beet molasses will not keep more than a month under our conditions."

The use of beet or cane molasses, or a mixture of both in varying proportions, in molasses feeds, is a problem yet to be determined by many manufacturers. Keeping qualities, palatability, effect on the animals and popularity of the feed are some of the problems to be considered.

Just about twelve years ago Louisiana blackstrap was a nuisance and the sugar planters had a hard time to dispose of it. On account of its value for feeding purposes the demand has become so great that it is now selling for \$19 to \$21 per ton retail, perhaps the highest figure it has ever reached. Louisiana blackstrap has been so scarce that some of our feeders are purchasing second molasses for feeding purposes. The time is coming when our manufacturers of commercial molasses feeds will be forced to obtain some of their supply of cane molasses from the tropical countries.

On the whole, there is a bright future for molasses feeds even at the high prices feeds are carrying. It is possible for manufacturers to put out wholesome feeds free from adulteration with inferior products and make a good profit. In the past two years there has been a great improvement in this class of feed, and some concerns have been forced to go out of business on account of flagrant adulteration of their feeds. The time has come when only those feeds that are pure and honest mixtures will meet with success, as adulterated feeds even if they do have a high chemical composition, most always show poor results on the animals to which they are fed.

LOUISIANA STATE EXPERIMENT STATION,
BATON ROUGE, LA.

A RAPID METHOD OF HYDROLYZING STARCH.

By GEO. A. OLSON.

Received May 3, 1909.

Numerous methods for the hydrolyzing of starch have been devised in the past, among which may be mentioned Sachsse's,¹ Märker's,² Guichard's,³ and others. Chemists who have tried these methods know what a long and tedious procedure it is to wash and hydrolyze materials according to any one of them. After a futile attempt to find a method that would give concordant results, the members of the A. O. A. C. finally adopted the Sachsse

method as official.¹ While this method gives fairly satisfactory results, it still has the disadvantage of time when compared with the method which will be discussed in the following pages. Where two and one-half hours were required to hydrolyze starch it is now possible to accomplish this in about four minutes.

Preparation of Samples.—Before discussing this method, it is deemed advisable to give a few suggestions as to the preparation of the samples to be hydrolyzed. Too much stress cannot be laid upon the importance of securing a uniform, representative sample. The material should be finely ground and thoroughly mixed. In case of some substances, *e. g.*, oats, etc., it is preferable after the first grinding to separate the hulls from the starchy material and grind separately, thus insuring a more uniform mixture.

Method of Hydrolysis.—Place in a 500 cc. Kjeldahl flask one gram of material. Tap the neck of the flask in order to settle the fine particles adhering to the sides. Mix with this weight ten cc. of distilled water and rotate slowly and steadily (one revolution per second) so as not to wash any of the sample too high up on the sides of the flask. Next cautiously add 6 cc. of sulfuric acid (specific gravity 1.84) and rotate somewhat more rapidly than in the first instance. After the acid and material have been thoroughly mixed hold the flask in one hand over a flame about two inches high and rotate about one revolution per second at first and then as the mixture increases in temperature rotate with an accelerated motion up to five revolutions per second. Continue rotating at this rate until the mixture becomes nearly transparent. At this stage add 10 to 15 cc. of distilled water, continue rotating and heat again, this time to boiling, cool, neutralize with sodium hydroxide (using phenolphthalein as indicator), cool again, transfer to 250 cc. volumetric flask, fill with distilled water to mark and proceed according to one of the recognized methods for dextrose determination.

Notes on Manipulation.—The hydrolysis commences with the addition of acid. In case of cereals, bran, shorts, etc., a slight coloring is first observed, then a milkiness, and finally the solution becomes almost transparent, varying in color from straw to a light brown. A further heating intensifies the color, and if this process is continued too far there is danger of caramelizing a part of the

¹ *Chem. Centralbl.*, 1877, p. 732; *Journ. Anal. Chem.*, II, 153.

² *Chem. Zeit.*, IX, 319; *Ztschr. anal. Chem.*, 24, 617.

³ *Bull. d. l. Soc. Chim. d. Paris* [3], 7, 554.

