Dr. W. J. Robbins,[7] of the University of Missouri, states that when thin sections of fruit tissue are mounted in alcohol and left for hours, the hesperidin appears to be highly concentrated in the immediate vicinity of the fibrovascular bundles, and to be almost entirely absent from the more remote portions. This can hardly be due to the slow infiltration of the alcohol because of the time of immersion, but indicates that the glucoside is really concentrated around the bundles.

These observations lead to the interesting idea that hesperidin and perhaps other phenolic glucosides, may serve as a medium for translocation of the glucose synthesized in the chlorophyllous tissue. The glucose is in combination with the glucoside, forming a soluble, easily hydrolyzable compound, and is thus temporarily withdrawn from metabolism until brought to that portion of the plant where it is to be stored or utilized.

Conclusions.—1. Indications have been found of the existence of a soluble compound of glucose and hesperidin in the endocarp of mature navel oranges. This compound is probably accompanied by another similar substance.

2. Another substance, related to hesperidin but probably not in combination with glucose, also exists in the endocarp.

3. An hypothesis is suggested to explain the possible function in plants of hesperidin and phenolic glucosides with free hydroxyl groups.

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Preparation of Diacetonamine.—There is only one method given in the literature for the preparation of diacetonamine which gives satisfactory yields. This method is given by Everest.[1] The other methods are not numerous and are very unsatisfactory, being laborious and giving low yields. There is one great advantage in using mesityl oxide instead of acetone for the preparation of diacetonamine, namely, the elimination of the possibility of the formation of such compounds as triacetonamine, triacetondiamine and resinous products.

Since it is relatively easy to obtain pure mesityl oxide in large quantities using the method of Roger Adams,[2] the preparation of diacetonamine from mesityl oxide and ammonia was tried. Only the results of experiments with aqueous ammonia are given since the yields from anhydrous ammonia were very poor.

Experiments.—Two hundred g. of mesityl oxide and 280 cc. of aqueous

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[7] Private communication.

[1] Everest, *J. Chem. Soc.*, **115**, 588 (1919).

[2] "Organic Syntheses," Vol. I, Wiley and Sons, **1921.**

ammonia (27%) were placed in a 2.5-liter round-bottom flask which was equipped with a mechanical stirring device so fitted that the flask was nearly air-tight. Considerable heat was generated during the reaction, so that it was found necessary to cool the flask. After the system had been stirred for three hours it was found to be homogeneous. The flask was then well stoppered and allowed to stand at room temperature. Analysis showed that the greatest quantity of diacetonamine was present after the solution had stood for three days. Dry air was blown through the solution to remove the excess of ammonia. The amine solution was then dissolved in its own volume of absolute alcohol and transferred to a large buret (500–1000cc. capacity). A sample of this solution was titrated with standard oxalic acid using litmus as an outside indicator. The calculated amount of oxalic acid was placed in a large evaporating dish and dissolved in 4 liters of a mixture of 95% alcohol with a small amount of absolute alcohol. The amine solution was then slowly run into the acid while the mixture was constantly stirred. During the addition of the last half of the amine solution, the dish had to be cooled in order to avoid the formation of the neutral oxalate salt. The resulting mixture was then heated on an electric hot-plate and constantly stirred until its temperature reached 70°, when it was filtered while hot, using a Büchner funnel. The filtrate was immediately placed in a large crystallizing dish for the crystallization of the diacetonamine hydrogen oxalate. The residue was again treated with boiling alcohol and filtered; the filtrate contained a small additional quantity of the amine acid oxalate. The mother liquor was distilled until nothing further passed over at 70°, and the residue was allowed to stand for a day in a cool place. The crystals that separated were washed with cold absolute alcohol and dried; yield, 310 g., or 68%; m. p., 126–127°.

Notes.—The mixture of mesityl oxide and alcohol can be fractionated and most of the alcohol can be recovered.

The product is entirely free from triacetonamine, triacetondiamine and other troublesome condensation products, making its purification very simple. There is, however, a small quantity of ammonium hydrogen oxalate mixed with this salt to the extent of from 1 to 1.2% but since it offers no difficulty in using the product for synthetic purposes it is usually ignored. It is possible to get pure diacetonamine hydrogen oxalate by recrystallization from absolute alcohol.

From 1000 g. of acetone, with the preparation first of the mesityl oxide and then the diacetonamine, about 850 g. of diacetonamine hydrogen oxalate is usually obtained.

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