proportion of the ingredients will vary with the purity of the lime, that of ordinary so-called good lime it may require 1.25 kilos to 1 k of sodium hydroxide and sometimes a little less water than stated before. The best proportion of the ingredients at hand to use may, therefore, at times have to be determined experimentally.

The pieces of soda lime should be porous, light gray in color and hard enough to break and crumble when pounded. They are broken up and sifted and the granules of about 1/16 inch or a little more in diameter are used in the U-tubes for carbon determinations.

Hydrogen Determination.—By means of the bomb calorimeter it is possible also to determine hydrogen, but it is a much more difficult and tedious operation, and it is very hard on the bomb, and with the kind of bombs in use at present, the writer considers it too costly and hence impracticable. Trials were made and it was found that the escapin acids attack the apparatus too much; but as soon as the bomb can be made with platinum or other non-corroding lining all through the gas outlet, a method for hydrogen can doubtless be worked out successfully.

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

MOLECULAR REARRANGEMENTS IN THE CAMPHOR SERIES I. HYDROXYLAURONIC ACID AND ISOCAMPHOLACTONE.

BY W. A. NOYES AND A. W. HOMBERGER.

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Terpenes, camphor and their derivatives often undergo molecular rearrangements which render conclusions drawn with regard to their structure fallacious. Very little is known with regard to the laws governing such rearrangements and it seems possible that a careful study of some of these changes may throw additional light upon the nature of the forces which exist in molecules containing carbon. The first step in such a study must consist in establishing the structure of compounds which result from such rearrangements. From the nature of the case this is often extremely difficult as we have very little to guide us in deciding whether a rearrangement has taken place in a given reaction or not. In the end, one is dependent on that sort of cumulative evidence which is so characteristic of many of the most difficult problems of organic chemistry and which is so trying to minds which desire an absolute proof for everything that is to be accepted as scientific.

The work described in this paper has, of course, made very little progress toward the study of the larger question and is quite incomplete. As it must be discontinued for some time, however, it seems desirable to record, briefly, the results obtained.

α -Camphoramidic acid, $C_8H_{14}(CO_2H)CONH_2$, gives with sodium hypo-

bromite, aminolauronic acid, $C_8H_{14}(CO_2H)NH_2$, in which the carboxyl is tertiary and the amino group secondary. When aminolauronic acid is treated with nitrous acid it gives, chiefly, γ -lauronic acid, $C_8H_{13}CO_2H$,

laurolene, C_8H_{14} , and isocampholactone, C_8H_{14} . The nitroso deriva-

tive of the anhydride of aminolauronic acid, C_8H_{14} , gives the same

products in different proportions and some other compounds, which need not be considered here, when it is boiled with a solution of sodium hydroxide.² When the ethyl ester of aminolauronic acid, $C_8H_{14}(NH_2)CO_2C_2H_5$, is treated with nitrous acid it gives a mixture of the esters of γ -lauronic acid and of hydroxylauronic acid,³ $C_8H_{14}(OH)CO_2C_2H_5$.

The ethyl ester of aminolauronic acid was prepared by heating 20 grams of aminolauronic acid hydrochloride, $C_8H_{14}(NH_2HCl)CO_2H$, 6 cc. of concentrated sulphuric acid and 80 cc. of absolute alcohol on a water bath in a stout bottle connected with an upright condenser, the top of which was connected with a glass tube dipping under a column of 50 cm. of mercury. After heating for 48 hours the alcohol was distilled under diminished pressure, water and a slight excess of sodium hydroxide were added and the ester taken up with ether. The yield was 35 per cent. Yields of 50 to 65 per cent. were obtained with pressure bottles but the bottles often exploded owing to the pressures developed from the ethyl chloride formed.

