synthesis.

Wachs, H.
Synthesis of a new lactone.
Riechstoffind 7, 196-7 (1932). C. A. 27, 2685 (1933).
Preparation of γ -tolyl- γ -butyrolactone.

Wagner, A. F., Walton, E., Hoffman, C. H., Peterson, L. H.,
Holly, F. W., and Folkers, K.
Synthesis of DL-dimethyldihydro- α -lipoic acid.
J. Am. Chem. Soc. 77, 5140-3 (1955). C. A. 50, 8456-8 (1956).

Walton, E. and Green, M. B.
Production of 3-aryl-1, 5-dibromopentane-3-carboxylic acid.
U. S. 2,421,729, June 3, 1947. C. A. 41, 5550 (1947).

Weizmann, C., Bergmann, A. B., and Bergmann, F.
Reactions of Grignard reagents with some succinic anhydrides.
J. Chem. Soc. 1935, 1370-1. C. A. 30, 94 (1936).

Weston, A. W.

Amino alkyl phenyl lactones.

U. S. 2,409,937, Dec. 13, 1949. C. A. 44, 2516 (1950).

Synthesis of some α , α -disubstituted- γ -butyrolactones.

Zimmer, H., Rothe, J., and Holbert, J. M.

Substituted γ -lactones. V. Synthesis of certain α , β -disubstituted γ -lactones. Route to lignans of the α , β -dibenzylbutyrolactone class.

J. Org. Chem. 25, 1234-5 (1960). C. A. 55, 5409, 2560 (1961).

Makes use of sulfuric acid.

D. Reactions of Unsubstituted γ -Butyrolactones.

Berti, F. A.

Amino derivatives of γ -butyrolactone.

Gazz. chim. ital. 84, 420-7 (1954). C. A. 49, 8120-1 (1955).

Bischoff, G.

α , γ -Dibromobutyryl bromide.

Swiss. 260,302, July 1, 1949 (Cl. 360). C. A. 44, 2549 (1950).

Butyrolactone + red phosphorus + Br₂ $\xrightarrow{20-60^\circ}$ Title product.

Bischoff, G.

α -Bromobutyrolactone.

Swiss. 264,598, Jan. 16, 1950 (Cl. 360). C. A. 45, 1622 (1951).

At 160-170° for 8 hours followed by cooling and distillation under reduced pressure.

Blicke, F. F. and Brown, B. A.

Interaction of an Ivanov and an Ivanov-like reagent with γ -butyrolactone and γ -valerolactone.

J. Org. Chem. 26, 3685-91 (1961). C. A. 56, 5909-11 (1962).

Reactions.

Dashunin, V. M., Maeva, R. V., Kazaletova, G. A., and Belov, V. N.

Substituted lactones and their transformations. III. Hydrogenation of the aromatic nucleus in α -alkylidenebutyrolactones.

Zhur. Obsheei Khim. 34 (9), 3096-3101 (1964). C. A. 61,

15984-5 (1964). C. A. 58, 470 (1963).

γ -Butyrolactone reacts with aldehydes to yield α -alkyl derivatives.

Dashunin, V. M., Samotuga, G. A., and Belov, V. N.

Substituted lactones and their transformations. V. Hydroxy and oxo derivatives of α -cyclohexylmethylbutyrolactone.

Probl. Organ. Sintez, Akad. Nauk. S. S. S. R., Otd. Obsheei i Tekhn. Khim. 1965, 64-8 (Russian). C. A. 64, 6513 (1966).

C. A. 63, 17980 (1965).

Ebel, F. and Weissbarth, O.

α -Acyl lactones.

Ger. 801, 276, Dec. 28, 1950 (Cl. 120, 15). C. A. 45, 2972-3 (1951).

γ -Butyrolactone reacts with esters in the presence of sodium metal to yield α -acyl derivatives.

Henry, L.

On butyrolactones and unsymmetrical dimethyltetramethyleneglycol.
Compt. rend. 143, 1221-25. C. A. 1, 714-15 (1907).

