

carbon dioxide. The fact that very little carbon dioxide was evolved indicated that essentially all of the dextrose that had not been oxidized to saccharic acid had been converted to keturonic acids. Oxidation with 70% nitric acid at 55° to 60° C. for 5 hours did not affect the yield of potassium acid saccharate appreciably. The stability of saccharic acid toward further oxidation is shown in Table I. Of the keturonic acids only 5-ketogluconic acid could be isolated and identified. The presence of oxalic and L-tartaric acids, however, shows that early oxidative degradation of the keturonic acids had occurred. Longer periods of reaction at higher temperatures (Table I) increased this degradation, as is evidenced by the increase in oxalic acid formation and the decrease in reducing value of the oxidation mixture. A considerable quantity of carbon dioxide also was produced during the latter oxidations.

The course of the nitric acid oxidation of the by-product sugar acids in the potassium acid saccharate mother liquor was changed by the use of vanadium catalyst. Apparently the oxidation was directed toward the keto groups in the sugar acids (7) with cleavage at such points to produce acids of lower molecu-

lar weight together with carbon dioxide and eventually oxalic acid. Oxidative degradation of oxalic acid also occurred to some extent.

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FOOD ANALYSIS

Detection of Ethylvanillin in Vanilla Extract

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A SIMPLE METHOD of detecting the addition of ethylvanillin to pure or imitation vanilla extract has long been desired. Several more or less adequate methods have been devised, but each has its drawbacks.

The most widely used of these procedures is probably the Chenoweth peroxide test (2). Utilizing readily available reagents, this color test takes about 45 minutes [after the usual extraction procedure of the Association of Official Agricultural Chemists (7)]. The time and temperatures used are critical, and even slight deviations from the procedure may inhibit proper color development. The need for a more reliable method has been felt by analysts. The Gailey chromatographic method (3) is excellent, but requires 7 hours or more to perform. Another test frequently referred to is the Nechamkin copper-iron thiocyanate test (4), which employs three special reagents to obtain a color reaction.

The present test requires only one

reagent, alkaline 1% *p*-aminophenol solution.

Procedure

The unknown is extracted with ether according to the usual AOAC method (7). The vanillin-ethylvanillin fraction is dried, preferably over sulfuric acid, and a 1% alcoholic solution is made. To 2 ml. of this unknown, 0.5 ml. of 1*N* sodium hydroxide and 0.25 ml. of fresh colorless 1% *p*-aminophenol alcoholic solution are added and the solution is shaken.

If the unknown is ethylvanillin, a dark purplish color will develop. Vanillin gives only a pale yellow or olive tint. A 1 plus 1 mixture of vanillin and ethylvanillin will give an intermediate shade. Mixtures containing considerably less than 50% ethylvanillin do not give enough distinction to be of much practical value.

It is important that the *p*-aminophenol reagent be freshly prepared, as it is rapidly oxidized to dark-colored products, especially in alkaline media. The reagent should be prepared in a full

container, and air excluded as much as possible.

Interfering substances are removed by the usual AOAC extraction (7). Coumarin and heliotropin are among the substances normally present in imitation vanilla, which give this color reaction with *p*-aminophenol. Both these compounds are eliminated during the ether extraction and hence give no difficulty.

A known control should be run for color comparison purposes, especially where a mixture of vanillin and ethylvanillin is suspected.

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