¹ *U. S. Dept. of Agr., Bur. of Chem., Bull.* 107 (rev. ed.), p. 53.

dextrose formed. Since no sharp line can be fixed, it will be necessary for the operator to use some judgment as to the extent that this process should be continued. A moderate overheating does not materially affect the results. On the second addition of water a milkiness reappears which again disappears on heating. In most cases there is enough coloring matter present in the material to act as an indicator; phenolphthalein, however, gives a sharper color and is therefore recommended. If the material tends to creep up on the sides of the flask during the rotating process, a slight agitation will obviate the difficulty. The time required to hydrolyze eight samples, comprising oats, barley, wheat and corn, was thirty minutes or a little less than four minutes for each sample.

Hydrolysis of Substances that Do Not Mix Well.—Occasionally it happens that the substance to be analyzed is difficult to mix uniformly. Under such circumstances larger samples should be used for the hydrolysis; from 2 to 5 grams of material can be as readily hydrolyzed as 1 gram. When larger quantities of material are taken the proportion of water and acid must be increased correspondingly, *e. g.*, with 5 grams of material add 50 cc. of water and 30 cc. of acid. With larger amounts of material, however, it is more difficult to determine the stage when the starch is completely hydrolyzed.

Accuracy of Method.—A considerable number of comparative analyses have been made by the official method (Sachsse) and the one herein described. The figures given in the following table and taken at random show the substantial accuracy of the method. The preliminary washing required by the official method is not followed in the one devised by the writer. A separate determination for sugar will be necessary for materials containing appreciable amounts of this component. Allihn's¹ method for sugar was used and the copper weighed as cuprous oxide.

It will be noted from the results given in the following table that slightly higher values for starch were obtained with the new method, probably due to the fact that the samples were not treated with alcohol, ether and water previous to hydrolysis. By omitting the preliminary washing much time was saved, particularly with legumes and oil meal; in some cases from five to eight hours were required to wash the samples before they were ready for hydrolysis.

¹ U. S. Dept. of Agr., Bur. of Chem., Bull. 107 (rev. ed.), p. 49.

COMPARATIVE RESULTS WITH NEW AND OFFICIAL METHOD.

	Official method. Starch, per cent.	New method. Starch, per cent.
Peas.....	44.03	44.73
	44.31	44.27
Oats.....	41.67	42.03
	42.13	42.48
Oats.....	41.54	41.83
	41.80	42.03
Wheat.....	52.92	52.74
	52.61	52.84
Wheat.....	45.90	46.33
	46.09	46.01
Wheat.....	53.48	53.84
	52.91	53.42
Barley.....	54.45	54.63
	54.21	54.16
Barley.....	54.87	55.62
	55.31	55.53
Potatoes (dry).....	66.78	66.78
" (calc. in original).....	16.34	16.34
Potatoes (dry).....	69.03	69.53
" (calc. in original).....	17.48	17.60
Oil meal.....	21.60	21.69
White corn.....	59.40	59.69
Yellow corn.....	56.61	56.34
Soy beans.....	9.93	10.50
Soy bean cake.....	8.17	8.63
Bran.....	44.57	45.80
Shorts.....	51.85	53.18
Baking powder.....	25.34	26.64
Baking powder.....	37.80	37.89

Diastase Method.—With one single exception (malt) the diastase method gave lower starch values than either of the other two, *e. g.*, white corn 55.44 per cent., potatoes (first one in table) 62.93 per cent., and bran 38.63 per cent. The material was gelatinized by boiling with water containing 1 per cent. of glycerol, cooled and converted with Merck's pure diastase.¹ The blue color with iodine was in evidence for hours. Disregarding the preliminary washing, a sample of malt gave 59.31 per cent. starch by Sachsse method, 59.40 per cent. starch by the writer's method and 59.40 per cent. starch by mashing. The mashing was conducted at temperatures increasing from 45 to 73° C. for one-half hour and maintained at 73° C. for another half hour.

Value of Starch Determination in Feeding Stuffs.—The starch content of a cereal, potato, or a feeding stuff is as valuable a component as a fat, protein or ash constituent, yet it is seldom determined. It has been the custom in the past, especially in fodder analyses, to call the undetermined constituents *nitrogen-free extract* or *carbohydrates*, and in turn the carbohydrates are frequently spoken of as *starch*. With the new method it is now possible to determine with facility the acid-hydrolyzable part of nitrogen-free extract and thus reduce the undetermined constituents to a minimum.

