

are not affected by incubation at 38° during the endogeneous respiration period.

The inhibitory activity of I and II is of an order rarely encountered with organic enzyme inhibitors. This fact would indicate that the inhibitors are acting against a highly active catalytic portion of the enzyme. In considering the nature of the inhibition shown by the compounds discussed, it should be noted that I and II completely inhibit xanthine oxidase at molar ratios of inhibitor/substrate of 1/1000 to 1/3000 while other pterines, such as xanthopterine, are highly active only in molar concentrations equal to the xanthine substrate.

Although no conclusive data are available, one possible mode of action is the antagonism of a prosthetic group of unidentified structure. The existence of a prosthetic group other than isoalloxazine-adenine dinucleotide has been postulated by Corran, *et al.*⁹ Another possibility is the

(9) Corran, Dewan, Gordon and Green, *Biochem. J.*, **33**, 1694 (1939).

competition of all inhibitors regardless of activity with the substrate for the active catalytic surface of the enzyme.

Summary

The inhibitory action of a number of simple pterines on xanthine oxidase from milk and rat liver has been studied. It has been shown that 2-amino-4-hydroxy-6-hydroxymethylpterine is as active an inhibitor as 2-amino-4-hydroxy-6-formylpterine, and the oxime of the latter compound has high inhibitory activity.

It has been shown that 2-amino-4-hydroxypterine is a better substrate than xanthopterine for the milk enzyme and that its action in this respect is similar to xanthine or hypoxanthine.

The data are self-consistent for both enzyme preparations and therefore may be of general importance.

A discussion of the data and their implication is given.

KALAMAZOO, MICH.

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[CONTRIBUTION FROM THE DEPARTMENT OF ORGANIC CHEMISTRY, THE ABBOTT RESEARCH LABORATORIES]

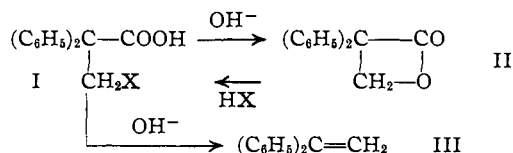
The Preparation and Behavior of α,α -Diphenyl- β -propiolactone

BY HAROLD E. ZAUGG

Many of the classic papers which have been published on theoretical organic chemistry have been concerned with reactions of β -halogen acids with various anions under a number of conditions.¹ These reactions are characterized by kinetic complexity and by the relatively large number of products formed. β -Alkoxy- and hydroxy-acids, α,β -unsaturated acids, β -lactones and ethylenes are among the products which have been reported from these reactions in aqueous or alcoholic solution.

2,2-Diphenyl-3-bromopropanoic acid (I, X = Br)² appeared to provide a structure which, on reaction with bases under suitable conditions, could be expected to give only two products, the β -lactone and the ethylene. Preparation of this acid according to the published procedure² called for purification through the sodium salt, m. p. 79–80°. Repetition of this method yielded a product, m. p. 91–92°, containing no bromine, which proved to be the β -lactone II. Indeed, treatment of the bromo acid with an equivalent amount of aqueous sodium hydroxide at room temperature for only thirty minutes gave the β -lactone in 60 to 70% yields. The expected³

by-product, 1,1-diphenylethylene (III), was always obtained in appreciable amounts, regardless of wide variation in reaction conditions.



Treatment of the β -lactone II with hydrochloric, hydrobromic and hydriodic acids in acetic acid solution resulted in formation of the three β -halogen acids I (X = Cl, Br, I, respectively). The reactivities of the mineral acids increased in the order, HCl < HBr < HI, but in all cases yields were good.

Each of these β -halogen acids was treated with an equivalent amount of aqueous sodium hydroxide (1.5 equivalents for the iodo acid) for thirty minutes at room temperature. The chloro acid gave 12% of II, no III and an 85% recovery of the acid; the bromo acid gave 65% of II, 24% (calculated by difference) of III, and the only recovered acid (11%) was that which had not been converted to the sodium salt; the iodo acid gave 25% of II, 52% of III and no recovered acid. The much slower rate of reaction of the chloro acid did not favor ethylene formation, for after three days, when the reaction was essentially complete at room temperature, a 95%

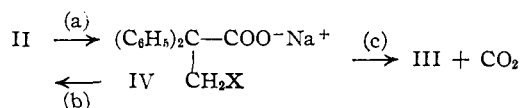
(1) (a) Walden, *Ber.*, **29**, 133 (1896); (b) Senter, *et al.*, *J. Chem. Soc.*, 1070 (1915); 1847 (1925); (c) Holmberg, *J. prakt. Chem.*, **88**, 553 (1913); (d) Olson, *et al.*, *THIS JOURNAL*, **56**, 1294 (1934); **60**, 2687 (1938); *J. Phys. Chem.*, **41**, 267 (1937); (e) Kohler, *et al.*, *THIS JOURNAL*, **56**, 729, 200 (1934); **60**, 2142 (1938); **63**, 1531 (1941).

