

THE ESTIMATION OF FORMALDEHYDE IN MILK.

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FORMALDEHYDE is added to milk as a preservative more than to any other food. Its quantitative determination in this universally used product is, therefore, of the greatest importance.

Formaldehyde distils slowly even from an aqueous solution. When it is added to milk, and has combined to a greater or less extent with the albuminoids present, the rate of distillation is naturally slower still. Should the distillation be continued until four-fifths of the volume taken has been driven over, which is as much as can be distilled conveniently, all of the formaldehyde will not have been recovered. The distillation is, moreover, a troublesome one, owing to the frothing and bumping of the boiling milk. The writer has found that the latter can be largely obviated by using with the ordinary condenser a 500 cc. Kjeldahl nitrogen flask and a round, flat evaporating burner. With this apparatus the distillation proceeds quietly and without difficulty.

In an article by Leonard and Smith¹ results are given showing that the addition of "a few drops of dilute sulphuric acid" largely increased the amount of formaldehyde recovered during the first part of the distillation. In their experiments 100 cc. of milk were used containing 1 part in 100,000 of formaldehyde, and three portions of the distillate of 20 cc. each were collected. Nearly one-third of the formaldehyde present was found in the first 20 cc. of the distillate.

In attempting to make use of this suggestion the writer found that the amount of sulphuric acid present had a great influence on the amount of formaldehyde found in the first 20 cc. of the distillate, as is shown by the following table:

0.3 cc. dilute H_2SO_4 (1 : 3). Per cent.	1 cc. dilute H_2SO_4 (1 : 3). Per cent.	5 cc. dilute H_2SO_4 (1 : 3). Per cent.	20 cc. H_2SO_4 not diluted. Per cent.
24	32.5	18	None

This would tend to show that in obtaining maximum results the quantity of sulphuric acid added is an important factor, and that while the milk should be more than simply acid, an excess of

¹ *Analyst*, **22**, 5 (1897).

acid is to be avoided. In subsequent work, 1 cc. of sulphuric acid of a dilution of 1 : 3 was used with 100 cc. of milk. One-fifth portions, or 20.02 cc. were collected and analyzed, using the potassium cyanide method.¹

The following work was undertaken in an attempt to decide as to whether milk, containing formaldehyde, would yield the same amounts in the first 20 cc. of distillate, after standing some time, as upon immediate distillation, and also as to whether different samples of milk containing various amounts of formaldehyde would give it up in the first 20 cc. of the distillate in direct proportion to the amount they originally contained.

Four samples of fresh milk were treated with formaldehyde so that they contained 1 part in 5,000, 1 part in 10,000, 1 part in 25,000, and 1 part in 50,000. One hundred cc. portions of each were distilled immediately, others at the end of twenty-four and forty-eight hours. The latter in the intervening time were kept in a cool place. The following percentages of the total formaldehyde added were obtained in the first 20 cc. of the distillates.

	1 part in 5,000. Per cent.	1 part in 10,000. Per cent.	1 part in 25,000. Per cent.	1 part in 50,000. Per cent.	Averages. Per cent.
Determined immediately..	31.50	33.90	36.10	41.00	35.62
After 24 hours	30.60	32.00	32.80	37.50	33.22
After 48 hours	30.75	31.50	35.00	36.00	33.31

From the average column on the right, it is seen that the average results on milk that has stood a short time after the addition of the formaldehyde are very close to $33\frac{1}{3}$ per cent. The next column shows that the samples of milk containing the least formaldehyde give the highest percentages of the total amount present. The table shows, furthermore, that if milk containing formaldehyde is kept cool, it may stand one or two days at least without altering the amount obtained on analysis. A trial proved that when milk containing formaldehyde is subjected to a warm and varying temperature, the amount of formaldehyde recovered by distillation in the first 20 cc. decreases so that at the end of forty-eight hours only 18 or 20 per cent. will be found, instead of approximately 33.

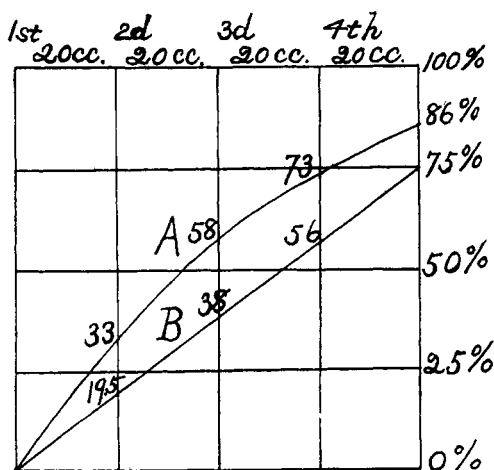
The following chart, the data for which were compiled from a number of analyses, gives the curves showing the rapidity of the

¹ See the preceding paper, p. 1032.

distillation of formaldehyde from milk containing 1 part in 10,000. The ordinates represent the per cent. of the total formaldehyde present, and the abscissas the amount of distillates.

The line A is the curve obtained from milk after the addition of 1 cc. of the dilute acid, while B is the one obtained from milk without the addition of acid.

In summarizing, it may be said that considerable time and trouble may be saved by using a Kjeldahl flask and a round, flat



evaporating burner in the distillation of milk; that the quantity of sulphuric acid added has a decided effect upon the amount of formaldehyde obtained in the first part of the distillation; that if the treated milk is kept in a cool place, the percentage of formaldehyde found will remain practically constant for at least forty-eight hours; and that where 100 cc. of milk are treated with 1 cc. of sulphuric acid of a dilution of 1 : 3 and distilled, the first 20 cc. of the distillate will contain very close to $33\frac{1}{3}$ per cent. of the total formaldehyde present.

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