that of dichloral glucose (both dextrorotatory), m. p., 135° and 74°, respectively.

The same four compounds were formed in a similar manner from starch and glucose.

2. Acetylation and methylation of the two levorotatory compounds showed the presence of only one free hydroxyl group. No crystalline compounds were obtained by these reactions on the dextrorotatory compounds.

3. Parachloralose was formed when the crude water-insoluble products of starch and glucose, but not those of cellulose, were boiled. The soluble reaction products from all three, however, produced parachloralose when heated, after dilution, on the water-bath.

MONTREAL, CANADA

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF ILLINOIS]

MOLECULAR REARRANGEMENTS OF THE CAMPHOR SERIES. XIV. STRUCTURE OF ISOCAMPHOLACTONE[1]

BY PHILIP KELSEY PORTER WITH WILLIAM A. NOYES

RECEIVED MAY 14, 1923

Isocampholactone was first prepared by Noyes[2a] by the action of nitrous acid on aminolauronic acid. It was later prepared by Bredt[2b] in a pure condition by decomposing the nitroso derivative of the anhydride of aminolauronic acid with sodium hydroxide. Noyes and Homberger[3] oxidized isocampholactone with nitric acid and isolated two products, one the nitro-isocampholactone and the other a lactone acid which was the lactone of an hydroxy dicarboxylic acid and has the formula, $O = \underline{C - O - C}_7H_{11}COOH$. The amide of this lactone acid was prepared. Nitro-isocampholactone was decomposed by dil. alkaline reagents forming an acid which has the formula $C_5H_8NO_2COOH$. Three carbon atoms and four hydrogen atoms were lost by the reaction but the other product of the reaction was not isolated. The amide of this acid was also prepared.

The structure of isocampholactone had not been determined when the investigation here described was begun. We have shown that it has Formula I.

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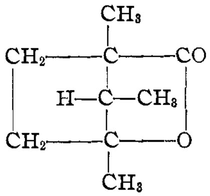
[1] An abstract of a thesis presented by Philip K. Porter in partial fulfilment of the requirements for the degree of Doctor of Philosophy at the University of Illinois, June, 1923.

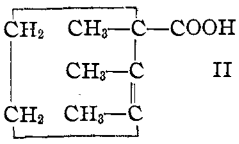
[2a] Noyes, *Am. Chem. J.*, **17,** 432 (1895).

[2b] Bredt., *Ber.*, **35,** 1291 (1902).

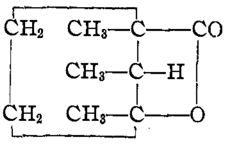
[3] Noyes and Homberger, THIS JOURNAL, **32,** 1665 (1910).



I



II



III

The lactone acid which was obtained by Noyes and Homberger[3] was converted to the disilver salt from which the dimethyl ester was prepared. On treatment of the hydroxy ester with Beckmann's[4] chromic acid mixture, which oxidizes primary and secondary hydroxy groups but not tertiary, the hydroxy group was shown to be tertiary and not secondary as it would be if no rearrangement had occurred. The lactone acid gives an amide which on treatment with sodium hypobromite is converted to a keto acid. The amide group and the hydroxy group are, therefore, on the same carbon atom. On treatment with sodium hypobromite, if the amino and hydroxy groups are on the same carbon atom, these split off ammonia, forming the keto group similar to the preparation of camphononic acid from camphanamide.[5]

This keto acid on treatment with methylmagnesium iodide regenerates isocampholactone and also forms another acid, which is probably the *trans*-hydroxy acid, as might be expected from the reaction. This compound was not fully identified however.

This series of reactions demonstrates that isocampholactone contains methyl groups in the 1,2,3 positions instead of the 1,2,2 positions of camphoric acid and should be called the lactone of 3-hydroxy-1,2,3-trimethyl cyclopentane-carboxylic acid.

