Camphoric Acid: Reduction of the Neighboring Xylic Acid.

By W. A. Noyes.

[Abstract.]

An account of the preparation of the neighboring xylic acid has been recently given by the author in the American Chemical Journal. The acid has now been reduced to the corresponding hexahydroxylic acid. The latter boils at 250°—252°, while dihydrociscampholytic acid boils at 244°. The *a*-brom derivative has also been prepared and treated with alcoholic potash. The resulting acid is not ciscampholytic acid. This proves that Collie's formula for camphor cannot be true.

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a-Hydroxy-dihydro-ciscampholytic Acid. By W. A. Noyes and J. W. Shepherd.

[Abstract.]

After many ineffectual attempts to prepare the acid by usual methods, it was finally obtained by shaking the ethyl ester of *a*-brom-dihydro-ciscampholytic acid for a long time, at a temperature of 40°—50°, with a strong aqueous solution of barium hydroxide. When the hydroxy acid is warmed with phosphoric acid and lead peroxide (a reaction for *a*-hydroxy acids, recently developed by Baeyer), it gives a ketone which is probably identical with that prepared by Mr. E. B. Harris, under the direction of one of us some years ago. We hope to secure the ketone in larger quantities and that a study of its derivatives will throw new light on the structure of camphor.

Iodine Absorption of Linseed Oil.* By P. N. Evans and J. O. Meyer.

The following statement concerning the necessary excess of iodine and the duration of the reaction, in determining the iodine absorption of oils, occurs in the 1897 edition (German) of Benedikt's "Analysis of Fats and Waxes" (page 152):

*This paper is an abstract of a thesis presented by Mr. J. O. Meyer, for the degree of B. Sc., and placed in the library of Purdue University.

"The [iodine] numbers are quite constant if the iodine solution is present in sufficient excess; the excess, according to Ulzer, when the reaction continues for six hours, must be at least 50 per cent. of the iodine used. Fahrion uses an excess of 100 per cent. of the absorbed iodine (i. e., also 50 per cent. of the iodine used) with a reaction of only two hours, and Holde recommends for this period of reaction the use of 75 per cent. excess of iodine. * * * With a sufficient excess of iodine the results obtained are the same after two hours, six hours, and longer periods."

This refers to the use of the ordinary Hübl's solution of iodine and mercuric chloride, and in the experiments to be described the determinations were carried out in the usual way, varying the two factors—(1) excess of iodine, and (2) duration of reaction.

Excess of Iodine.—It is stated in the passage above quoted that the iodine in excess must be at least 75 to 100 per cent. of the iodine absorbed, and that a larger excess will not affect the results if the reaction is not less than two to six hours in duration.

To test this statement the writers made twenty determinations with a linseed oil, the mixture being allowed to stand six hours, and the excess of iodine ranging from .008 to 3.658 per unit of iodine absorbed. The experiments were made in three series, each series being carried out under as nearly as possible identical conditions, and in spite of two slight discrepancies in the twenty experiments, there was unmistakable evidence that the iodine number steadily and materially increased with the excess of iodine, the increase being nearly as marked between an excess of 1 and of 3.6 as below 1. The iodine numbers obtained varied from 131.2 to 175.3, those with an excess of over 1 from 161.9 to 175.3.

Benedikt gives as minimum and maximum results for commercial linseed oil, according to twenty authorities, 148 and 181, with an average of 170.

Duration of Reaction.—In the above citation from Benedikt, a duration of two hours is said to be sufficient if the excess of iodine is equal to that absorbed (Fahrion), or 0.75 times as great (Holde), while six hours is said to be enough for an excess of 1 according to Ulzer.

The writers carried out eighty experiments to test this point, the excess of iodine varying from .5 to 1.4, and the duration of the reaction from two to eight hours. The eighty experiments were made in six series, and only three of the eighty experiments failed to confirm the conclusion that

six hours is not long enough to yield satisfactory results. The iodine numbers obtained ranged from 131 to 184.3.

These results seem to show that the figures given by Benedikt for excess of iodine and duration of reaction are too low, and point to those as preferable which are elsewhere recommended by Schweitzer and Lungwitz, Holde and Dieterich, summed up by A. H. Gill in his "Short-Handbook of Oil Analysis," as follows: "The excess of iodine recommended is from 150 to 250 per cent.; some observers recommend from 400 to 600 per cent. * * * Two hours is sufficient for olive oil, tallow and lard, while for linseed oil, balsams and resins, twenty-four hours should be allowed.

TABLE I.

Showing Effect of Excess of Iodine.

Series No.	Number of Nearly Identical Experiments.	Average Excess of Iodine per 1 Absorbed.	Duration of Reaction.	Average Iodine Number Found.
1	3	.009	6 hours	131.2
	2	.341	6 hours	147.9
	3	.737	6 hours	151.6
2	3	2.888	6 hours	161.9
	3	3.648	6 hours	162.4
3	2	1.503	6 hours	172.3
	2	1.954	6 hours	175.3
	2	3.075	6 hours	173.3

TABLE II.

Showing Effect of Duration of Reaction.

1	3	.589	2 hours	165.0
	3	.566	4 hours	168.0
	3	.540	6 hours	170.7
	3	.526	8 hours	172.3
2	3	.740	2 hours	155.4
	3	.624	3 hours	156.4
	3	.638	4 hours	158.3
	2	.444	6 hours	159.8
	3	.498	8 hours	160.8

TABLE II—CONTINUED.

3	3	.637	2 hours	162.4
	3	.601	4 hours	166.4
	3	.584	6 hours	167.9
	3	.557	8 hours	170.9
4	3	.655	2 hours	175.3
	3	.618	4 hours	179.1
	3	.610	6 hours	182.9
	3	.589	8 hours	184.3
5	3	.951	2 hours	131.9
	3	.940	3 hours	133.4
	3	.951	4 hours	131.0
	3	.896	6 hours	134.4
	3	.889	8 hours	134.6
6	3	1.535	2 hours	165.8
	3	1.495	3 hours	168.2
	3	1.493	4 hours	168.8
	3	1.453	6 hours	170.8
	3	1.396	8 hours	171.5

Some Desmids of Crawfordsville. By Mason B. Thomas.

In looking over the bibliography of Indiana cryptogams we have been greatly surprised at the very meager representation of our Algæ. This we believe to be in some measure due to the lack of correlation of work already done in different parts of the State and upon which no report has been made.

The past spring one of our students, Mr. F. Corey, in working on some Algæ made a list of Desmids that we believe worth while to record. Nothing need be said about the list except that the determinations were carefully made and mounted specimens preserved of each form, together with notes on distribution, etc. It is the intention to prepare as complete a list as possible of Crawfordsville Algæ with a view to some studies on distribution. Permit us to suggest that