yellow. The aqueous solution, boiled with sodium hydroxide, splits off chlorine ion.

0.1080 g. subst.; 16.0 cc. N, 770 mm., 20.5°.
Calc. for $C_{10}H_{12}O_2N_3Cl$: N, 17.39%. Found: N, 17.46%.

NEW YORK CITY.

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[CONTRIBUTION FROM THE LABORATORIES OF THE ROCKEFELLER INSTITUTE FOR MEDICAL RESEARCH.]

# THE PREPARATION OF β-CHLORO- AND β-BROMOPROPIONIC ACIDS.

BY WALTER A. JACOBS AND MICHAEL HEIDELBERGER.

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Of the chief methods to be found in the literature for the preparation of β-chloro- and β-bromopropionic acids only that involving the oxidation by nitric acid of β-chloro- and β-bromopropionaldehydes,[1] obtained by saturation of acrolein with halogen acids, seemed to offer promise for the preparation of considerable quantities of material. Although the yields claimed for this method are excellent, the necessity of working with unpleasant substances led us to seek for a more convenient procedure.

Beckurts and Otto[2] prepared the two acids in question by treating hydracrylic acid with hydrochloric and hydrobromic acids. Hydracrylic acid, in its turn, was obtained by Moureau[3] by hydrolysis of ethylene cyanohydrin, which he prepared easily and satisfactorily by the interaction of ethylene chlorohydrin and potassium cyanide. It occurred to the writers that, starting with ethylene cyanohydrin, the two steps of saponification of the nitrile to the acid and replacement of the hydroxyl group by halogen could be accomplished simultaneously by heating the cyanohydrin with concentrated halogen acid. These expectations were confirmed. Since all of the steps involved may be rapidly and conveniently executed the authors feel that this procedure will prove to be the most satisfactory method for those who have occasion to prepare these substances.

## Experimental.

**Ethylene Cyanohydrin,** $HOC_2H_4CN$.—The only variation from Moureau's method was that the product was purified by distillation *in vacuo*. 32 g. ethylene chlorohydrin were dissolved in 160 cc. absolute alcohol and boiled under a reflux condenser. To the boiling solution were added, drop by drop, 27.2 g. potassium cyanide dissolved in 42 cc. water. The boiling was continued for eight to ten hours. At the end the

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[1] Krestovnikov, *J. Russ. Phys. Chem. Soc.*, **11**, 248 (1879); Lederer, *J. prakt. Chem.*, [2] **42**, 384 (1890).

[2] Beckurts and Otto, *Ber.*, **18**, 226 (1885).

[3] Moureau, *Bull. soc. chim.*, [3] **9**, 426 (1893).

solution was filtered from potassium chloride and the filtrate concentrated to a syrup, which was fractionated *in vacuo*. The cyanohydrin boiled at 110° at 15 mm. The yield was 20 g.

**β-Chloropropionic Acid.**—10 g. ethylene cyanohydrin were heated in a sealed tube with 75 cc. concentrated hydrochloric acid at 100° for three hours. Ammonium chloride separated from the solution on cooling. The contents of the tube were diluted with water and extracted with ether. The ethereal extract was dried over sodium sulfate and concentrated, yielding a syrup which easily crystallized. The yield of β-chloropropionic acid so obtained was 10.5 g. Recrystallized from hot ligroin, the acid melted at 38.5–39.5° (corr.) with slight preliminary softening. It possessed the characteristic odor and other properties of β-chloropropionic acid.

0.1964 g. subst.; 0.2560 g. AgCl.
Calc. for $C_3H_5O_2Cl$: Cl, 32.68%. Found: Cl, 32.25%.

**β-Bromopropionic Acid.**—10 g. ethylene cyanohydrin were boiled with 100 cc. hydrobromic acid (d. 1.49) for three hours. The mixture was cooled, diluted with water, and extracted with ether. The dried ethereal extract yielded on concentration 17 g. β-bromopropionic acid. As so obtained the substance is practically pure. Recrystallized from hot ligroin it melted at 60–61° (corr.) and possessed the recorded properties of β-bromopropionic acid.

0.1554 g. subst.; 0.1910 g. AgBr.
Calc. for $C_3H_5O_2Br$: Br, 52.24%. Found: Br, 52.30%.

NEW YORK CITY.

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[CONTRIBUTION FROM THE BUREAU OF CHEMISTRY, U. S. DEPARTMENT OF AGRICULTURE.]

# GINGEROL AND PARADOL.[1]

BY E. K. NELSON.

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The pungent principles of ginger and of grains of paradise (*amomum melegueta*) were first separated and studied by Thresh.[2]

Thresh describes both principles as pungent oils which possess the properties of phenols.

To the pungent principle of ginger he assigned the name "Gingerol" and to the similar principle in grains of paradise the name "Paradol." Neither substance was obtained in a state of purity, nor were any crystalline derivatives prepared from them.

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[1] While engaged in this research it came to the attention of the writer that Dr. Lapworth, of Manchester, has made a study of gingerol, has isolated its pungent constituent, established its constitution and synthesized it. (*Pharm. J.*, [4] **44**, 201.) Accordingly the work here recorded was discontinued and the results published in their present form.

[2] *Pharm. J.*, [3] 10, 171 (1879); [3] **12**, 721 (1881); [3] **14**, 798 (1883); [3] **15**, 208, (1884).