¹ Bought at least three years ago.

When a starch is hydrolyzed with an acid, there is, of course, always the danger of converting a part or all of the pentosans, cellulose and allied bodies into sugars. Again the rôle played by diastase upon starch has not been settled. If, however, the analyst is content in reporting 6.25 times nitrogen as protein, the ether extract as fat, and the residue obtained after boiling the ether-extracted material with acid and alkali as crude fiber, then on equally good grounds he should be content in reporting starch as obtained by acid hydrolysis.

CHEMICAL LABORATORIES,
WASHINGTON STATE EXPERIMENT STATION,
PULLMAN, WASHINGTON.

THE PREPARATION OF SOIL SAMPLES FOR CHEMICAL ANALYSIS.

By G. M. MACNIDER.

Received March 15, 1909.

The following investigation was undertaken to determine the proper sized sieve to use in preparing soil samples for chemical analysis when the total amount of plant food elements are to be determined. When the available or soluble plant food is determined, by the Official Method of the Association of Official Agricultural Chemists or by some other method using some solvent to abstract the plant food, a preparation using a sieve with perforations smaller than 2 mm. in diameter seems to be adequate, as it has been shown by several investigators that the greater portion of the soluble plant food is found in the smaller particles of the soil, *i.e.*, the very fine sands, silt and clay. The Official Method of preparation has, however, been recently changed from 0.5 mm. sieve to 1 mm. sieve.¹

The size of sieve to be used, which is to determine what portion of the soil shall be taken for analysis, is of course an arbitrary standard. In making total analyses it is very necessary that the standard adopted be such that the analyses will give the fairest idea of the plant food contained in the soil. With some very coarse soils even the 2 mm. preparation excludes from the analysis particles which it would seem should be included. From the data here presented and from a large number of chemical analyses of soils made in this laboratory we have arrived at the conclusion that the most satisfactory method of preparation, when the total plant food constituents are to be determined, is to pass the

sample through a sieve having circular perforations 2 mm. in diameter and take the portion passing the sieve as the "fine earth" which is to be analyzed and to regard the portion not passing the sieve as coarse gravel which should not be classed as "fine earth" or soil. This method also gives a uniform preparation for both the chemical and mechanical analyses.¹

The method of preparation is as follows:

Spread the sample on a board covered with heavy paper and roll with a wooden rolling pin to break up lumps. The sample is then passed through a sieve having circular perforations 2 mm. in diameter. If there are any coarse particles they should be put in a mortar and rubbed with a rubber-tipped pestle to remove the adhering, fine particles. This operation should be repeated till the coarse particles are clean. When all the fine particles have been passed through the sieve the sample should be thoroughly mixed before the portion for analysis is taken out. In some samples the large particles passing the 2 mm. sieve may be large enough to interfere with the fusion in the analysis. In such cases a portion may be pulverized for the determinations requiring fusion.

In the following table are analyses of representative soils made on the 0.5 mm. preparation and on the 2 mm. preparation. The 0.5 mm. was taken for comparison as this was for a number of years the official method of preparation of soil samples and a large number of analyses have been made in this laboratory on that preparation. The table shows the percentages of plant food constituents and the pounds per acre of each constituent to a depth of 8 inches, calculated from the apparent specific gravity, also the apparent specific gravity and the per cent. of fine soil passing the sieve in each preparation. The samples have been selected so as to represent a large variety of soils in North Carolina, the sands and sandy loam soils of the Atlantic Coastal Plain, the coarse sandy loams of the Piedmont Plateau and the sandy loams of the Mountain Region. Comparative analyses were not made of the clay and loam soils as practically all of these pass the 0.5 mm. sieve.

DISCUSSION OF RESULTS.

Reference to the table will show the differences in the analyses so that it is only necessary here to point out a few of the more marked differences.

¹ The method of mechanical analysis in use in this laboratory is the same as that used by the Bur. of Soils. See *Bull.* 24, *Bur. of Soils*, by L. J. Briggs.

¹ U. S. Dept. of Agr., *Bur. of Chem.*, *Bull.* 107 (revised).