γ -Butyrolactone reacts with the appropriate Grignard reagent to yield α , α -dimethyltetrahydrofuran.

Isham, R. M.

Succinic and maleic acids from butyrolactones.

U. S. 2,385,518, Sept. 25, 1945. C. A. 40, 1173 (1946).

Oxidation of butyrolactone in the vapor phase with V_2O_5 or CuO .

Isham, R. M.

Electrolytic oxidation of butyrolactone to succinic acid.

U. S. 2,420,954, May 20, 1947. C. A. 4727 (1947). C. A. 40, 1173 (1946).

Kadyrov, Ch. Sh.

Synthesis of arylbutyric acids. I. Reaction of benzene and chlorobenzene with butyrolactone.

Uzbeesk. Khim. Zhur. 8 (2), 52-7 (1964) (Russian). C. A. 61, 4254 (1964).

γ -Butyrolactone reacts with benzene in the presence of aluminum chloride to yield β -phenylbutyric acid.

Knobler, Y., Livergand, S., and Frankel, M.

Preparation of O-phenyl-DL-homoserine and of DL-homoserine from α -phthalimido- γ -butyrolactone.

J. Org. Chem. 24, 1794-5 (1959). C. A. 55, 13325 (1961).

Uses sodium metal, phenol, and γ -butyrolactone.

Korte, F. and Machleidt, H.

Syntheses in the pyran series. I. Rearrangement of α -hydroxymethylene- δ -lactones to 2-methoxy-3-tetrahydropyrancarboxylic acid esters.

Chem. Ber. 88, 136-43 (1955). C. A. 50, 5615-16 (1956). C. A. 49, 12453 (1955).

γ -Butyrolactone and ethyl formate and sodium in ether yield α -hydroxymethyl- γ -butyrolactone.

Krzikalla, H. and Dornheim, O.

Esters of etherified aliphatic hydroxy carboxylic acids.

Ger. 745,312, Dec. 2, 1943 (Cl. 120, 11). C. A. 40, 3130 (1946).

Synthesis from butyrolactone in the presence of acids.

Krzikalla, H., Plieninger, H., and Maier, K.

Lactone-like products.

Ger. 844,292, July 17, 1952 (Cl. 120, 11). C. A. 52, 10199 (1958).

Synthesis of benzylidene derivatives of γ -butyrolactone.

Mayhew, R. L. and Williams, E. P.

Nematocidal α -halogenated- γ -butyrolactones.

U. S. 2,974,084, March 7, 1961. C. A. 55, 14807 (1961).

Michael, A. and Weiner, N.

Formation of enolates from lactonic esters.

J. Am. Chem. Soc. 58, 999-1005 (1936). C. A. 30, 5183-4 (1936).
 γ -Butyrolactone does not react with sodamide in diethyl ether.
It reacts with pulverized sodium or with 40% sodium amalgam only
slightly upon long standing. No experimental data is given to
substantiate this statement.

Oelshlaeger, H., Schmersahl, P., and Toporski, W.
Synthesis of new local anesthetics. IV. γ -Halobutyric acids
from γ -butyrolactone and several new omega-amino-3-alkyl-6-halo-
acrylanalides.

Arch. Pharmacol. 294, 488-98 (1961). C. A. 56, 3342-3 (1962).

C. A. 55, 19835 (1961). C. A. 53, 3136 (1959).

γ -Butyrolactone + SOCl_2 = γ -chlorobutyryl chloride.

γ -Butyrolactone + HCl = γ -chlorobutyric acid.

γ -Butyrolactone + HBr = γ -bromobutyric acid.

γ -Butyrolactone + HCO_2H = pentanedioic acid.

γ -Butyrolactone + HI with P_2O_5 = γ -iodobutyric acid.

Petrov, G. I.

Free radical addition of butyrolactone and unsaturated compounds.

Izvest. Akad. Nauk S. S. S. R., Otd. Khim. Nauk. 1962, 146-51.

C. A. 57, 16390 (1962). C. A. 56, 308, 7155 (1962). C. A. 55,
22093 (1961).

Plieninger, H.