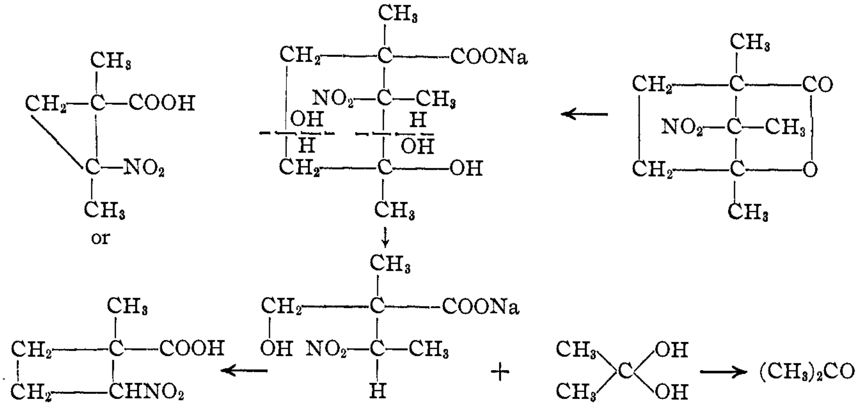
The isomerism of campholactone and isocampholactone may be explained when we consider the second asymmetric carbon atom. Lauronolic acid, from which campholactone is formed, has a double bond which would cause the two methyl groups to be in the same plane, as shown in Formula II. On breaking the double bond to form the hydroxy acid, campholactone is formed and would have Structure III. Isocampholactone then would have the second methyl group in the *trans* position with reference to the other two methyl groups. We consider this explanation as very probable but not as definitely proved. The work here described proved the structure of isocampholactone conclusively but can not be considered conclusive concerning its stereometric configuration.

Nitro-isocampholactone, which evidently has the nitro group in the 2 position, was decomposed by sodium hydroxide in such a manner as to isolate the volatile reaction products; 60% of the calculated amount

[4] Beckmann, *Ann.*, **250**, 325 (1889).

[5] Lapworth and Lenton, *J. Chem. Soc.*, **79**, 1293 (1901).

of acetone was obtained. The only possible explanation of this reaction is shown in the following formulas:



The acid is either a cyclobutane derivative with a secondary nitro group or a cyclopropane derivative with a tertiary nitro group. The nitrous acid test[6] applied to the amide, the acid or its ester gives a blue solution which is the test for a secondary nitro group. Secondary nitro groups would enolize and form a sodium salt when treated with sodium alcoholate.[7] The amide and the methyl ester of the acid formed sodium salts quite easily in quantitative yields. Hydrolysis of the sodium salt of an enolic nitro group with dil. sulfuric acid would form a ketone if the nitro group is secondary,[8] and a blue solution would be formed, while nitrous oxide would be given off. Hydrolysis of the sodium salt of the amide showed that no gas and no color was given to the solution. The original nitro acid was isolated from the reaction mixture.

Hydrolysis of the sodium salt of the methyl ester gave a deep blue solution and about $^1/_3$ of the calculated amount of nitrous oxide was obtained along with about the same amount of carbon dioxide. However, the original nitro acid was the only product isolated from the reaction. The keto acid which would form α-methyl-glutaric acid was not isolated, although the reaction seemed to have partially taken place. The nitrous acid test and the formation of the sodium salt of an enolic nitro group proves that this acid is a cyclobutane derivative and should be called 2-nitro-1-methyl-cyclobutane-carboxylic acid.

Experimental Part

Oxidation of Isocampholactone.—The isocampholactone required for these experiments was prepared by decomposing the nitroso derivative of the anhydride of aminolauronic acid with sodium hydroxide;[9] 65 g. of isocampholactone was heated for 72 hours

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[6] V. Meyer, *Ann.*, **175,** 93, 120 (1875).

[7] V. Meyer. *Ann.*, **256,** 28 (1889).

[8] Nef, *Ann.*, **280,** 273, 267 (1894).

