cold, while the adulterated sugar will continue in the state of a pasty, sticky mass.

This test is founded on the property possessed by cane sugar to form viscous, uncrystallizable compounds when mixed with many organic or inorganic substances, among which are anhydrous and hydrated dextrose. An example of compounds of this kind is *molasses*, obtained as as a residuum in sugar manufacture.

As long as a mixture of cane sugar and starch glucose is sufficiently dry, it may look fair enough, as the elements which form molasses are kept from combining by want of water. The sugar adulterator is well aware of this, and he is careful to dry his sugar before mixing with glucose. Indeed, one characteristic of adulterated sugars is that they are always dryer than refined sugars of the same grade, which are known as *coffee sugars*, and are always sold moist.

As soon as sufficient water is added to an adulterated sugar, and moderate heat is applied, enough viscous syrup is formed to make the sugar sink into a paste, which remains permanently soft.

Useful indications may be obtained as to adulteration by starch glucose, by means of Fehling's solution. An ordinary refined coffee sugar will rarely show more than 5 percent. of glucose, while a sugar adulterated with the usual dose of starch glucose, will show about 20 per cent. of glucose.

ANALYSIS OF SOAP.

By Dr. Albert R. Leeds.

In the analysis of soap it is necessary to determine :—

(1) Water.

(2) Uncombined fat.

(3) Soap consisting of (3a.) combined Fatty Acids, estimated as Fatty Anhydrides, and (3b.) Combined Alkali, usually Soda (Na_2O).

(4) Uncombined Alkali.

(5) Glycerine.

(6) Resin.

(7) Sodic Carbonate.

(8) Sodic Chloride.

(9) Sodic Sulphate.

(10) Sodic Silicate—consisting of (10a) Soda combined in Silicate, and (10b.) Silica.

(11) Starch.

(12) Insolible Residue, or mineral impurities, such as talc, clay, ochre, sand, etc.

(1) *Water.*—Weigh out about 5 grms. in very fine, small shavings upon a dried, weighed, plaited filter. Dry at 110° until weight is constant. The loss is water.

(2) *Uncombined fat.*—Transfer the filter containing the dried soap to the funnel connected with the return cooler as in the determination of the albuminoids in milk, and connect with the funnel a small tared flask containing 50cc. petroleum ether. After complete extraction distil off the ether, and the residue in the flask dried at 110° will be the uncombined fat.

(3) Soap, (4) Free Alkali, (5) Glycerine. Allowing the funnel with the soap freed from moisture and from fat to remain on the return cooler, attach to it a flask containing about 75cc of 95% alcohol and extract. To the alcoholic solution add a few drops of phenol-phthalëin, if free alkali be present neutralize with normal sulphuric acid and calculate the amount of uncombined soda.

After neutralization add a large excess of water and boil off the alcohol. To the aqueous solution, add a large excess of normal sulphuric acid. Boil, cool and decant through a small filter, wash with hot water and decant after cooling through the filter until litmus paper is no longer reddened by the washings. The filtrate consists of the combined soda and glycerine; the residue of fatty acids and resin. Neutralize the filtrate with normal soda solution and calculate the amount of combined soda as Na_2O. Evaporate to dryness and extract the glycerine with absolute alcohol. Transfer the alcoholic solution to a tared flask, distil off the alcohol, dry at 100° and weigh the residue as glycerine.

Fatty acids and Resin. Dissolve the small amount of the fatty acids and resin that may be on the filter, through which the decantation was effected, with a little petroleum ether, add the solution to the larger bulk in the beaker, evaporate off the ether, dry at 110° and weigh the combined fatty acids and multiply this result after subtracting the amount of resin by 0.97, and the product is the Fatty Anhydrides and resin.

(6) Resin. The resin was separated from the fatty acids according to the method proposed by Gladding (American Chemical *Journal*, Vol. III, p. 416.)

The original article by Gladding, reads as follows :—

THE QUANTITATIVE SEPARATION OF ROSIN FROM FATS.

"About 0.5 gram of the fat acids containing the rosin is introduced into a small flask. Twenty cubic centimetres of 95 per cent. alcohol are added, and the flask rotated till the fat acids and the rosin are dissolved. A drop of phenol phthalëin is now added, and then a saturated solution of caustic potash in alcohol drop by drop, with thorough agitation after the addition of each drop, until the deep red color characteristic of alkalinity is obtained. One or two additional drops are now added, and the flask placed on a water-oven and kept at the temperature of boiling alcohol for ten minutes to ensure the saponification of the last portions of fat. The flask should be loosely corked. It is now cooled and the contents washed into a graduated 100 cc. cylinder by means of concentrated ether. The cylinder is filled with the ether exactly to the 100 cc. mark ; then corked, best by a common cork twisted tightly in, and the contents mixed by a moment's shaking. About one gram of C. P. *neutral* silver nitrate is now rubbed to an impalpable powder in a a small mortar and then introduced into the cylinder. The latter is vigorously shaken for ten or fifteen minutes until the flocculent precipitate of silver stearate and oleate collects in the same manner as silver chloride upon shaking, and settles clear. Fifty to seventy cc. of the supernatant liquid are now syphoned off by means of a slender syphon, previously filled with ether, into a second 100 cc. cylinder, passing the liquid through a small filter paper if. it is not perfectly clear. A small quantity of pulverized silver nitrate is shaken up with this to make certain that all the oleate and stearate are precipitated. If no flocculent precipitate appears this is the case. Twenty cc. of a mixture of hydrochloric acid and water, one-third the former and two-thirds the latter, are now added, and the cylinder vigorously shaken until the decomposition of all the silver salt present is complete. After the silver chloride has settled, an aliquot portion of the supernatant ether solution is syphoned off into a platinum dish and evaporated on the top of a water-oven to dryness. The residue is rosin containing a small amount of oleic acid, which can be accurately allowed for."