Cleavage of γ -butyrolactone and α -amino- γ -butyrolactone with
sodium methyl mercaptide or selenide. A synthesis of methionine.

Chem. Ber. 83, 265-8 (1950). C. A. 44, 9919 (1950).

γ -Butyrolactone + $\text{RSNa} \rightarrow \text{RS}(\text{CH}_2)_3\text{COOH}$.

Rimbaud, R., Ducher, S., and Boudet, R.

Butenolide. III.

Bull. soc. chim. France 1956, 1419-24. C. A. 51, 3602 (1957).

C. A. 25, 4850 (1931).

Reactions.

Schotte, L.

γ -Butyrolactone reactions involving ring fission.

Arkiv. Kemi 8, 457-61 (1955). C. A. 51, 1838 (1957).

Späth, E. and Lintner, J.

Formation of lactams from lactones.

Ber. deut. chem. Ges. 69B, 2727-31 (1936). C. A. 31, 2172 (1937).

γ -Butyrolactone to γ -butyrolactam.

Stacey, M., Barker, S. A., Ward, R. B., Grant, P. M., and
Lloyd, I. R. L.

Polyamino and polyhydroxycarboxylic acids.

Brit. 901,037, July 11, 1962, Appl. Jan. 7, 1959; 8 pp. C. A.

58, 3519 (1963).

Polymerization of γ -butyrolactone by irradiation.

Stepanov, F. N.

3-indolebutyric acid.

U. S. S. R. 66,681, July 31, 1946. C. A. 41, 2087 (1947).

Suzuki, K.

Synthesis of resorcylobutyrolactone mono- and dimethyl esters.
Bull. Inst. Phys. Chem. Research (Tokyo) 15, 71 (1936). C. A. 31, 6640 (1937).

Treatment of the acid form with sodium amalgam and dilute acetic acid results in formation of the lactone.

Takayama, Y. and Mizuno, S.

Formation of succinic acid from tetrahydrofurfuryl alcohol and γ -butyrolactone by electrolysis.

J. Electrochem. Soc. Japan 18, 298-301 (1950). C. A. 45, 8923 (1951).

Talbot, G, Gauaby, R., and Berlinguet, L.

Synthesis of 4-aminobutyric acid and 2, 4-diaminobutyric acid from butyrolactone.

Can. J. Chem. 36, 593-6 (1958). C. A. 52, 15425-6 (1958).

Tani, H. and Fudo, K.

Reaction of γ -butyrolactone with alkyl mercaptans.

Mem. Inst. Sci. Ind. Res., Osaka Univ. 6, 100-1 (1948). C. A. 45, 10198 (1951).

Wagner, A. F.

Substituted butyric acids.

U. S. 2,842,587, July 8, 1958. C. A. 52, 18221 (1958).

γ -Butyrolactone reacts with PBr_3 at 90-135 $^\circ$ to yield α , γ -dibromobutyric acid.

Wagner, A. F.

Addition product of metal hydrocarbon mercaptide with an α -halo- γ -butyrolactone.

U. S. 2,872,458, Feb. 3, 1959. C. A. 53, 12184 (1959).

Yur'ev, Yu. K., Vendel'shtein, E. G., and Zinov'eva, L. A.

Pyrrole derivatives. XXXV. Transformation of butyrolactone into 2-thiophanone, 2-pyrrolidone, and 1-phenyl-2-pyrrolidone.

Zhur. Obschchei Khim. (J. Gen. Chem.) 22, 509-13 (1952). C. A. 47, 2747-8 (1953). C. A. 46, 8086 (1952). C. A. 44, 5869 (1950).

Zimmer, H. W. and Holbert, J. M.

Butyrolactone derivatives.

U. S. 3,030,361, April 17, 1962, Appl. July 14, 1958; 5 pp. C. A. 57, 9742-3 (1962).

Zimmer, H. W. and Walter, R.

Substituted γ -lactone. XX. Reaction of α -benzylidene- γ -butyrolactones with bromine and some chemical properties of the resulting products.