[9] Noyes and Taveau, *Am. Chem. J.*, **32,** 288 (1904).

with nitric acid, d. 1.27,[10] and 40 g. of nitro-isocampholactone was obtained; m. p., 121–122°; yield, 47.6%.

The mother liquors from the oxidation were evaporated to dryness. The viscous residue was dissolved in benzene upon evaporation of which crystals appeared; 11.7 g. of the lactone acid or the lactone of 3-hydroxy-1,2-dimethyl-cyclopentane-dicarboxylic acid was obtained; m. p., 136–138°.

Analyses. Subs., 0.1621: CO_2, 0.3493; H_2O, 0.1010. Calc. for $C_9H_{12}O_4$: C, 58.67; H, 6.57. Found: C, 58.77; H, 6.98.

The Dimethyl Ester of 3-Hydroxy-1,2-dimethyl-1,3-cyclopentane-dicarboxylic Acid.—0.9666 g. of the lactone of 3-hydroxy-1,2-dimethyl-1,3-cyclopentane-dicarboxylic acid was dissolved in the calculated amount of sodium hydroxide by heating them together on the water-bath for 24 hours. The silver salt was obtained by adding an excess of silver nitrate; 1.9788 g. of salt was obtained or 90.5%.

Analysis. Subs., 0.2437: AgCl, 0.1683. Calc. for $C_9H_{12}O_5Ag_2$: Ag, 51.88. Found: 51.97.

The methyl ester was obtained by treating an ether solution of 1.1093 g. of silver salt with methyl iodide; 0.5343 g. of the ester was obtained; yield, 87.8%; b. p., 156–160° (25 mm.).

Analyses. Subs., 0.2053: CO_2, 0.4341; H_2O, 0.1439. Calc. for $C_{11}H_{18}O_5$: C, 57.39; H, 7.83. Found: C, 57.66; H, 7.86.

Beckmann's chromic acid mixture[4] oxidizes primary and secondary hydroxyl groups but not tertiary hydroxy groups. On treating the ester with Beckmann's reagent, only a slight darkening in color (a test for a tertiary hydroxy group) was produced even after the mixture had stood for 12 hours.

The 3-Amide of the Lactone of 3-Hydroxy-1,2-dimethyl-1,3-cyclopentane-dicarboxylic Acid.—1.1559 g. of the lactone of 3-hydroxy-1,2-dimethyl-1,3-cyclopentane-dicarboxylic acid was heated for 5 minutes in a 50cc. flask with 1.3 g. of phosphorus pentachloride.[11] The phosphorus oxychloride was distilled under diminished pressure and 5 cc. of dry ether was then added to dissolve the acid chloride. Cold concd. ammonium hydroxide (d., 0.9) was slowly added until the solution was strongly ammoniacal, after which the excess was evaporated under diminished pressure. The dry residue was dissolved in hot benzene, from which the amide crystallized. It was recrystallized from benzene; yield, 0.8638 g. or 75%; m. p., 162–163°.

3-Keto-1,2-dimethyl-cyclopentane-carboxylic Acid.—2.02 g. of the 3-amide of the lactone of 3-hydroxy-1,2-dimethyl-1,3-cyclopentane-dicarboxylic acid was dissolved in 14.3 cc. of a 10% solution of sodium hydroxide. After cooling the solution, the calculated amount of a cold sodium hypobromite solution was added. The reaction mixture was allowed to remain at room temperature for 12 hours and then heated on the water-bath for one hour. The cold solution was acidified with dil. sulfuric acid. Sodium sulfite was added to remove free bromine. The 3-keto-1,2-dimethyl-cyclopentane-carboxylic acid was extracted with ether and the extract dried over sodium sulfate. After distilling the ether, the acid was obtained as an oily residue from which 1.85 g. of the barium salt was obtained. The phenylhydrazone and the *p*-bromophenylhydrazone of the keto acid were prepared, both being oily materials. A crystalline hydrazone was not isolated.

Analyses. Subs. (Ba salt), 0.1194: loss at 120°, 7.20%. Calc. for $(C_8H_{11}O_3)_2$: Ba-$2H_2O$: 7.45.