(2) Wegmann and Dahn, *Helv. Chim. Acta*, **29**, 415 (1946).

(3) Johansson and Hagman, *Ber.*, **55**, 647 (1922).

yield of β -lactone was isolated with no detectable formation of III.

Results of a similar nature were obtained by treating the β -lactone II with excess sodium chloride, sodium bromide and sodium iodide, the ethylene III being produced in yields of 6, 54 and 86%, respectively. No trace of the β -halogen acid could be detected in any case, recovered β -lactone accounting for the bulk of the material not accounted for as III. These results not only substantiate the relative tendencies for ethylene formation observed above, but also further illustrate the reversibility of the reaction leading to the β -lactone.^{1c,4}



The presence of the carboxylate anion IV as an intermediate could not be demonstrated. Acidification of each of the three reaction mixtures, after removal of diphenylethylene and unreacted β -lactone, led to none of the corresponding β -halogen acid I. This fact indicates that in contrast to β -propiolactone itself, from which β -halopropionic acid can be isolated on treatment with sodium halide,⁴ compound II reacts with sodium halide (reaction a) at a rate much slower than the rate of decomposition of the corresponding anion IV (reaction c).⁵ Treatment of the β -lactone under similar conditions with the solvent, but in the absence of the sodium halide, resulted in complete recovery of the lactone as long as neutrality was maintained. An acidic or basic medium, however, effected hydrolysis of the lactone. This subject will be discussed in a forthcoming paper.

All of the above facts indicate that the rates of the reactions (a) and (b) increase in the order $\text{Cl} \ll \text{Br} < \text{I}$, with the greatest increase occurring in going from the chloro acid to the bromo acid. Likewise the rate of reaction (c) increases in the same order with a still greater increment observed in going from the chloro acid to the bromo acid.⁶ The evident reluctance of the chloro acid to undergo reaction (c) can be explained by analogy to the anion catalyzed mechanism for elimination of halides from 1,2-dihalides.⁷ According to this scheme, the anion IV could eliminate carbon dioxide and bromide ion by an autocatalytic (inter- or intramolecular) process at a rate dependent to a large extent on the polarizability (electron withdrawal ca-

(4) Gresham, Jansen, Shaver and Gregory, *THIS JOURNAL*, **70**, 999 (1948).

(5) When the methyl ester of the bromo acid (I) was treated with excess sodium methoxide for twenty-five hours in refluxing methanol, no appreciable reaction was observed. This behavior is quite different from the high degree of reactivity shown by the bromine atom in the anion IV.

(6) These results are in accord with the observation of Simpson, *THIS JOURNAL*, **40**, 674 (1918), regarding the stability of β -chloropropionic acid relative to the β -bromo and β -iodo acids.

(7) (a) Winstein, Pressman and Young, *ibid.*, **61**, 1645 (1939); (b) Nozaki and Ogg, *ibid.*, **64**, 704 (1942).

capacity) of the halogen atom. Whereas bromine and iodine are capable of expanding their outer rings of electrons, chlorine is reluctant to do so.⁸ This can account for the lack of ethylene formation (reaction c) from the β -chloro acid even though the competitive reaction (b) is relatively slow.

Acknowledgment.—The author is indebted to Mr. E. F. Shelberg, Head of the Abbott Microanalytical Laboratory, for the microanalyses, and to Mr. Bruce Horrom for the preparation of intermediates. Grateful acknowledgment is likewise due to Prof. W. M. Lauer, University of Minnesota, and to Prof. D. S. Tarbell, University of Rochester, for helpful advice in the preparation of the manuscript.