J. Heterocyclic Chem. 2(4), 477-8 (1965) (English). C. A. 64, 9631 (1966). C. A. 63, 1728 (1965). C. A. 63, 4236 (1965).

E. Reactions of Substituted γ -Butyrolactones.

Bremer, K. H. and Seidel, L.

The enolization of γ -lactones.

Angew. Chem. 76 (9), 376 (1964). C. A. 61, 1751-2 (1964).

Grignards.

Bryusova, L. Ya., Simanovskaya, E., and Ul'yanova, A.

Substituted butyrolactones and their transformation into cyclopentanone derivatives.

Sintezy Dushistykh Veshchestv, Sbornik Statei 1939, 165-77;

Khim. Referat. Zhur. 1940, No. 4, 115. C. A. 36, 3784 (1942).

Synthesis.

Curtius, T. and Sauerberg, H.

Action of hydrazine upon butyrolactone- α -carboxylic esters.

J. prakt. chem. 125, 139-51 (1930). C. A. 24, 3215 (1930).

Carbethoxybutyrolactone + hydrazine + water = ethyl β -hydroxyethylmalonyl hydrazine. Carbethoxybutyrolactone + an excess of hydrazine + water = the dihydrazide. Carbethoxybutyrolactone + nitrous acid = butyrolactone- α -carboxylic azide. Carbethoxybutyrolactone + para-methylaniline = β -hydroxyethyl-malonyl di-para-toluidide.

Feofilaktov, V. V. and Onishchenko, A. S.

Action of nitrous acid on α -substituted butyrolactones. I.

J. Gen. Chem. (U. S. S. R.) 9, 304-13 (1939). C. A. 34, 378 (1940).

Action depends on the nature of the α -substituent.

Feofilaktov, V. V. and Onishchenko, A. S.

Action of phenyl diazonium chloride on α -substituted butyrolactones. II.

J. Gen. Chem. (U. S. S. R.) 9, 314-24 (1939). C. A. 34, 378-9 (1940).

Similar to the action of nitrous acid but no polymerization or isomerization.

Fischhoff, G.

Derivatives of α -keto- γ -butyrolactone. Ethyl ester of α -keto- γ -butyrolactone- β -carboxylic acid (α -keto-paraconic ester) and its β -hydroxymethyl derivative.

Ann. chim. (12), 6, 227-45 (1951). C. A. 45, 9475-78 (1951).

Gault, H.

Derivatives of α -keto- γ -butyrolactone. γ -Alkyl- and γ -aryl- α -keto- γ -butyrolactone- β -carboxylic esters. Introduction.

Ann. chim. (12), 6, 220-6 (1951). C. A. 45, 9475-78 (1951).

Harradence, R. and Lions, F.

Indoles. VIII. 3-hydroxymethylindole-2-carboxylic lactone.

J. Proc. Royal Soc. N. S. Wales 72, 221-7 (1939). C. A. 33, 6338-9 (1939).

Acetobutyrolactone is the starting material.

Kawasaki, H.

Vitamin B₁ and related compounds. LXXVI. Thiotype thiamine

derivatives. 7. S-acylation of thiazolium compounds.
J. Pharm. Soc. Japan 76, 702-5 (1956). C. A. 51, 429-30 (1957).
C. A. 50, 13044 (1956).

Treat α -benzoylbutyrolactone with SO_2Cl_2 to get α -chloro derivatives.

Kohles, E. P. and Kimball, R. H.
Hydroxy derivatives of α , γ -diphenylbutyric acid.
J. Am. Chem. Soc. 55, 4632-9 (1933). C. A. 28, 130 (1934).

Krzikalla, H. and Tartter, A.
Sulfur-containing lactone derivatives.
Ger. 801,992, Feb. 1, 1951 (Cl. 120, 2303). C. A. 45, 5179 (1951).

Mitchell, H. K., Weinstock, H. H., Jr., Snell, E. E., Stanbery, S. R.,
and Williams, R. J.

Pantothenic acid. V. Evidence for structure of non- β -aniline portion.