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[10] Noyes and Homberger, THIS JOURNAL, 31, 280 (1909).

[11] Ref. 3, p. 1666.

Subs., 0.1108: $BaSO_4$, 0.0572. Calc. for $(C_8H_{11}O_3)_2Ba$: Ba, 30.69. Found: 30.37

Subs., 0.2159: $BaSO_4$, 0.1039. Calc. for $(C_8H_{11}O_3)_2Ba.2H_2O$: Ba, 28.41. Found: 28.31.

Action of Grignard's Reagent on 3-Keto-1,2-dimethyl-cyclopentane-carboxylic Acid. Preparation of Isocampholactone.—Methylmagnesium iodide was slowly added to an ether solution of 3-keto-1,2-dimethyl-cyclopentane-carboxylic acid in the proportion of two moles of the former to one mole of the latter; 3.4 g. of methyl iodide was dissolved in 4 cc. of dry ether and the solution was added to 0.5837 g. of magnesium ribbon covered with ether. The methylmagnesium iodide thus formed was slowly added to an ether solution of 1.8665 g. of the keto acid. A complex was formed immediately. The reaction mixture was slowly refluxed for 20 minutes, after which it was cooled and the complex was decomposed by the addition of 2.5 cc. of 1:1 sulfuric acid. The ether extract was washed with a solution of sodium acid sulfite to remove the free iodine and then washed with a 10% solution of sodium carbonate to remove the unchanged keto acid and the *trans*-hydroxy acid which would be formed by the reaction, after which it was dried over sodium sulfate. On evaporation of the ether, a viscous residue was obtained which was further purified by steam distillation. The distillate was again extracted with ether and a solid was obtained which melted at 31–32°. A mixed melting point with pure isocampholactone was 31–32°; $[\alpha]_D^{26}$ −60.1° (0.1406 g. in 5 cc. of absolute alcohol). Noyes and Homberger[12] gave $[\alpha]$ −63.1°.

To prove further its identity with isocampholactone, nitro-isocampholactone was prepared from this material by treatment with nitric acid, and was found to melt at 120–121°. A mixed melting point with pure nitro-isocampholactone was 120–121°; $[\alpha]_D^{26}$ −88.6° (0.0326 g. in 5 cc. of absolute alcohol). Noyes and Homberger[12] gave $[\alpha]$ −85.4. This reaction definitely proves the structure of isocampholactone; it should be called the lactone of 3-hydroxy-1,2,3-trimethyl-cyclopentane-carboxylic acid.

The sodium carbonate solution of the *trans*-hydroxy acid and unchanged keto acid was acidified and extracted with ether. An oily material was obtained. It was found that the calcium or barium salt of the hydroxy acid was difficultly soluble. This enabled a rough separation of the two acids to be made. A white solid was obtained which was acidic and melted at 96–97°; $[\alpha]_D^{28}$ —37.5° (0.1681 g. in 5 cc. of absolute alcohol).

Treatment of this hydroxy acid with fuming hydrobromic acid failed to give the corresponding bromo acid. The presence of the hydroxy group was not positively shown, yet from its method of formation it seems quite probable that the material melting at 96–97° was a *trans*-hydroxy acid.

The Amide of 2-Nitro-1-methyl-cyclobutane-carboxylic Acid.—0.3257 g. of nitro-isocampholactone was slowly decomposed by 5 cc. of concd. ammonium hydroxide,[12] the excess being evaporated under diminished pressure. The dry residue was dissolved in hot benzene from which the amide crystallized; yield, 0.215 g., or 83%; m. p., 94–95°.

Analyses. Subs., 0.1686: CO_2, 0.2806; H_2O, 0.1013. Calc. for $C_5H_8NO_2CONH_2$: C, 45.5; H, 6.3. Found: C, 45.4; H, 6.73.

