

sample. In all cases duplicates represent separate extractions.

SAMPLE	Per cent moisture	Per cent ash	Per cent indigotin
Purified synthetic indigo.....			99.71
Awa indigo No. 1.....	9.71	27.75	100.59
Awa indigo No. 2.....	9.41	27.41	6.78
Awa indigo No. 3.....	9.18	27.28	6.86
Awa indigo No. 4.....	9.30	27.17	5.05
Awa indigo No. 5.....	10.08	30.56	5.15
Awa indigo No. 6.....	10.10	30.47	4.23
Awa indigo No. 7.....	12.44	32.39	4.32
Awa indigo No. 8.....	12.44	32.48	1.94
Indigo by Nagai process.....	9.24	33.89	2.02
	9.21	33.77	1.26
	11.56	34.50	1.34
	11.56	34.55	2.46
	9.92	34.34	2.47
	9.95	34.32	3.17
	9.46	35.26	3.23
	9.38	35.08	3.59
	3.18	33.25	3.72
	3.28	33.25	31.45
			31.53

STATE COLLEGE OF WASHINGTON
PULLMAN, WASHINGTON

EXPERIMENTAL DATA COMPARING THE DELICACY OF DIFFERENT TESTS FOR HYDROGEN PEROXIDE IN MILK

By IRWIN T. DARLINGTON

Received April 12, 1915

From references on the detection of hydrogen peroxide, the reagents listed in Table I were found to be in most general use and appear to give the most delicate tests for the detection of hydrogen peroxide in milk.

A stock solution of hydrogen peroxide was made from a commercial sample containing 3.2 per cent of

TABLE I

REAGENT	SOLUTION	Color in presence of H ₂ O ₂
Starch	Dilute (water) + KI	Bluish green
Paraphenylene diamine	2 per cent (water)	Blue
Benzidine	4 per cent (alcoholic) + 10 cc. milk (+ 2 or 3 drops acetic acid)	Blue
Titanic acid	2 per cent solution titanium hydrate in dilute H ₂ SO ₄	Yellow
Vanadic acid	2 per cent in dilute H ₂ SO ₄	Faint orange-red

hydrogen peroxide by analysis with potassium permanganate. This stock solution was made so that each cc. contained 0.01 g. of hydrogen peroxide.

The milk to which the hydrogen peroxide was added was received from the city inspectors and was proven free from hydrogen peroxide by each of the above reagents.

Hydrogen peroxide was added to each 100 cc. of milk in separate bottles to the amounts indicated in Table II, and tests made on 10 cc. of each sample

TABLE II

Grams per 100 cc.	Results of tests made immediately after samples were made up												After standing 18 hrs. in a warm room
	15.0	9.0	3.0	1.5	0.75	0.6	0.3	0.15	0.03	0.015	0.006	0.003	
Starch.....	++	++	++	++	++	++	++	++	++	++	++	++	++
Paraphenylene.....	++	++	++	++	++	++	++	++	++	++	++	++	++
Benzidine.....	++	++	++	++	++	++	++	++	++	++	++	++	++
Titanic acid.....	++	++	++	++	++	++	++	++	++	++	++	++	++
Vanadic acid.....	++	++	++	++	++	++	++	++	++	++	++	++	++
	15.0	9.0	3.0	1.5	0.75	0.6	0.3	0.15	0.03	0.015	0.006	0.003	15.0
	9.0	3.0	1.5	0.75	0.6	0.3	0.15	0.03	0.015	0.006	0.003	0.00105	9.0
	3.0	1.5	0.75	0.6	0.3	0.15	0.03	0.015	0.006	0.003	0.00105	0.00075	3.0
	1.5	0.75	0.6	0.3	0.15	0.03	0.015	0.006	0.003	0.00105	0.00075	0.00037	1.5

with each of the above reagents. Tests were made immediately after the samples were made up and again after standing 18 hours in a warm room.

It will be seen from Table II that paraphenylene diamine and benzidine proved to give the most delicate tests for hydrogen peroxide in raw milk. The limits

of these two reagents proved to be 0.00075 g. of hydrogen peroxide in 100 cc. of raw milk. No experiments were made with milk that had been previously heated to destroy bacterial action.

Practically all the hydrogen peroxide was reduced after standing 18 hours in a warm room. This confirmed previous results obtained in this laboratory when it was found that after 18 hours' standing at room temperature no reaction could be obtained from the addition of 1 g. of magnesium peroxide to 100 cc. of raw milk. Positive tests were given only when the milk contained 9 per cent and 15 per cent of hydrogen peroxide.

The samples containing 9 per cent and 15 per cent of hydrogen peroxide failed to give a positive test with any of the above reagents, except paraphenylene diamine, which gave a delicate positive test with 15 per cent of hydrogen peroxide after standing in a warm room for 24 hours. Tests on distillates of milk treated with peroxide were negative.

A few tests were made with magnesium dioxide added as a preservative. The experiments made with this material were insufficient to afford any positive conclusions, but it appears that magnesium dioxide does not lend itself to as delicate tests as does hydrogen peroxide.

From the foregoing it is seen that paraphenylene diamine is the test of the reagents tested for the detection of small quantities of hydrogen peroxide in raw milk, and that it is probable that peroxide in amounts that would be added to milk as a preservative cannot be detected after 18 hours by these reagents.

CHEMICAL LABORATORY, DEPARTMENT OF HEALTH
CITY OF NEW YORK

THE OFFICIAL METHOD FOR DETERMINING CRUDE FIBER AS APPLIED TO COTTONSEED MEAL

By C. K. FRANCIS

Received April 7, 1915

The well known Weende method¹ for determining crude fiber has been subjected to criticism by many chemists. The chief trouble seems to be with the filtering materials, linen, asbestos, or glasswool, which vary in their physical characteristics and, necessarily, in filtering efficiency. In a number of laboratories it has been the practice to use linen cloth for filtering. The cloth has been used in the same manner as filter paper, with and without suction, in plain and in ribbed funnels. It has been difficult to duplicate linen of a selected pick, so that the fineness of the filter has been liable to vary from time to time. However, the method seemed to be satisfactory when working with such substances as corn, but when one endeavored to filter the alkaline mixture of certain seeds like flaxseed and cottonseed it became necessary to devise some scheme to hasten the process.

A novel arrangement for filtering the mixture through cloth by upward suction has been suggested by Pickel.² The cloth stretched over a funnel or thistle-tube furnishes a flat surface and the liquid passes rapidly,

¹ "Official Methods of Analysis," U. S. Bureau of Chemistry, *Bull.* **107** (Rev.), 65.

² *THIS JOURNAL*, **2** (1910), 280; see MacNider, *Ibid.*, 281.