J. Am. Chem. Soc. 62, 1776-9 (1940). C. A. 34, 6224 (1940).
C. A. 33, 6368 (1939).

Decomposition of hydroxyl substituted butyrolactones using sulfuric acid and/or potassium permanganate.

Moss, A. R.
A resolution in the pantothenic acid series.
Jubilee Vol. Emil Borell 1946, 79-81 (in English). C. A. 41,
6199 (1947).

Synthesis from substituted butyrolactones and Raney nickel.

Nef, J. U.
Dissociation reactions of the sugar group. II. Behavior of sugars towards caustic alkalies.
Ann. chim. 376, 1-119. C. A. 5, 85-8 (1911). C. A. 2, 799 (1908).
dl-1-hydroxybutyrolactone + HNO_3 = dl-malic acid.
dl-2-hydroxybutyrolactone + HNO_3 = brucine salt.

Opferman, A. J.
2-hydroxybutyrolactone.
Brit. 688, 253, March 4, 1953. C. A. 48, 3996-7 (1954).
Synthesis from the 2-bromo derivative in two steps.

Ozawa, T. and Nagaoka, S.
New utilization of α -acetyl- γ -butyrolactone. I. A new synthesis of quinoline derivatives.
J. Pharm. Soc. Japan 75, 1405-7 (1955). C. A. 50, 10001-2 (1956).

Ozawa, T., Nagaoka, S., Mitsuno, K., and Tsukiyama, T.
New Utilization of α -acetyl- γ -butyrolactone. II. A new synthesis of quinoline derivatives.
J. Pharm. Soc. Japan 75, 1407-10 (1955). C. A. 50, 10001-2 (1956).

Ozawa, T. and Nagaoka, S.
Syntheses of heterocyclic compounds derived from 2-acetyl- γ -butyrolactone. V. Synthesis of dihydrofuro[3, 2-c] quinoline

compounds.

Yakugaku Zasshi 79, 226-30 (1959). C. A. 53, 13146-8 (1959).
C. A. 51, 8749 (1957).

Halogenated organic compounds.

Brit. 490, 571, Aug. 17, 1938. C. A. 33, 813 (1939).
Halogenation of α -aceto- γ -butyrolactone.

Butyrolactones.

Brit. 600, 865, April 21, 1948. C. A. 42, 7324 (1948). C. A.
40, 1872 (1946).

Pantothenic acid.

Swiss. 215, 779, Oct. 16, 1941. C. A. 42, 4606 (1948).

Price, C. C. and Kaplan, W.

Reactions of γ -anisyl- γ -butyrolactone with potassium cyanide.
6-Methoxy-1, 2, 3, 4-tetrahydro-2-naphthoic acid.
J. Am. Chem. Soc. 66, 477-8 (1944). C. A. 38, 2031-2 (1944).
Preparation.

Schnider, O.

Synthesis of panthenol and its transformation into pantothenic
acid.

Jubilee Vol. Emil. Barell 1946, 85-91 (in German). C. A. 41,
6199 (1947).

Synthesis from substituted butyrolactones and Raney nickel.

Stepanov, F. N. and Smirnov, O. K.

Butyrolactone and its derivatives. II. Some reactions of acyl
derivatives of butyrolactone.

Zhur. Obsheei Khim. 27, 1041-4 (1957). C. A. 52, 2747-8 (1958).
C. A. 51, 7344 (1957).

α -Acetyl- γ -butyrolactone + sodium metal refluxed with methanol
and methyl iodide yields α -acetyl- α -methyl- γ -butyrolactone.

Stepanov, F. N. and Smirnov, O. K.

Butyrolactone and its derivatives. II. Some reactions of acyl
derivatives of butyrolactone.

J. Gen. Chem. U. S. S. R. 27, 1124-6 (1957) (in English).
C. A. 53, 4126 (1959). C. A. 52, 2447 (1958).

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Studies on γ -butyrolactone derivatives. III. Acylation of α -
amino- γ -butyrolactone.

Nippon Kagaku Zasshi 79, 81-4 (1958). C. A. 54, 5448 (1960).
C. A. 49, 12298 (1955).

Sudo, R.

