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

was good reason to believe that the preservative was present. To solve this problem and devise a method which could be successfully applied to canned tomatoes, catsups and similar products the work herein described was undertaken.

As is well-known ether extracts from many foods substances which seriously interfere with the color reaction between salicylic acid and ferric salts. In order to meet this difficulty the food must receive some preliminary treatment which removes these interfering extractives, or the salicylic acid must be taken up from the ether extract by a solvent which will not dissolve the other substances present. The solvent most commonly used for this purpose is petroleum ether. Our experience with it, however, has been very unsatisfactory. When salicylic acid is mixed with the gummy, highly colored bodies which are extracted from many foods by ether, gasoline dissolves it with great difficulty, and we have found it impossible to get even good qualitative tests in some cases when we had added a liberal amount of the preservative. The ether extract from tomatoes contains bodies which completely mask the color test with ferric salt. Gasoline proving useless for separating the salicylic acid from them, we have accordingly confined our attention to devising a way of rejecting these substances before extraction with ether.

In all the methods tried 50 grams of pulped tomatoes were weighed as a sample and the salicylic acid added. This was considered a better test than adding the salicylic acid to a large quantity of tomatoes and weighing a sample therefrom, owing to the possible difficulty in sampling by the latter procedure.

Method 1.—Fifty grams of tomatoes were shaken thirty minutes with 150 cc. of water made alkaline with sodium hydroxide. The mixture was centrifuged, the supernatant liquid poured through a filter, and an aliquot portion extracted with ether after acidifying. The residue remaining after evaporating the ether contained considerable coloring-matter and other foreign substances. Method abandoned.

Method 2.—Fifty grams of tomatoes and 100 cc. of water were acidified with phosphoric acid and distilled with steam till 250 cc. had passed over. The distillate was made alkaline, concentrated to 100 cc., acidified and extracted with ether. No coloring-matter or other foreign substances were present in the ether residue, but neither was salicylic acid in any quantity. No test

was obtained with samples containing up to 100 mg. per kilogram. In one having present 200 mg. per kilogram, 0.5 mg. was found, corresponding to 10 mg. in the kilogram.

For such samples, separation of salicylic acid by distillation with steam is not quantitative. A very large volume of distillate is required to carry over any amount of salicylic acid, and that amount is only a small percentage of the preservative present. The method is not to be recommended where a better procedure is available.

Method 3.—Transfer 50 grams of pulped tomatoes to a 200 cc. flask with 50 cc. of water, and make alkaline with milk of lime. Complete to volume, and filter as large an aliquot portion as possible. Usually 150 cc. to 160 cc. of filtrate may be obtained. Acidify with dilute hydrochloric acid and extract with ether four times, using from 75 to 100 cc. of ether at each extraction. Wash the combined ether solution twice with 25 cc. of water, and distil the ether slowly, allowing the last 20 to 25 cc. to evaporate spontaneously. Take up the ether extract in dilute alcohol, make to a definite volume, and match an aliquot portion against a standard solution of salicylic acid, using a few drops of a 2 per cent. solution of ferric alum to produce the color. The results obtained by this method are shown in the table:

Number.	Salicylic acid added. Milligrams.	Salicylic acid Found. Milligrams.	Recovered. Per cent.
A	2.5	0.0	0.0
B	5.0	1.82	36.4
C	10.0	5.0	50.0
D	20.0	12.5	62.4
E	25.0	16.92	67.7
F	30.0	22.70	75.7

In the ether extract from A crystals appearing to be salicylic acid were present. No test for salicylic acid was obtained. This led to an investigation of the effect of alcohol on the color produced by ferric salts and salicylic acid and the following experiment was carried out.

A solution of 1 mg. of salicylic acid in 50 cc. of water to which were added 3 drops of ferric solution was used as a standard. The solutions matched against this contained 1 mg. of salicylic acid and various quantities of alcohol in 50 cc. as shown in the table below.

Alcohol in 50 cc.	Reading of standard.	Reading of solution examined.	Salicylic acid indicated in solution. Milligrams.	Remarks.
5	20	20	1.0	
10	18	18	1.0	
15	18	18	1.0	Quality of color not same.
20	20	21	0.95	Color of sample decidedly bluer.
25	20	27	0.74	Color quality identical.
30	20	37	0.54	" " "
35	18	...		Color of sample too light to read.
40	18	...		" " " " " " "

It appears from these results that the presence of more than a small amount of alcohol in the solution used is inadvisable. We, accordingly, have abandoned its use and are now dissolving the ether extract in warm water, cooling, and making to volume.

It is also an improvement to make the tomatoes alkaline with ammonia before adding the milk of lime. When this is done about 15 cc. milk of lime (200 grams quicklime in 2000 cc. water) are sufficient, whereas much more is necessary when the ammonia is not used. These two modifications in the method given above have solved the problem and give us excellent results as is shown by the following figures:

Salicylic acid used. Milligrams.	Salicylic acid recovered. Milligrams.	Recovered. Per cent.
5	4.7	94.0
10	8.0	80.0
10	8.11	81.1
15	13.33	88.8
20	19.20	96.0
25	25.00	100.0
30	26.70	89.0
50	46.9	93.8

[CONTRIBUTIONS FROM THE BUREAU OF CHEMISTRY, DEPARTMENT OF AGRICULTURE. SENT BY H. W. WILEY.]

A STUDY OF THE METHODS FOR THE DETERMINATION OF ESTERS, ALDEHYDES AND FURFURAL IN WHISKY.

BY L. M. TOLMAN AND T. C. TRESCOT.

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IN THE course of an examination of a large number of whiskies made in the Bureau of Chemistry under the direction of H. W. Wiley the following work on the methods for the determination