Experimental

α, α -Diphenyl- β -propiolactone (II). **A. From 2,2-Diphenyl-3-bromopropanoic Acid (I, X = Br).**—A suspension of 11.05 g. (0.0362 mole) of finely-ground 2,2-diphenyl-3-bromopropanoic acid (I)² in 90 cc. of water was treated with 18.1 cc. (0.0362 mole) of 2 *N* sodium hydroxide and shaken mechanically for ninety minutes. The solid product was separated by filtration and washed well with water. It weighed 7.95 g. and melted at 70–85°. Recrystallization from 50 cc. of Skellysolve C (heptane) gave 4.95 g. (61% conversion) of colorless needles, m. p. 91–92°. *Anal.* Calcd. for $\text{C}_{15}\text{H}_{12}\text{O}_2$: C, 80.33; H, 5.40; mol. wt., 224. Found: C, 80.51; H, 5.56; mol. wt. (Rast), 231. It is interesting to note that with methylmagnesium iodide in the quantitative Grignard apparatus⁹ this β -lactone was practically inert, but with lithium aluminum hydride the theoretical quantity of reducing agent for one ester group was consumed.

A small amount (1.25 g., 11%) of unreacted bromo acid (I) was recovered as a Skellysolve C insoluble fraction. Based on the bromo acid consumed, the yield of β -lactone was therefore 69%. When the reaction time of a 0.01 mole run was shortened to thirty minutes, the yield of β -lactone was unaffected, and immediate acidification with dilute sulfuric acid of the aqueous filtrate gave no recovered bromo acid.

In one run (0.01 mole) using somewhat altered conditions, 0.95 g. of a colorless neutral oil was obtained, n_D^{25} 1.6032, which had the odor of 1,1-diphenylethylene (III). Rough titration of it and of an authentic sample of III, n_D^{25} 1.6054, with bromine in carbon tetrachloride indicated it to be identical with III.

Attempts to improve the yield of β -lactone (II) by altering other reaction conditions were unsuccessful. Use of aqueous alcohol (45%), aqueous dioxane (39%) and a water-chloroform mixture (30%) as solvents reduced the yield considerably. The following conditions were also tried with the yields indicated in parentheses: equivalent aqueous sodium carbonate (47%), addition of equivalent aqueous sodium hydroxide slowly with stirring instead of in one portion (53%), excess liquid ammonia alone (0%), excess sodium acetate in refluxing benzene (22%), equivalent sodium acetate in dioxane at 55° (35%), equivalent silver nitrate in aqueous dioxane at 50° (no pure product obtained), and equivalent ethanolic potassium hydroxide in dry dioxane (57%). Aqueous barium hydroxide formed an unstable insoluble salt which decomposed to give some β -lactone together with considerable amounts of the hydrocarbon III. Only equivalent aqueous potassium, lithium and ammonium hydroxides at room temperature gave yields equal to but no greater than that obtained with sodium hydroxide.

The β -lactone seemed remarkably thermostable. It could be distilled *in vacuo*, b. p. 175–180° (15 mm.), but not without some decomposition to a liquid by-product

(8) Williams, *Trans. Faraday Soc.*, **37**, 760 (1941).

(9) Zaugg and Horrom, *Anal. Chem.*, **20**, 1026 (1948).

which was almost certainly 1,1-diphenylethylene as indicated by its unsaturation.

B. From 2,2-Diphenyl-3-chloropropanoic Acid (I, X = Cl).—To 86.2 cc. (0.04 mole) of 0.464 *N* sodium hydroxide solution was added 10.4 g. (0.04 mole) of finely-powdered 2,2-diphenyl-3-chloropropanoic acid. The mixture was shaken mechanically for two hours and then filtered. There was obtained 3.55 g. of product, m. p. 88–90°. However, on standing for three more days at room temperature, the filtrate deposited more product bringing the total to 8.51 g. (95%), m. p. 88–90°. Recrystallization from 35 cc. of Skellysolve C gave 7.76 g. (86%), m. p. 90–92°. The odor of 1,1-diphenylethylene could not be detected in this reaction and bromine in carbon tetrachloride failed to reveal unsaturation in any of the residues. When the reaction time of a 0.04 mole run was shortened to thirty minutes, only 1.12 g. (12%) of β -lactone, m. p. 86–90°, was obtained. Immediate acidification of the aqueous filtrate with dilute sulfuric acid gave 8.85 g. (85%) of starting material (chloro acid), m. p. 200–203°.