γ -Butyrolactone derivatives. VI. Reactions of α -amino- γ -
butyrolactone with aromatic aldehydes.

Nippon Kagaku Zasshi 80, 924-6 (1959). C. A. 55, 4528 (1961).
C. A. 54, 5448 (1960).

Sudo, R.

γ -Butyrolactone derivatives. V. The α -ureido- and α -thioureido- γ -butyrolactone derivatives.

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C. A. 54, 5448 (1960).

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Preparation of 5-alkyl-5-(2-phenyl- and 2-vinyl-2-hydroxyethyl) barbituric acid via the corresponding α -carbethoxy- γ -butyrolactone.
J. Am. Chem. Soc. 72, 1357-9 (1950). C. A. 44, 5816-7 (1950).

Zumbrunn, R.

α -Chloro- α -acetyl- γ -butyrolactone.

U. S. 2,440,229, April 20, 1948. C. A. 42, 5467 (1948).
Preparation from the unchlorinated compound.

F. Hydrolysis.

(a) General.

Adickes, F.

Addition of alkali alcoholates to acid esters. II.

Ber. deut. chem. Ges. 59B, 2522-33 (1926). C. A. 21, 734 (1927).
C. A. 20, 737 (1926).

The addition of alcoholate can apparently always take place when the grouping RCOCOR' is present, even when none of the carbon atoms forms part of a COOH group, but one of these CO unions cannot be replaced by an aliphatic carbon-to-carbon double bond.

Adickes, F.

Addition of alkali alcoholates to acid esters. III. Addition of alkali alcoholates to formic esters.

Ber. deut. chem. Ges. 60B, 272-7 (1927). C. A. 21, 1628 (1927).

Adickes, F. et al.

Addition of alkali alcoholates to acid esters. IV. Addition of sodium ethanolate to formic acid esters.

Ber. deut. chem. Ges. 63B, 3012-27 (1930). C. A. 25, 2115-16 (1931). C. A. 21, 1628, 1795 (1927). C. A. 20, 2824 (1926).

Adickes, F. and Sch#fer, G.

Kinetics and statics of the decomposition reaction of ethyl formate by sodium ethoxide.

Ber. deut. chem. Ges. 65B, 950-5 (1932). C. A. 26, 4793 (1932).
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Kinetics of reactions in moderately concentrated aqueous acids. II. An empirical criterion of mechanism.

J. Am. Chem. Soc. 83, 4968-73 (1961). C. A. 57, 1598 (1962).

Evans, D. P., Gordon, J. J., and Watson, H. B.

Influence of alkyl groups upon reaction velocities in solutions. The alkaline hydrolysis of saturated aliphatic esters.

J. Chem. Soc. 1938, 1439-44. C. A. 33, 457 (1939).
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Gyngell, E. S.

The alkaline hydrolysis of esters in aqueous alcohols. I. The interaction of phenoxides and aliphatic esters in alcoholic solutions.

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Potassium phenoxide + Ethyl acetate = ethyl phenoxide + potassium acetate.

There is also no evidence in favor of the view that the reaction proceeds as a result of "alcoholysis" of the phenoxide. The reaction is a hydrolysis of the phenoxide by water.

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Lukes, R., Nemeč, J., and Jury, J.

Lactones. IV. Alkaline hydrolysis of α -hydroxy- β -bromo- γ -valerolactone.

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Z. physik. Chem. 133, 233-52 (1928). C. A. 23, 1337 (1929).

C. A. 21, 1581 (1927).

Scheibler, H. and Voss, J.

Metal compounds of the enol forms of carbonyl compounds and their use for syntheses. I. Preparation and properties of the metal compounds of the enol forms of aliphatic acid esters.

Reaction of potassioacetic ester with halogen-acyl compounds.

Ber. deut. chem. Ges. 53B, 388-409 (1920). C. A. 14, 2931-2 (1920). C. A. 9, 3245 (1915).

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Wegscheider, R. and Amann, W. W.

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Monatsh. Chem. 36, 549-610. C. A. 9, 3007 (1915).