The following experiment gives the allowance to be made :

"*Exp.* II. One-half a gram of pure fat acid was treated in the above way. Eighty cc. shaken with HCl and evaporated gave

0.0188 gram of residue ; 10 cc. contain therefore 0.00235 gram of oleic acid. This very small and accurate correction of 0.00235 gram for every 10 cc. of liquid shaken up with HCl was applied in the subsequent experiments."

The manner of calculating the allowance may be most clearly shown by another experiment :

"*Exp.* IV. One-half a gram of the fat acids from linseed oil was add to 0.050 gram of rosin and treated as the above.

"Seventy cc. shaken with HCl gave 0.0517 gram of residue. Subtracting the allowance for 70 cc., namely, 0.0164 gram, leaves 0.0353 gram of 0.0504 gram of rosin on the whole quantity taken instead of 0.050 gram taken."

About 0.5 grm. of the mixture of the fatty acids and resin are dissolved in twenty cc. of strong alcohol and, with phenol-phthalëin as an indicator, soda is run in to a slight supersaturation. The alcoholic solution, after boiling for ten minutes to ensure complete saponification, is mixed with ether in a graduated cylinder till the volume is 100 cc. To the alcoholic and ethereal solution 1 grm. of very finely powdered Ag. NO_3 is added and the contents of the cylinder are shaken thoroughly for ten or fifteen minutes. After the precipitate has settled, 50 cc. are measured off and if necessary filtered into a second graduated cylinder. A little more Ag. NO_3 is added to see if the precipitation is complete and then 20 cc. of dilute hydrochloric (1:2) to decompose the silver resinate. An aliquot part of the ethereal solution in the cylinder is evaporated in a tared dish and weighed as resin, deducting a small correction (for 10 cc. 0.00235 grm.) for oleic acid. The amount of resin substracted from the combined weight of fatty acids and resins, as found before, gives the fatty. acids. (7) Sodic carbonate ; (8) Sodic chloride ; (9) Sodic sulphate ; (10) Sodic silicate ; (11) starch ; (12) insoluble residue. The filter in the funnel connected with the return cooler, after treatment with alcohol contains the mineral constituents of the soap. The contents of the filter are washed with cold water till the washings amount to 60 cc. The filter is then dried and weighed. The weight gives the insoluble residue and starch.

The starch is converted into $C_6H_{12}O_6$ with dilute acid and titrated with Fehling's solution. The weight of starch found substracted from the total weight of insoluble residue and starch gives the insoluble mineral constituents.

The aqueous solntion of 60 cc. just mentioned, is divided into

four equal parts, in one of which is determined the carbonate of soda titration and in the other parts, the chloride, the sulphate and the silicate respectively, by any convenient method.

My thanks are due to my assistant, Dr. Edgar Everhart, for his assistance in working out the details of this scheme.

SCHEME FOR SOAP ANALYSIS.

Weigh out 5 grms. Dry at 110°. Loss corresponds to water. Treat with petroleum ether.

Extract is uncombined fat. Dry at 110° and weigh.

Residue = Soap and Mineral constituents. Treat with alcohol.

Extract = Soap (Fatty anhydride, resin and combined alkali), Glycerine and Free alkali. Add two or three drops of phenol-phtalëin. If necessary, titrate with normal H_2SO_4.

H_2SO_4 used corresponds to free alkali. Calculate as NaHO

Add a large excess of water and boil off the alcohol. Decompose with excess of normal H_2SO_4. Boil, filter and wash.

Filtrate. Combined soda and glycerine. Titrate with normal soda solution.

H_2SO_4 used corresponds to combined soda in soap. Calculate as Na_2O.

After titration evaporate to dryness on the water bath. Treat with absolute alcohol. Evaporate the alcoholic solution to dryness in a tared dish and weigh as glycerine.

Residue—Fatty acids and Resin. Dry at 110° and weigh. Dissolve an aliquot part in 20 cc. strong alcohol, and using phenol-phtalëin as an indicator, saponify with soda in slight excess. Boil, cool and add ether to 100 cc. Decompose with $AgNO_3$ by adding in fine powder, and shake well for 10 minutes. Allow to settle.

Precipitate. Stearate palmitate and oleate of silver.

Solution—Resinate of silver. Filter 50 cc. from the total 100 cc. Decompose with 20 cc. HCl (1:2) Allow the AgCl to settle and evaporate an aliquot part of the ethereal solution in a tared dish. Dry at 110° and weigh. After applying correction for oleic acid the weight corresponds to the resin. This weight subtracted from the combined weight of fatty acids and resins, gives the fatty acids.

Residue.—Na_2CO_3. NaCl, Na_2SO_4, sodium silicate, starch and insoluble residue. Wash with 60 cc. water.

Filtrate Na_2CO_3, NaCl, Na_2SO_4, and Sodium silicate. Divide into 4 equal parts.

Na_2CO_3 Titrate with normal H_2SO_4 and calculate as Na_2CO_3.

NaCl Titrate with $AgNO_3$, or weigh as AgCl. Calculate as NaCl.

Na_2SO_4 Weigh as $BaSO_4$. Calculate to Na_2SO_4.

Sodium silicate. Decompose with HCl, and determine soda combined in Silicate and Silica.

Residue—Starch and Insoluble Residue. Dry the filter. The weight is the Starch and Insoluble Residue Starch. Convert the starch into $C_6H_{12}O_6$ Titrate with Fehling's solution. Subtract weight of starch found and difference is Insoluble Mineral constituents.