and having *double* brass wire strung across for keeping the beakers about $^3/_8$ of an inch apart. These two wires, side by side, passing between each beaker, prevent clashing and resulting breakage. The bottom is lined with two-ply rubber, perforated.

The farmer's tickets accompanying the sample are placed in a tin case having a large brass number soldered to it, and a corresponding number is upon a basket. The basket, placed with the number facing the chemist, is filled with beakers in a definite order, and the sample tickets are slipped into the corresponding tin box in a similar order or arrangement.

After digestion and cooling, the beaker is wiped outside (or allowed to drain and dry) and placed upon a balance, and with the contents brought with a few drops of water to the desired mass, *i. e.*, 209.2 grams plus the counterpoise. The analysis is then completed as usual.

This method will of course apply to sugar-cane and bagasse analyses, where, weight being applied instead of volume, the quantities may be proportionately increased and the difficulty in sampling thereby overcome.

CARO, MICHIGAN, March 10, 1906.

[CONTRIBUTION FROM THE BUREAU OF CHEMISTRY, U. S. DEPARTMENT OF AGRICULTURE].

THE RAPID DETERMINATION OF WATER IN BUTTER.

BY G. E. PATRICK.

Received August 11, 1906.

SINCE the laws regulating the percentage of water in butter have come into vogue there has grown up a persistent demand for some method whereby the butter-maker, the renovator and the butter merchant can easily and quickly determine the amount of water in his product or his commodity. The most exacting demand is that of the butter-maker in the creamery, or the churn-man in the renovating factory, who needs a method which will yield its results in a few minutes, while his butter-worker stands waiting with its load, further working to be dependent upon the result of the test.

The writer has studied this problem at intervals for two years or more and has at last found a method which is sufficiently accurate, and at the same time so simple in principle and so rapid

in execution that it appears in large measure to meet the demands of the butter-maker.

At the outset of his investigations, before the idea of this simple method had suggested itself, the writer studied first, and very exhaustively, Poda's method[1] which seemed the most promising of the sulphuric acid centrifugal methods proposed up to that time, but to his disappointment found it quite inadequate for the purpose in view, both as regards speed and accuracy. Next he tried several rather elaborate schemes which presented themselves to mind, all attractive in theory but all proving unsuccessful in practice, for which reason it is needless to detail them here.

Finally, discouraged in these more elaborate lines of effort, he turned back to the familiar, commonplace principle of expelling the water by heat; but the ordinary way of doing this, namely, at the temperature of boiling water, is far too slow, requiring several hours' time, so a glycerol bath, easily affording a temperature upwards of 150°, was arranged of form and size to allow of immersing, to the neck, a tall slim flask (assay flask) containing the weighed portion of butter. In this way the water was expelled in twelve to fifteen minutes, the temperature being kept at 150° to 155°, and the whole determination, including both weighings, was completed in about twenty-five minutes.

The results were sufficiently accurate, and the method seemed promising, but the time consumed by the test was still too great, and moreover, the glycerol bath—one of sufficient size to allow of immersing the flask in an inclined position, to prevent loss by spattering—had to be heated up beforehand, and this required quite a long time. How to further shorten the time demanded of the operator was the question.

This change of plan suggested itself: To replace the assay flask by a wide test-tube, to boil off the bulk of the water *over a naked flame*—there being no danger of too high a temperature at this stage—and to finish the drying in a *small* glycerol bath, deep and narrow, in which the tube could at that stage be held vertical without danger of loss by spattering. Such a bath was made of a piece of steam pipe 15 cm. long and of 38 mm. bore, capped at the lower end, and at the upper end screwed into a

[1] Z. Unters. Nahr. Genussm. 4, 492 (1901).

cup-shaped "floor-plate," by which the whole was suspended by a firm ring support. It was charged with only four fluid-ounces (about 120 cc.) of glycerol; a "dummy" tube, kept in the bath while it was heating, raised the surface of the glycerol to the mouth of the bath. The flame of an alcohol[1] lamp heated this bath in fifteen minutes to 130–140°, a temperature high enough to finish the drying of the sample in three to five minutes, after the water had been nearly all expelled by the naked flame.

The plan worked well, but still required about twenty minutes for the complete test; and to regulate the temperature of so small a bath required considerable attention.

All of the above is preliminary, by way of reaching the important point, which is this: After a few trials it was found that just as good results could be obtained by *dispensing with the bath entirely* and drying the sample completely over the naked flame, of course using due care not to overheat. That satisfactory results could be obtained in this manner, uniformly, was a surprise; but the writer has obtained such results on many samples of both creamery and renovated butter, so many in fact that he does not hesitate to declare the method perfectly feasible and that it will give sufficiently accurate results for the purpose here in view, in the hands of any careful person, with a little practice. The complete test, including both weighings and the calculation, can be made in fifteen minutes, and after a little experience the results obtained will seldom if ever be more than 0.3 per cent. from the truth, and frequently less than 0.1 per cent.

The most objectionable feature of the method, from the standpoint of the butter-maker, is that it requires a good balance, one sensitive to say 5 mg. In his own experiments the writer weighs to 1 mg. or less, but this is not necessary. The amount of butter taken for the test may vary from 10 to 18 grams; 12 to 16 grams is perhaps the best amount. With such a charge an error as large as 15 mg. means only 0.1 per cent.

Beside the balance the only things required are:

An alcohol lamp with a wick (made of ball wicking) about 17 mm. thick and giving a flame 75 to 90 mm. tall. It should stand in a place free from drafts in order to afford a steady flame.