Subs., 0.0987; 15.8 cc. of N_2 (25°, 744.5 mm.). Calc. for $C_5H_8NO_2CONH_2$: N, 17.72. Found: 17.41.

2-Nitro-1-methyl-cyclobutane-carboxylic Acid.—Nitro-isocampholactone was decomposed by warming it with about 2*N* sodium hydroxide solution.[12] When all of the nitro-isocampholactone had dissolved, the solution was acidified and the 2-nitro-1-methyl-cyclobutane-carboxylic acid was extracted with ether. It was converted to the barium salt and crystallized from water in long needles. The yields varied from 80 to 100%.

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[12] Ref. 3, p. 1668.

In the first attempts to determine the other reaction product, the reaction was carried on in a flask sealed to a drying tube which was in turn connected to a combustion tube, and the volatile reaction product burned and analyzed. About $^1/_3$ of the calculated amount of carbon and hydrogen which was lost by the reaction was accounted for by this arrangement.

Finally, 4.6931 g. of nitro-isocampholactone was decomposed by sodium hydroxide in a 100cc. flask which was connected to a 50cc. flask (cooled with ice and salt) by a tube that extended to the bottom of the flask. This flask was in turn connected to a 5cc. flask by a tube which extended to the bottom of the flask and was cooled by carbon dioxide snow and ether. The reaction mixture was slowly warmed on a water-bath until all of the nitro-isocampholactone had dissolved. Then it was slowly boiled until about 15 cc. of liquid had collected in the 50cc. flask. This material was boiled until about 3 cc. had collected in the 5cc. flask. The liquid material, which was acetone, was distilled; b. p., 56–58°; yield, 60%. It was identified as being acetone by the iodoform test and by the preparation of dibenzylidene acetone which melted at 111–112°.

The free acid was obtained from the barium salt by acidifying the solution of salt and extracting the acid with ether; m. p., 70–72°. The specific rotation was taken with a mercury-vapor lamp as the source of light at three different wave lengths, corresponding to the yellow line or 546.1$\mu\mu$, the green line or 578$\mu\mu$, and the blue line or 435$\mu\mu$, and also with a sodium vapor light; $[\alpha]^{25}_{578}$, —101.51°; $[\alpha]^{25}_{435}$, —246.81°; $[\alpha]^{25}_{546.1}$, —119.3°; $[\alpha]^{25}_{D}$, —87.31°.

Analyses. Subs., 0.1381: CO_2, 0.1485; H_2O, 0.0658. Calc. for $(C_5H_8NO_2COO)_2$-$Ba.2^1/_2H_2O$: C, 28.9; H, 4.2. Found: C, 29.3; H, 5.34.

Subs., 0.1657: $BaSO_4$, 0.0866. Calc. for $(C_5H_8NO_2COO)_2Ba$: Ba, 30.30. Found: 30.75.

Subs., 0.2168, 0.5331: $BaSO_4$, 0.1035, 0.2502. Calc. for $(C_5H_8NO_2COO)_2Ba$.-$2^1/_2H_2O$: Ba, 27.55. Found: 28.09, 27.62.

Subs., 0.1835: H_2O, 0.0178 (at 80° for 2 hours). Calc. for $(C_5H_8NO_2COO)_2Ba$.-$2^1/_2H_2O$: H_2O, 9.04. Found: 9.70.

Nitrous Acid Test for the Secondary Nitro Group.—According to V. Meyer,[6] nitrous acid reacts with primary nitro groups to form a red solution of a nitrolic acid and with secondary nitro groups to form a blue solution of a pseudonitrole, while it does not react with tertiary nitro groups. The 2-nitro-1-methyl-cyclobutane-carboxylic acid and its corresponding amide and methyl ester were treated with nitrous acid and the blue color obtained in each case. The results of this test support the cyclobutane formula.

Silver Salt of 2-Nitro-1-methyl-cyclobutane-carboxylic Acid.—5.261 g. of the barium salt was dissolved in a small amount of water and an excess of silver nitrate was added; 4.122 g. of the silver salt was obtained; yield, 73.2%. The salt is slightly soluble in water and darkens on exposure to the light.