C. From 2,2-Diphenyl-3-iodopropanoic Acid (I, X = I).—To 126 cc. (0.0625 mole) of 0.496 *N* sodium hydroxide was added 14.7 g. (0.0417 mole) of finely-powdered iodo acid I (X = I). After shaking mechanically for thirty minutes in a stoppered bottle, the mixture was nearly neutral and a slight internal pressure (presumably carbon dioxide) had developed. The semi-solid product was filtered off and washed with cold pentane to give 2.35 g. (25%) of β -lactone, m. p. 90–93°. The pentane washings were separated from the aqueous filtrate and dried over anhydrous magnesium sulfate. Acidification of the aqueous filtrate with dilute sulfuric acid yielded no unreacted iodo acid.

From the pentane extract was obtained 5.5 g. (73%), calculated as $(C_6H_5)_2C=CH_2$ of a pale yellow highly unsaturated oil, which on distillation *in vacuo* gave 3.94 g. (52%) of a red oil, b. p. 137–140° (13 mm.), n_D^{20} 1.6053. The red color was due to the presence of a trace of iodine, for on shaking with a little aqueous sodium bisulfite, drying and redistilling a colorless product, b. p. 140° (13 mm.), n_D^{20} 1.6064, was obtained.

Anal. Calcd. for $C_{14}H_{12}$: C, 93.29; H, 6.71. Found: C, 93.38; H, 6.84.

For the purpose of further identification, a sample of the 1,1-diphenylethylene was converted to its maleic anhydride addition product, m. p. 276–277° (uncor.) (lit.¹⁰ m. p. 279–281° cor.), which gave no depression of melting point when mixed with an authentic sample. When only an equivalent quantity of sodium hydroxide was employed instead of the 50% excess as in the above procedure, the conversion to β -lactone occurred in essentially the same yield. However, its purification was complicated by the presence of considerable amounts of iodo acid which had not been brought into reaction.

Reaction of α,α -Diphenyl- β -propiolactone (II) with Hydrohalic Acids. A. Hydrochloric Acid.—A solution of 15 g. of the β -lactone II in 250 cc. of glacial acetic acid and 75 cc. of water was saturated with hydrogen chloride gas and heated on the steam-bath for sixteen hours with a slow stream of hydrogen chloride passing through it. The reaction mixture was then cooled in ice and filtered. Washing of the crystalline product with water and drying gave 14.7 g. (84%), m. p. 200–203° with slight decomposition. When mixed with an authentic sample of 2,2-diphenyl-3-chloropropanoic acid,¹¹ m. p. 201–203°, it gave no depression of melting point. Use of the above reaction conditions at room temperature resulted only in predominant recovery of starting material.

B. Hydrobromic Acid.—One gram of the β -lactone II was added to 25 cc. of glacial acetic acid previously saturated with dry hydrogen bromide. The β -lactone dissolved and a crystalline material began to separate. After standing at room temperature for twenty hours, the product was filtered off and washed with dilute acetic acid and water. The dry product weighed 1.27 g. (93%) and melted at

200–201° (dec.). It gave no depression of melting point when mixed with a sample of 2,2-diphenyl-3-bromopropanoic acid.²

C. Hydriodic Acid.—A solution of 14.7 g. of the β -lactone II in 200 cc. of glacial acetic acid was treated with 75 cc. of 47% aqueous hydriodic acid. After standing several minutes at room temperature, crystallization began and proceeded more rapidly than with hydrobromic acid. After standing in the dark for twenty hours at room temperature, the product was filtered off, ground to a fine powder and washed with dilute ethanol. The dry product weighed 23 g. (99%), m. p. 182–184° (dec.). Recrystallization from dilute ethanol did not improve the melting point. When the product was heated in the solvent over too long a period considerable decomposition occurred, and a sample giving completely satisfactory elementary analyses could not be obtained.

Anal. Calcd. for $C_{15}H_{13}IO_2$: C, 51.15; H, 3.72; I, 36.04. Found: C, 51.84; H, 3.89; I, 36.28.

Reaction of α,α -Diphenyl- β -propiolactone (II) with Sodium Halides. A. Sodium Iodide.—A solution of 10 g. of the β -lactone II in 350 cc. of 95% ethanol was treated with a solution of 44 g. of sodium iodide (Analytical Reagent) in 50 cc. of distilled water. The homogeneous solution was kept at 40° for one hundred and twelve hours. Dilution of the solution to a volume of four liters precipitated an oil which was taken up in ether and dried over anhydrous magnesium sulfate. Acidification of the aqueous solution with dilute sulfuric acid gave no precipitate of the iodo acid. From the dry ethereal extract was obtained 7.9 g. of a light red oil which on distillation yielded 6.89 g. (86%) of 1,1-diphenylethylene (contaminated with traces of iodine), b. p. 136–139° (13 mm.), n_D^{20} 1.6060. It was further characterized by conversion to the dibromide, m. p. 60–62°, which on heating characteristically evolved hydrogen bromide to give 1,1-diphenyl-2-bromoethene.¹²

