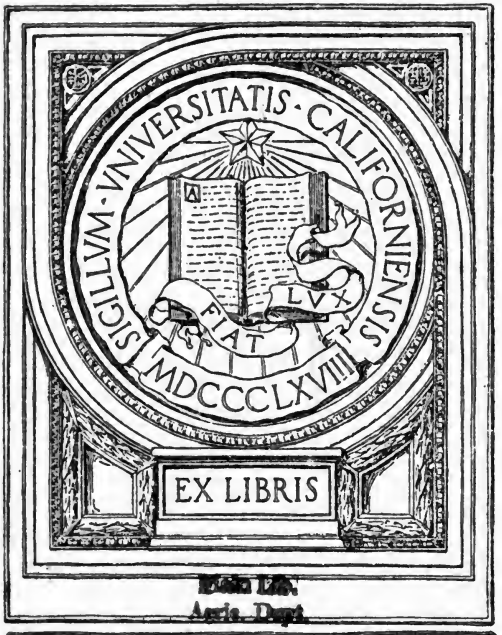

CIRCULAR NO. 7, REVISED EDITION.

United States Department of Agriculture,

DIVISION OF CHEMISTRY.

H. W. WILEY, Chief.

AMENDED METHODS ADOPTED AT THE EIGHTEENTH ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

DIASTASE METHOD FOR STARCH.

Extract 3 grams of the finely powdered substance on a hardened filter with five successive portions of 10 c. c. of ether, wash with 150 c. c. of 10 per cent alcohol, and then with a little strong alcohol. Place the residue in a beaker with 50 c. c. of water, immerse the beaker in a boiling water bath, and stir the contents constantly until all of the starch is gelatinized; cool to 55° C.; add 20 c. c. of malt extract and maintain at this temperature for an hour. Heat again to boiling for a few minutes, cool to 55° C., add 20 c. c. of malt extract, and maintain at this temperature until a microscopic examination of the residue with iodin reveals no starch. Cool and make up directly to 250 c. c.; filter. Place 200 c. c. of the filtrate into a flask with 20 c. c. of 25 per cent hydrochloric acid (specific gravity, 1.25); connect with a reflux condenser and heat in a boiling water bath for two and a half hours; nearly neutralize while hot with sodium hydrate, and make up to 500 c. c. Mix the solution well, pour through a dry filter, and determine the dextrose in an aliquot part. Convert the dextrose into starch by the factor 0.90.

Preparation of malt extract.—Digest 10 grams of fresh, finely ground malt two or three hours at ordinary temperature, with 200 c. c. of water, and filter. Determine the amount of dextrose in a given quantity of the filtrate after boiling with acid, etc., as in the starch determination, and make the proper correction.

PROVISIONAL METHOD FOR THE DETERMINATION OF PENTOSANS BY MEANS OF PHLOROGLUCOL.

A quantity of the material, chosen so that the weight of the phloroglucid obtained shall not exceed 0.300 gram, is placed in a flask, together with 100 c. c. of 12 per cent hydrochloric acid (specific gravity, 1.06), and several pieces of recently heated pumice stone. The flask, placed upon wire gauze, is connected with a condenser and heat applied, rather gently at first, using a gauze top to distribute the flame, and so regulated as to distill over 30 c. c. in about ten minutes, the distillate passing through a small filter paper. The 30 c. c. driven over are replaced by a like quantity of the dilute acid added by means of a separatory funnel and in such a manner as to wash down the particles adhering to the sides of the flask, and the process continued until the distillate amounts to 360 c. c. To the completed distillate is gradually added a quantity of phloroglucol (purified if necessary) dissolved in 12 per cent hydrochloric acid, and the resulting mixture thoroughly stirred. The amount of phloroglucol used should be about double that of the furfural expected. The solution first turns yellow, then green; and very soon an amorphous greenish precipitate appears, which grows

rapidly darker, till it finally becomes almost black. The solution is made up to 500 c. c. with 12 per cent hydrochloric acid, and allowed to stand over night.