The ester of aminolauronic acid was treated with a little less than the calculated amount of sulphuric acid (25 per cent.), the solution cooled and the calculated amount of sodium nitrite (1:5) added carefully, with cooling. After several hours the layer of esters formed was taken up with ether, washed with a dilute solution of sodium carbonate and with water, dried with anhydrous sodium sulphate, and distilled under diminished pressure. The weight of mixed esters obtained was only one-half that of the aminolauronic ester.

At a pressure of 25 mm. the γ -lauronic ester, $C_8H_{13}CO_2C_2H_5$, comes over at $110-115^\circ$. The specific gravity, $d_4^{20} = 0.9514$. The specific rotation, $[\alpha]_D^{26} = +56.6^\circ$.

The hydroxylauronic ester, $C_8H_{14}(OH)CO_2C_2H_5$, boils at 150° at 30 mm. The specific gravity, $d_4^{20} = 1.100$. The specific rotation $[\alpha]_D^{33.5} = +6.73$.

Analysis:

¹ Noyes, *Am. Chem. J.*, **17**, 432.

² Noyes and Taveau, *Ibid.*, **32**, 285; **35**, 379. Bredt, who first studied this reaction, mentions only isocampholactone as resulting from this decomposition. *Ber.*, **35**, 1291.

³ Noyes, *Am. Chem. J.*, **18**, 687.

acid, $C_8H_{11}(OH)CO_2H$, must be a stereoisomer of hydroxylauronic acid. In order to obtain further light on this question isocampholactone has been subjected to oxidation. Three grams of the lactone were heated for 72 hours with nitric acid (sp. gr. 1.27) in a 50 cc. flask sealed to an upright condenser. The heating was continued till the oily layer of lactone disappeared, a little concentrated acid being added from time to time. The study of the products of the oxidation is still quite incomplete. The products thus far obtained are a lactone, $C_8H_{12}O_4$, an acid melting at 228° , probably camphononic acid, and a compound, probably a lactone, which melts at 111° .

The lactone, $C_8H_{12}O_4$, is the chief product of the oxidation. It partly sublimes into the condenser during the oxidation and the remainder separates in crystalline form on cooling the solution. It is quite volatile and crystallizes from ligroin in needles. It is moderately soluble in ligroin, more easily soluble in ether and benzene. It melts at 122° and boils at 272° . A part of the analyses were not very satisfactory, possibly because of a partial hydrolysis of the lactone group.

Calculated for $C_8H_{12}O_4$:	C, 55.79;	H, 7.03.
Found:	C, 56.09, 56.38, 55.89, 54.85;	H, 7.19, 7.01, 7.25.
	54.81, 54.9, 54.9	7.12, 7.24, 7.12, 6.95.

The analyses were made with four different preparations.

Victor Meyer determinations gave a molecular weight of 164, 176.6, 180, 175, 166.2; average, 172; calculated, 172.1.

The molecular weight found by the lowering of the freezing point of glacial acetic acid was 175.3.

When the lactone is warmed with a solution of barium hydroxide or of sodium hydroxide it dissolves and somewhat more than one equivalent of the alkali is neutralized but the results were not concordant or satisfactory. As the presence of two lactone groups is impossible in a compound of this formula, it seems probable that the compound is decomposed by the alkali.

The formation of camphononic acid in the oxidation, if confirmed, will, of course, point to the formula given above for isocampholactone. We do not, however, feel justified in drawing any conclusions with regard to its structure from the work done thus far.

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[CONTRIBUTION FROM THE CHEMICAL LABORATORY, OF THE OHIO STATE UNIVERSITY.]
THE ACTION OF UNSYMMETRICAL BENZOYLPHENYLHYDRAZINE ON ORTHOBENZOQUINONE.

BY WILLIAM MCPHERSON AND H. J. LUCAS.

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The action of the unsymmetrical acyl phenylhydrazines on parabenzquinone has been shown¹ to be as follows:

¹ Ber., 28, 2414. Am. Chem. J., 22, 364. THIS JOURNAL, 22, 141; 30, 816.