If this empirical formula is correct, the substance is probably triiodobromoacetone, and we have adopted this structure for it provisionally.

Properties of Triiodobromoacetone?—It crystallizes from tetrachloride of carbon in slender, lemon-yellow needles, which turn brown in the light. It melts with decomposition at 122–125°, but this point is decidedly indefinite. It is soluble in alcohol, methyl alcohol, benzene, chloroform, or tetrachloride of carbon; essentially insoluble in petroleum ether. All of these solvents decompose it somewhat with liberation of iodine, but this action is least with tetrachloride of carbon, which is therefore the best solvent for it. Fuming nitric in the cold or strong nitric acid when heated decomposes it, setting free iodine. Nitric acid diluted with acetic acid had no action in the cold, but on warming iodine was set free. Ammonium hydroxide gave a smell like iodoform, and ammonium iodide was found in the liquid. Sodium hydroxide also gave iodoform to judge by the smell. Hydric dioxide did not act on it.

Hexabromodiacetylmonoethylhemiacetal was not affected by potassium iodide solution even at 100°.

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[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF HARVARD UNIVERSITY.]

NORMAL NONANE.

BY LATHAM CLARKE AND ROGER ADAMS.

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Normal nonane was first synthetically produced by Krafft,[1] by reduction of pelargonic acid, $C_9H_{18}O_2$, with hydriodic acid and phosphorus in a sealed tube heated to 240°. The boiling point at 760 mm. is given by Krafft as 149.5° and the specific gravity, compared to water at 4°, is as follows: 0°, 0.7330; 13.5°, 0.7228; 15°, 0.7217; 20°, 0.7177; 99.1°, 0.6541.

We found the boiling point as 150.4–150.6° at 759 mm. The specific gravity at 15°, compared to water at 15°, is 0.7219. The index of refraction, $n_D^{25°} = 1.4025$.

The starting point in the preparation of normal nonane was castor oil. By distillation this yielded oenanthol (normal heptyl aldehyde), which, after purification by means of the bisulfite compound, was treated with ethyl magnesium bromide giving, after the usual decomposition of the organo-magnesium derivative, ethyl hexyl carbinol, or 3-nonanol. This carbinol was converted into the corresponding carbinol iodide by treatment with iodine and red phosphorus, and the resulting 3-iodononane was boiled with alcoholic potash, which gave nonylene. This last yielded normal nonane on reduction.

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[1] *Ber.*, 15, 1692 (1882).

Summary.

Castor oil ⟶

$CH_3CH_2CH_2CH_2CH_2CH_2CHO$, oenanthol ⟶

$CH_3CH_2CH_2CH_2CH_2CH_2CH(OH)CH_2CH_3$, 3-nonanol ⟶

$CH_3CH_2CH_2CH_2CH_2CH_2CH(J)CH_2CH_3$, 3-iodononane ⟶

$CH_3CH_2CH_2CH_2CH_2CH_2CH = CH - CH_3$ ⟶

$CH_3CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_3$, normal nonane.

Experimental.

Oenanthol.—The process used for obtaining oenanthol was a slight modification of the one used by Erlenmeyer.[1] Five hundred grams of castor oil were poured into a three-liter flask, which was connected to a condenser containing water in the jacket, but not running water. The flask was placed on a sand bath and strongly heated. A rather rapid distillation of liquid ensued, and the heating was continued until the flow of distillate had considerably slackened, then the burners were removed. The residue in the flask was poured out while still hot. If the heating was continued too long, the residue began to foam badly and became a sort of spongy material, exceedingly hard to remove from the flask.

The distillate was redistilled and a fraction collected between 90° and 180°. This was shaken with a concentrated solution of pure sodium bisulfite and the aldehyde-bisulfite compound crystallized out. This was filtered and pressed out on a clay plate. The best method for the resolution of this into pure normal heptyl aldehyde was to treat with an excess of saturated sodium carbonate solution and distil carefully with steam.