Test-tubes—several should be kept on hand to provide for

[1] An alcohol lamp was used in all of this work because gas is not available in most creameries.

breakage—190 mm. in length and 35 mm. inside diameter, made of rather thin glass, of a quality especially adapted to stand boiling.

A small beaker to hold the tube upright while it is being weighed and charged.

A strong wire test-tube holder, large and strong enough to hold the tube firmly while it is shaken and rotated during the heating.

A charging tube—a tube of heavy glass, 140 mm. in length and of 21 or 22 mm. inside diameter—open at both ends, and a ramrod, of any material, fitting loosely into this tube, to force its contents through. In the laboratory a test-tube makes a convenient ramrod.

To charge the tube, small portions of butter, taken with a knife from the homogeneous sample to be tested, are successively forced into one end of the charging tube until it is judged that 12 to 16 grams have been secured, the tube is then lowered, clean end down, well toward the bottom of the previously weighed or counterpoised test-tube, and the butter forced out by the ramrod. Or the butter, in a semi-fluid state, may be poured directly into the test-tube.

The weight being taken, the drying is conducted in the following manner.

The tube, gripped firmly with the tube-holder a little above the charge, is heated at first cautiously, and throughout the operation with almost constant rotation, in order to avoid local overheating—with renovated butter to avoid, also, violent boiling and spattering. The first foam (referring now to genuine butters, for renovated butters produce no foam) can usually be dispersed by merely shaking the tube, but the foam which appears later, after most of the water is boiled off, and which will overflow the tube unless the heating be cautiously done at this stage, is persistent and can be dispersed only by heating the *upper part* of the tube, passing it repeatedly through the flame, lengthwise, exposing first one side and then the other to the heat, and by *vigorous* shaking. This flaming of the tube above the charge is often useful even before the final foaming occurs, as it facilitates the dispersing of the foam later, when it threatens to overflow the tube. During the boiling off of the water the tube should be held inclined at an angle of 45°.

When the large mass of foam has been nearly all dispersed in

the manner described, the lower part of the tube is agian heated enough to make certain that no more foam can be produced, but not enough to cause any decided darkening in the color of the butter. The drying is then completed by alternately heating the tube *above the charge*—passing it repeatedly through the flame as before—and rotating vigorously to throw the fat and remaining foam upon the heated glass. The temperature of the latter should be just below that at which it will "sizzle" when touched by the moistened finger. This treatment, continued for a minute or two, will reduce the foam to a layer of not more than 3 to 6 mm. in thickness and will slightly darken the color of the butter. This will now be of a light muddy yellow, or a pale brownish yellow, or perhaps even a dark orange-yellow color, depending upon the original color of the butter, which should be carefully noted at an earlier stage of the operation. More than a moderate discoloration should be avoided, as indicating overheating. The small amount of foam remaining contains only a very minute quantity of water, if indeed any at all. As much of the foam should be dispersed as possible, with only slight discoloration of the butter.

With renovated butters the chief danger to be guarded against is loss by sputtering or violent boiling. Such loss is avoided by cautious heating, by rotating the tube almost constantly, and by holding it inclined even more than 45° from the vertical, after the boiling has begun. When the water has been nearly all boiled away, that condensed upon the walls of the tube is expelled by flaming the latter, in the manner described for dispersing the foam of genuine butters. The charge is then heated again cautiously, with *vigorous* rotation of the tube, which is now held vertical, until it is evident that all water has been expelled from the curd covered by the butter oil—this point being determined by frequent examination—care being constantly taken to avoid discoloration of the curd, so far as this is possible. Then the tube, above the charge, is again flamed cautiously, in the manner previously described, to complete the drying. The curd on the sides of the tube should now be no more discolored than to a pale lemon-yellow, while that on the bottom will be considerably darker, ranging from muddy orange-yellow to brownish yellow or yellowish brown. After cooling, the colors are a trifle darker than when the tube is hot. The aim should, of

course, be to discolor the curd as little as possible, while completely drying it.

The tube is now cooled, first with warm or hot water, cautiously at first to avoid breaking, then with cold water at about 15°, until the tube feels cold to the hand, then wiped dry and immediately weighed.

These details take long to describe and the process sounds difficult, but it is not, as a little practice will convince any one. Any careful person can, after a little practice, make the test successfully.

As regards the use of this method by the butter-maker for his immediate guidance in the working of his product, the greatest difficulty lies not in making the test, but in quickly obtaining a small sample for testing that truly represents the large mass of butter in the worker—at least such is the opinion of the writer at the present time. Also, the question presents itself, how nearly does a true sample of the finished butter as it lies in the worker, agree in respect to water content with a true sample of the same butter after it is packed in tubs?

Definite knowledge upon these points is desirable, and must be had before a rigid interpretation can be placed upon the results obtained with any immediate control-test in the butter factory.

Just as this article is being sent to the Journal the writer learns that a method almost identical with the one here described is in use in certain creameries in the West. The principle *is* identical. The butter sample is dried in a flask, directly over the flame of a gasoline torch, according to the oral information received.

[CONTRIBUTION FROM THE DIVISION OF FOODS, BUREAU OF CHEMISTRY, U. S. DEPARTMENT OF AGRICULTURE. SENT BY H. W. WILEY.]

DETERMINATION OF SALICYLIC ACID IN CANNED TOMATOES, CATSUPS, ETC.

By W. L. DUBOIS.

Received September 6, 1906.

IN THE course of the regular food inspection work of this Bureau we have had frequent occasion from time to time to examine canned tomatoes for salicylic acid. The methods in use for the determination of salicylic acid in this class of goods have been very unsatisfactory, and frequently gave negative results when there