The amorphous black precipitate is filtered into a tared gooch through an asbestos felt, washed with 100 c. c. of water, dried to constant weight by heating four hours at 100° C, cooled and weighed in a weighing bottle, the increase in weight being reckoned as phloroglucid. To calculate the furfural from the phloroglucid, use the following formulæ:

Phloroglucid (less than and up to 0.2 gram) ÷ 1.82 = Furfural.
Phloroglucid (from 0.2 to 0.3 gram) ÷ 1.895 = Furfural.
Phloroglucid (from 0.3 to 0.4 gram) ÷ 1.92 = Furfural.
Phloroglucid (from 0.4 gram) ÷ 1.93 = Furfural.

To calculate the furfural to pentosan or pentose, use the following formulæ:

I. (Furfural − 0.0104) × 1.68 = Xylan.
II. (Furfural − 0.0104) × 2.07 = Araban.
III. (Furfural − 0.0104) × 1.88 = Pentosan.
IV. (Furfural − 0.0104) × 1.91 = Xylose.
V. (Furfural − 0.0104) × 2.35 = Arabinose.
VI. (Furfural − 0.0104) × 2.13 = Pentose.

QUALITATIVE TEST OF THE PURITY OF THE PHLOROGLUCOL.

Dissolve a small quantity of the phloroglucol in a few drops of acetic anhydrid, heat almost to boiling, and add a few drops of concentrated sulphuric acid. A violet color indicates the presence of diresorcol. A phloroglucol which gives more than a faint coloration must be purified by the following method:

METHOD OF PURIFICATION OF PHLOROGLUCOL.

About 300 c. c. of hydrochloric acid (specific gravity, 1.06) are heated in a beaker, and 11 grams commercial phloroglucol added in small quantities at a time, stirring constantly until it has almost entirely dissolved. Some impurities may resist solution, but it is unnecessary to dissolve them. The hot solution is poured into a sufficient quantity of the same hydrochloric acid (cold) to make the volume 1,500 c. c. It is allowed to stand at least over night—better several days—to allow the diresorcol to crystallize out, and filtered immediately before using. The solution may turn yellow, but this does not interfere with its usefulness. In using it the volume containing the required amount is added to the distillate.

METHOD FOR ESTIMATING GALACTAN.

Extract 3 grams of the substance on a hardened filter with five successive portions of 10 c. c. of ether, place the extracted residue into a beaker about 5.5 cm. in diameter and 7 cm. deep, together with 60 c. c. of nitric acid of 1.15 specific gravity, and evaporate the solution to exactly one-third its volume on a water bath at a temperature of 94° to 96° C. After standing twenty-four hours, add 10 c. c. of water to the precipitate, and allow it to stand another twenty-four hours. The mucic acid has in the meantime crystallized, but is mixed with considerable material only partially oxidized by the nitric acid. The solution is therefore filtered through filter paper, washed with 30 c. c. of water to remove as much of the nitric acid as possible, and the filter and contents replaced in the beaker. Thirty c. c. of ammonium carbonate solution, consisting of 1 part ammonium carbonate, 19 parts water, and 1 part strong ammonia, are added, and the mixture heated on a water bath at 80° C. for fifteen minutes with constant stirring. The ammonium carbonate takes up the mucic acid, forming the soluble mucate of ammonia. The filter paper and contents are then washed

several times with hot water by decantation, the washings being passed through a filter paper, to which the material is finally transferred and thoroughly washed. The filtrate is evaporated to dryness over a water bath, avoiding unnecessary heating, which causes decomposition; 5 c. c. of nitric acid of 1.15 specific gravity are added, and the mixture thoroughly stirred and allowed to stand for thirty minutes. The nitric acid decomposes the ammonium mucate, precipitating the mucic acid, which is collected on a tared filter or gooch, washed with from 10 to 15 c. c. of water, then with 60 c. c. of alcohol, and a number of times with ether, dried at 100° C. for three hours, and weighed. The mucic acid multiplied by 1.33 gives galactose, and this product multiplied by 0.9 gives galactan.

WILLIAM H. KRUG,
Referee on Foods and Feeding Stuffs for 1901.

Approved:
JAMES WILSON, *Secretary.*

WASHINGTON, D. C., *April 18, 1902.*

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