F. Hydrolysis.

(b) γ -Butyrolactone.

Arendaruk, A. P., Serebryakov, L. A., Skoldinov, A. P.

Synthesis and some problems of pharmacodynamics of γ -amino- and γ -hydroxybutyric acids.

Med. Prom. S. S. S. R. 17 (6), 6-8 (1963). C. A. 59, 11234 (1963).

To a hot solution of 225 ml. of rectified ethanol, 45 ml. water, and 220 g. sodium hydroxide, 489 g. γ -butyrolactone was slowly added with stirring and the mixture kept boiling for 30 min. The precipitate was recrystallized from ethanol. Yield = 560 g. salt.

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Analgesics. II. Synthesis of amidone and some of its analogs.
J. Chem. Soc. 1949, 510-18. C. A. 43, 9053-4 (1949).
Preparation of the sodium salt of α , α -diphenyl- γ -butyrolactone.

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Concurrent alkaline hydrolysis and isotopic oxygen exchange of
several alkyl benzoates and lactones.
J. Am. Chem. Soc. 83, 4193-6 (1961). C. A. 56, 14158-9 (1962).
 K_h/K_{ex} (γ -butyrolactone) = 30 (water); calculated value.

Coffin, F. D. and Long, F. A.
Arrhenius parameters for acid hydrolysis of γ -butyrolactones;
search for a reaction with water.
J. Am. Chem. Soc. 74, 5767-8 (1952). C. A. 47, 10977 (1953).
C. A. 45, 9345 (1951).

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hydroxybutyrate with benzyl alcohol.
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C. A. 56, 311 (1962).
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Hollo, E.
Influence of the oxygen atom of the ring on the reaction velocity
of several lactones.
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water, and acetone solutions at 25°.

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Ester catalysis of γ -lactones.
Ber. deut. chem. Ges. 51, 480-5 (1918). C. A. 12, 2566 (1918).
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normal case of ester catalysis.

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Formation and hydrolysis of lactones.
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acid solution is very much less than that of the hydrogen ion of
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Kohler, E. P. and Conant, J. B.
Cyclopropane series.
J. Am. Chem. Soc. 39, 1404-20 (1917). C. A. 11, 2194-6 (1917).
Alcoholic sodium hydroxide and β -benzoyl- γ -phenylbutyrolactone.

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Determination of the mechanisms of γ -lactone hydrolysis by a
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J. Am. Chem. Soc. 72, 3692-5 (1950). C. A. 45, 1514 (1951).
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Spencer, E. Y. and Wright, G. F.
Action of sodium alcoholate on lactones.
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 γ -hydroxy-COOR.

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Acid catalysis in lactone formation.
J. Phys. Chem. 29, 1085-92 (1925). C. A. 19, 3411 (1925).
C. A. 18, 2219 (1924). C. A. 11, 1811 (1917).
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Wheeler, O. H. and Gamble, D. S.
Saponification of methyl substituted γ -butyrolactones.
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Compounds and acetoacetic ester condensation.
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Mechanism.

Antia, M. B. and Kaushal, R.
Reformatskii condensation of ketonic esters with halo esters.
Agra Univ. J. Res., Sci. 3, 197-201 (1954). C. A. 49, 6121 (1955).
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Mechanism of aldehyde, ketone, and ester condensations.
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ethanolate condensations according to Schlenck-Ziegler-Lebedev.

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Condensations of lactones with benzene.
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J. Am. Chem. Soc. 80, 6409-12 (1958). C. A. 53, 14119-20 (1959).

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Extension of the modified Stobbe condensation. Acid catalyzed
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C. A. 39, 4867-8 (1945).