The aldehyde was separated from the water in the distillate, dried over calcium chloride,[2] and redistilled. The fraction boiling at 152–157° was that used in subsequent work.

Ethyl-hexyl Carbinol (3-Nonanol).—A solution of ethyl magnesium bromide was prepared by dissolving 12 g. of magnesium in a mixture of 60 g. of ethyl bromide and twice its volume of absolute ether. To this, 57 g. of oenanthol in its own volume of absolute ether was gradually added, and the whole allowed to stand several hours at room temperature. The organo-magnesium compound was decomposed in the usual way with water and dilute sulfuric acid. The ether layer containing the carbinol was separated, and dried over potassium carbonate. The ether was then distilled off and the residue fractionated. 45 g. of carbinol were thus obtained, boiling at 192–193° at 755 mm.

This alcohol has been previously made by Wagner from oenanthol and zinc ethyl,[3] and by Gerard from oenanthol and ethyl magnesium bromide.[4]

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[1] *Ann.*, **176**, 342 (1875).

[2] Calcium chloride did not have any detectable chemical action on this aldehyde, and was found to be a very efficient drying agent for it.

[3] *J. Russ. Chem. Soc.*, **16**, 306.

[4] *Chem. Zentr.*, **1907**, I, 1398.

3-Iodononane.—This was obtained from 3-nonanol by the usual procedure for the conversion of alcohols into iodides. In a typical experiment 40 g. of 3-nonanol were mixed with 4 g. of red phosphorus and 36 g. of iodide. The mixture, which at first was artificially cooled, was later heated for two hours on the steam bath, then filtered to remove unchanged red phosphorus, washed with water to remove acids of phosphorus, and without further purification was converted into nonylene.

Normal Nonylene.—The above 3-iodononane was refluxed with excess of strong alcoholic potash for about an hour. The liquid in the reaction flask was distilled, and yielded a solution of nonylene and alcohol, these forming a mixture of minimum boiling point. On the addition of much water, the hydrocarbon separated as a floating layer, which was removed, washed with water, dried with calcium chloride and distilled from metallic sodium. On subsequent fractionation, the main portion boiled at 149.4–149.9°.

Properties.—Colorless liquid, very mobile, pleasant petroleum-like odor. It boils at 149.4–149.9° and has at 15° the specific gravity 0.7540 compared to water at 15°.

Calculated for C_9H_{18}: C, 85.7; H, 14.3. Found: C, 85.3; H, 14.2.

Normal Nonane.—Normal nonylene was reduced to normal nonane by passing the vapor of the former, together with hydrogen, over freshly reduced nickel at a temperature of 160°.[1]

Properties.—Colorless, very mobile liquid, with a very slight "sweetish" odor. B. p. 150.4–150.6° at 759 mm. The specific gravity at 15° compared to water at 15° is 0.7219. The index of refraction $n_D^{25°}$ = 1.4025. It is miscible with the common organic solvents.

Calculated for C_9H_{20}: C, 84.4; H, 15.6. Found: C, 84.1; H, 15.7.

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[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF MICHIGAN.]

THE CONSTITUTION OF THE NITRO-α-CARBOPYRROLIC ACIDS.[2]

BY WILLIAM J. HALE AND WILLIAM V. HOYT.

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In 1882 Weidel and Ciamician[3] prepared the first nitro derivative of the pyrrole ring in the form of a nitrocarbopyrrolic acid. The dry distillation of gelatin yielded pyrocoll, and this product, upon careful nitra-

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[1] The exact procedure may be found in an article by Clarke and Beggs, THIS JOURNAL, **34**, 59 (1912).

[2] The work described in this article forms part of a thesis submitted in partial fulfilment of the requirements for the degree of Doctor of Philosophy in the University of Michigan by William V. Hoyt.

[3] *Gazz. chim. ital.*, 12, 39 (1882).