A small scale run using 1.0 g. of the β -lactone II under identical conditions gave 0.78 g. of an unsaturated oil which decolorized 5.5 cc. of an arbitrarily standardized solution of bromine in carbon tetrachloride (7.9 cc. was decolorized by 1.0 g. of pure 1,1-diphenylethylene). This represented a 1,1-diphenylethylene content of 0.70 g. (88%) which compared favorably with the 86% yield actually isolated in the large-scale run.

B. Sodium Bromide.—A warm solution of 1.0 g. of the β -lactone II in 35 cc. of 95% ethanol was treated with a solution of 3.0 g. of sodium bromide (U. S. P.) in 15 cc. of distilled water and the mixture kept at 40° for one hundred and twelve hours. At first the β -lactone crystallized partially but then gradually dissolved.

The mixture was worked up in the usual manner. Here also no bromo acid could be obtained by acidification of the aqueous fraction with dilute sulfuric acid. From the neutral organic fraction was obtained 0.84 g. of an oil which crystallized partially on seeding with β -lactone. Filtering and washing with cold pentane gave 0.25 g. of β -lactone. The pentane washings decolorized 3.4 cc. of the standardized bromine-carbon tetrachloride solution. This represented 4.3 g. (54%) of 1,1-diphenylethylene.

In like manner, refluxing 1.0 g. of the β -lactone for one hour with 3.0 g. of sodium bromide in a mixture of 25 cc. of dioxane and 15 cc. of distilled water gave 0.43 g. of recovered β -lactone and 0.36 g. (45%) of 1,1-diphenylethylene. Similar treatment in the absence of the sodium bromide resulted in recovery of 96% of the β -lactone.

C. Sodium Chloride.—The reaction was carried out exactly as in B, only 1.72 g. of sodium chloride (Analytical Reagent) was substituted for the 3.0 g. of sodium bromide. Here again a part of the β -lactone crystallized from the reaction mixture and about half of it remained undissolved even after one hundred and twelve hours at 40°.

The mixture was worked up as described in A for the sodium iodide reaction and 0.80 g. of unreacted β -lactone was recovered. The non-crystallizable fraction decolorized only 0.4 cc. of the standard bromine solution correspond-

(10) Wagner-Jauregg, *Ber.*, **63**, 3223 (1930); *Ann.*, **491**, 1 (1931).

(11) Zaugg and Horrom, *THIS JOURNAL*, **72**, 3004 (1950).

(12) Lipp, *Ber.*, **56**, 567 (1923).

ing to only 0.05 g. (6%) of 1,1-diphenylethylene. Acidification of the aqueous fraction with dilute sulfuric acid precipitated none of the chloro acid.

Methyl 2,2-Diphenyl-3-bromopropionate.—Treatment of the corresponding carboxylic acid chloride¹¹ with excess dry methanol at room temperature for four hours gave, on fractionation *in vacuo*, a 94% yield of the methyl ester, b. p. 153–155° (0.9 mm.), n_D^{20} 1.5968. The distillate solidified and melted at 44–46°.

Anal. Calcd. for $C_{16}H_{15}BrO_2$: C, 60.20; H, 4.74. Found: C, 60.52; H, 4.66.

Treatment of Methyl 2,2-Diphenyl-3-bromopropionate with Sodium Methoxide.—A solution of sodium methoxide (from 0.4 g. of sodium) in 50 cc. of dry methanol was treated with 2.80 g. of the methyl ester and refluxed for twenty-five hours. The solution remained colorless and no sodium bromide precipitated. The methanol was removed by distillation and the residue was shaken with an ether–water mixture. The aqueous layer was separated and acidified. No acidic product was precipitated. The neutral ether layer was washed with water and dried over anhydrous magnesium sulfate. Filtration and distillation of the ether gave 2.23 g. of an almost colorless oil, n_D^{20} 1.5940 which on distillation gave 1.3 g., b. p. 136° (0.3 mm.). The distillate solidified, m. p. 44–46°. Mixed

with a sample of starting material it gave no depression of melting point.