Analyses. Subs., 0.1104, 0.1281: AgCl, 0.0596, 0.0692. Calc. for $C_5H_8NO_2$-$COOAg$: Ag, 40.56. Found: 40.62, 40.65.

Methyl Ester of 2-Nitro-1-methyl-cyclobutane-carboxylic Acid.—4.122 g. of the silver salt was suspended in ether and a solution of 10 cc. of methyl iodide dissolved in 10 cc. of ether was added. The mixture was refluxed on a water-bath for 6 hours. The silver iodide was filtered off and the ether evaporated in a vacuum; 2.4335 g. of the ester was obtained; yield 90.7%. The ester boiled at 120–125° at 5 mm. pressure with some decomposition. The specific rotation was taken with a mercury vapor lamp as a source of light at two different wave lengths, that corresponding to the yellow line or 578.1$\mu\mu$ and that corresponding to the green line or 546.1$\mu\mu$; $[\alpha]^{25}_{578.1}$.—80.2°; $[\alpha]^{25}_{546.1}$, —93.2°; d^{20}, 1.16907; *n*, 1.4416.

Treatment of the Amide of 2-Nitro-1-methyl-cyclobutane-carboxylic Acid with Sodium Alcoholate and the Hydrolysis of the Sodium Salt.—1.27 g. of the amide was dissolved in absolute alcohol and the calculated amount of sodium alcoholate was added. After this had stood at room temperature for two hours, two volumes of dry ether was added and the sodium salt was precipitated and filtered off. It was dried in a vacuum desiccator. The yield was quantitative.

Analyses. Subs., 0.1762, 0.1046, 0.1247, 0.1538: Na_2SO_4, 0.0630, 0.0393, 0.0446, 0.0564. Calc. for $C_6H_9N_2O_3Na$: Na, 12.77. Found: 11.58, 12.17, 11.66, 11.87.

Subs., 0.1071, 0.0897: NaCl, 0.0335, 0.0282. Calc. for $C_6H_9N_2O_3Na$: Na, 12.77. Found: 12.31, 12.35.

On hydrolysis of the sodium salt with dil. sulfuric acid, no gas was given off and the solution was only a faint blue. The solution was extracted with ether and a solid was obtained melting at 70–72°. A mixed melting point with pure 2-nitro-1-methyl-cyclobutane-carboxylic acid was 68–70°.

Treatment of the Amide with Two Molecular Equivalents of Sodium Hydroxide.—One g. of the amide was warmed with 5 cc. of a 10% solution of sodium hydroxide until *the ammonia had ceased to come off. The residue was attached to a gas buret and acidified.* A blue color was obtained which disappeared on warming. A gas was given off which caused a glowing splinter to burn, which is a qualitative test for nitrous oxide. About $^1/_3$ of the theoretical amount of gas was given off. The reaction mixture was extracted with ether and an oily acidic material obtained. The silver salt was prepared.

Analysis. Subs., 0.0723: AgCl, 0.0386. Calc. for $C_6H_8NO_4Ag$: Ag, 40.56. Found: 40.18.

Recovery of the acid from the analysis gave a product melting at 71–72°. A mixed melting point with pure 2-nitro-1-methyl-cyclobutane-carboxylic acid was 69–70°.

Treatment of the Methyl Ester of 2-Nitro-1-methyl-cyclobutane-carboxylic Acid with Sodium Alcoholate and the Hydrolysis of the Sodium Salt.—2.27 g. of the ester was dissolved in absolute alcohol and the calculated amount of sodium alcoholate was added. After this had stood for a few hours, two volumes of dry ether was added, which precipitated the sodium salt. This was filtered off and dried in a vacuum desiccator. It is very unstable and decomposes easily. The yield, however, was almost quantitative. The analysis was made on very impure material because of decomposition.