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Decarboxylation and lacto-enoic tautomerism. III. Stobb condensation with para, para'-dimethoxybenzophenone.
J. Am. Chem. Soc. 72, 511-13 (1950). C. A. 45, 1985-6 (1951).
C. A. 44, 8887 (1950). C. A. 41, 2408 (1947).
Preparation.
- Morton, A. A. and Stevens, J. R.
Condensations by sodium instead of by the Grignard reaction. I. Tertiary carbinols.
J. Am. Chem. Soc. 53, 2244-7 (1931). C. A. 25, 3643 (1931).
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Condensations by sodium instead of by the Grignard reaction. II. Reaction with cyanobenzene. Preparation of diphenylketazine.
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J. Am. Chem. Soc. 54, 1919-24 (1932). C. A. 26, 3249 (1932).
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Condensations by sodium. IX. Preparation and properties of trixyenyl and trimethyltrixenyl carbinols and some of their derivatives.
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Condensations by sodium. X. Some side reactions occurring in the Wurtz synthesis and a novel formation of phenyliodide.
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Condensations by sodium. XI. Trimethoxytrixenylcarbinol and comparisons of colors of some carbonium salts in this series.
J. Am. Chem. Soc. 60, 284-5 (1938). C. A. 32, 2524 (1938).
- Morton, A. A., Fallwell, F., Jr., and Palmer, L.
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J. Am. Chem. Soc. 60, 1426-9 (1938). C. A. 32, 5807 (1938).
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Condensations by sodium. XIII. Phthalic acids and some factors

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Condensations by sodium. XXXI. Effects of salts and other substituents on the alkylation of toluene, a modified Wurtz reaction.
J. Am. Chem. Soc. 73, 4363-7 (1951). C. A. 47, 109-10 (1953).
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Condensations by sodium. XXXVII. Dimetalation of benzene, thiophene, para-t-butylphenol, i-propylbenzene and the effect of alkoxides on the meta:para ratio for benzene.
J. Am. Chem. Soc. 76, 4935-8 (1954). C. A. 49, 13169-70, 8861 (1955).

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Brit. 489, 026, July 14, 1938. C. A. 33, 282 (1939).
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Metal compounds of the enol forms of monocarbonyl compounds.
XIV. Action of sodium alcoholate on acid esters, ester condensations and substitution reactions of the metal compounds of acid esters.
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C. A. 26, 1898 (1932).
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Contribution to our knowledge of condensations produced by sodium.
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Acetoacetic ester condensation. II. Reactions of aliphatic esters with sodium.
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J. Am. Chem. Soc. 55, 416-21 (1933). C. A. 27, 955 (1933).
C. A. 25, 3625 (1931). C. A. 22, 387 (1928).

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Acyloin condensation. II. Free radical formation accompanying the decarbonylation of an aliphatic ester by sodium. J. Am. Chem. Soc. 77, 4016-19 (1955). C. A. 50, 6317-18 (1956). C. A. 47, 12270 (1953).

Wacker, A.

Condensation of esters.

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Zimmer, H., Gracian, D., and Rothe, J.

Substituted γ -lactones. IV. Some aldehyde condensations with Δ^{β} , γ -angelica with γ -valerolactone.

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Synthesis.

G. Condensations.

(b) γ -Butyrolactone.

Cannon, G. W., Carler, J. J., Jr., and Gaines, W. A.

Acylation studies. II. Condensation of γ -butyrolactone and γ -valerolactone with methyl ketones.

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the condensation of ethylene oxide with malonic ester.
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with ethyl acetoacetate.
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C. A. 35, 3627-8 (1941).
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Initiation of the polymerization of caprolactone with butyro-
lactone.
Brit. 757,539, Sept. 19, 1956. C. A. 51, 6216 (1957).
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Products of condensation of α -formyl carboxylic esters with
esters of α -halo acids. II. Carboxylation of vinylalkyl esters.
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guanidines.
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synthesis of derivatives of butyrolactone.
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Synthesis of derivatives of butyrolactone.
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Synthesis of aliphatic polyesters. VII. Cationic copolymeri-
zation of γ -butyrolactone and ϵ -caprolactone.
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coumarins by hydrogenation of α -(2-hydroxybenzylidene)- γ -
lactones.
J. Heterocyclic Chem. 1, (4), 217-8 (1964). C. A. 62, 5247,
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butyrolactones by condensation of γ -butyrolactone with aldehydes.
Hydrogenation of the condensation products.
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H. Miscellaneous.

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