Summary

1. The preparation of α, α -diphenyl- β -propiolactone is reported.

2. The formation of this β -lactone from 2,2-diphenyl-3-chloro, bromo and iodopropanoic acids has been studied together with formation of the by-product, 1,1-diphenylethylene. The re-formation of these β -halogen acids from the β -lactone on treatment with the corresponding hydrohalic acids is also described.

3. The reverse reactions of α, α -diphenyl- β -propiolactone with sodium chloride, bromide and iodide have been studied.

4. The reactivity of the halogen atoms in these β -halogen acids is contrasted with the lack of reactivity of the bromine atom in the methyl ester of 2,2-diphenyl-3-bromopropionic acid.

NORTH CHICAGO, ILLINOIS RECEIVED NOVEMBER 21, 1949

[CONTRIBUTION FROM THE DEPARTMENT OF ORGANIC CHEMISTRY, THE ABBOTT RESEARCH LABORATORIES]

α -Phenyltropic Acid and Basic Esters. Acid and Base-catalyzed Reactions of α, α -Diphenyl- β -propiolactone and of 2,2-Diphenyl-3-bromo- and Chloropropanoic Acids

BY HAROLD E. ZAUGG

During recent years a large number of basic esters of benzoic acid have been prepared by numerous workers^{1,2} and many of them have been shown to exert a high order of antispasmodic and/or local anesthetic activity. Basic esters of α -phenyltropic acid (II) obviously would be of considerable interest in this connection since this acid approaches more nearly than does benzoic acid the structure of tropic acid of which the naturally occurring, potent antispasmodic, atropine, is a basic ester. Indeed, Burtner and Cusic² have already reported a basic ester of α -phenyltropic acid. The latter was prepared by the action of nitrous acid on the amino ester $(C_6H_5)_2C(COOCH_3)CH_2NH_2$, followed by saponification. They reported a melting point of 167–168° for this hydroxy acid.

In the present work, α, α -diphenyl- β -propiolactone (I)³ was submitted to both acid and base hydrolysis with the expectation of arriving at the same acid II reported by Burtner and Cusic. However, this was not the case. An isomeric hydroxy acid melting at 157–158° was obtained. Since the method of preparation and the reactions of this acid, which are summarized in the accompanying flow-sheet, seemed to establish its

structure as II, the work of Burtner and Cusic was repeated. The melting point (164.5–165.5°) found for their acid checked reasonably well; however, by a mixed melting point determination with an authentic specimen,⁴ their acid proved to be identical with the isomeric α, β -diphenyllactic acid,⁵ $C_6H_5CH_2C(OH)(C_6H_5)COOH$, m. p. 164.5–165.5°, formed by rearrangement of a phenyl group.

In retrospect the formation of this isomeric hydroxy acid is not surprising since a number of closely allied rearrangements under similar conditions are known.⁶ On the other hand, it should be mentioned that tropic acid itself is formed by the action of nitrous acid on α -phenyl- β -alanine.⁷ Thus the unpredictable nature of the Demjanov reaction is once more exemplified.

The dimethylaminoethyl and diethylaminoethyl esters of α -phenyltropic acid (V, R = CH_3 and C_2H_5) were prepared. However, both the antispasmodic and local anesthetic activities of these compounds were disappointing, being much lower than those of the corresponding benzoic esters.

Best yields of α -phenyltropic acid (II) were obtained by acid hydrolysis of the β -lactone I

(1) Ford-Moore and Ing, *J. Chem. Soc.*, **55**, 952 (1947); King and Holmes, *ibid.*, 164 (1947); Holmes and Hill, U. S. Patent 2,399,736 and 2,430,116; Blicke, U. S. Patent 2,401,219; Northey and Hultquist, U. S. Patent 2,419,366; Chen, *et al.*, *J. Lab. Clin. Med.*, **30**, 700 (1945).

(2) Burtner and Cusic, *THIS JOURNAL*, **65**, 262 (1943).

(3) Zaugg, *ibid.*, **72**, 2998 (1950).

(4) The author is indebted to Dr. A. W. Weston of these Laboratories for a sample of α, β -diphenyllactic acid prepared by the general method of Rohrmann, Jones and Shonle, *ibid.*, **66**, 1856 (1944).

(5) "Beilstein's Handbuch der org. Chem.," Suppl. Vol. X, p. 155.

(6) Levy and Gallais, *Bull. soc. chim.*, **43**, 862 (1928); Hickinbottom, "Reactions of Org. Compounds," 2nd ed., 313 (1948).

(7) Natarajan and Swaminathan, *THIS JOURNAL*, **69**, 2560 (1947).