Analyses. Subs., 0.1143, 0.0853, 0.1072: Na_2SO_4, 0.0660, 0.0513, 0.0683. Calc. for $C_7H_{10}NO_4Na$: Na, 12.57. Found: 18.70, 19.48, 20.64.

Hydrolysis of the sodium salt with dil. sulfuric acid gave a blue solution, and about $^1/_3$ of the calculated amount of nitrous oxide was obtained that was identified by the test with a glowing splinter. About the same amount of carbon dioxide was also obtained. The reaction mixture was extracted with ether and the oily residue which was obtained was treated with barium carbonate, which separated the unchanged ester from the acids; these may be a mixture of the original nitro acid and the keto acid, which would break down into α-methyl-glutaric acid. The barium salts were fractionally crystallized. Since the barium salt of the nitro acid is difficultly soluble, the most soluble portion was converted into the silver salt.

Analysis. Subs., 0.1300: AgCl, 0.0921. Calc. for $C_6H_8O_4NAg$: Ag, 40.56; calc. for $C_6H_7O_3Ag$: 45.92; calc. for $C_6H_8O_4Ag_2$: 59.96. Found: 53.318.

The mixed acids obtained from a second experiment were warmed with acetyl chloride to form the anhydride of the α-methyl-glutaric acid and distilled. The low-boiling fraction, boiling at 130–140° (5 mm.), was taken. It was then boiled with water to regenerate the acid, and the silver salt was prepared.

Analysis. Subs., 0.0614: AgCl, 0.0428. Found: Ag, 52.46.

The analyses and the behavior of the reaction indicates that α-methyl-glutaric acid might have been formed, but in quantities too small to be isolated.

Summary

1. When isocampholactone is heated with nitric acid, the two main products formed are nitro-isocampholactone and a lactone acid. This lactone acid gives an amide which on treatment with sodium hypobromite is converted to a keto acid. This, on treatment with methylmagnesium iodide, regenerates isocampholactone and also probably forms a *trans*-hydroxy acid. This series of reactions demonstrates that isocampholactone contains methyl groups in the 1,2,3 positions and should be called the lactone of 3-hydroxy-1,2,3-trimethyl-cyclopentane-carboxylic acid.

2. A possible explanation of the isomerism of campholactone and isocampholactone is given.

3. Nitro-isocampholactone on treatment with alkali gives almost quantitatively acetone and a nitro acid of 6 carbon atoms. The nitro group in this acid is secondary, as proved by the nitrous acid test and by the formation of a sodium salt of the enolic form of the nitro group in both the acid and its ester. This test proves that it is a cyclobutane derivative and should be called 2-nitro-1-methyl-cyclobutane-carboxylic acid.

URBANA, ILLINOIS

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF ILLINOIS]

SIMPLIFICATION OF THE GATTERMANN SYNTHESIS OF HYDROXY ALDEHYDES

BY ROGER ADAMS AND I. LEVINE[1]

RECEIVED MAY 21, 1923

The Gattermann synthesis of hydroxy aldehydes[2] which consists in saturating an anhydrous ether solution of certain phenols and anhydrous hydrogen cyanide with dry hydrogen chloride, sometimes with the addition of anhydrous zinc chloride, gives excellent yields of products which are readily purified. The method has proved to be invaluable for the preparation of certain intermediates in the synthesis of many natural compounds, and is still the only available process for preparing many representative hydroxy aldehydes. In spite of the ease with which the reaction generally takes place, its use in the laboratory is not as frequent as might be expected. This is due to the necessity of using anhydrous hydrogen cyanide, a product the preparation and handling of which in-

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[1] This communication is an abstract of a thesis submitted by I. Levine in partial fulfilment of the requirements for the degree of Master of Science in Chemistry at the University of Illinois.

[2] Gattermann, *Ber.*, **31**, 1765 (1898); **32**, 278, 284 (1899); *Ann.*, **357**, 313 (1907). Morgan and Vining, *J. Chem. Soc.*, **119**, 177 (1921).