a crystal of silver nitrate from a powder containing no free tartaric acid. From a powder containing less than 0.5 per cent. of free tartaric acid, however, an extract giving a pronounced test was obtained.

The so-called Wolff test[1] for tartaric acid, either free or combined, in which 5 grams of the sample are shaken with 250 cc. of water for some time, filtered, the filtrate evaporated to dryness, a few drops of 1 per cent. resorcinol solution and 3 cc. strong sulphuric acid added and the mixture heated slowly with the development of a supposedly characteristic rose-red color when tartaric acid is present, proved to be very unsatisfactory in the writer's hands. Practically every usual adulterant of cream of tartar or baking-powder containing such, gave a similar color, or one easily confused with it, by the above test, when absolutely *no tartaric acid, either free or combined, was present.*

The writer has always found the simple method of shaking a little of the sample with 10 per cent. ammonia water, filtering, adding a crystal of silver nitrate and warming (with the appearance of a silver mirror if free or combined tartaric acid be present) very satisfactory. No cream of tartar adulterant or substitute or any other usual ingredient of baking-powders gave the least trace of a mirror by this test; and from the standpoint of time required it is, of course, much quicker than any test involving the evaporation of a large volume of water, as does the so-called Wolff test. The use of this simple test upon a portion of the sample for tartaric acid generally and the similar testing of the alcoholic extract from another portion for free tartaric acid leaves nothing to be desired along the line of qualitative tartaric acid tests in baking-powder analysis.

STATE COLLEGE,
CENTRE CO., PA.

THE DETERMINATION OF WATER IN SUBSTANCES THAT ARE TO BE AFTERWARDS EXTRACTED WITH VOLATILE SOLVENTS.

BY R. M. BIRD.

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DURING the course of an investigation it became necessary to determine accurately the water and fat in a large number of

[1] *Rev. chim. anal. appl.*, **4**. 263 (1899).

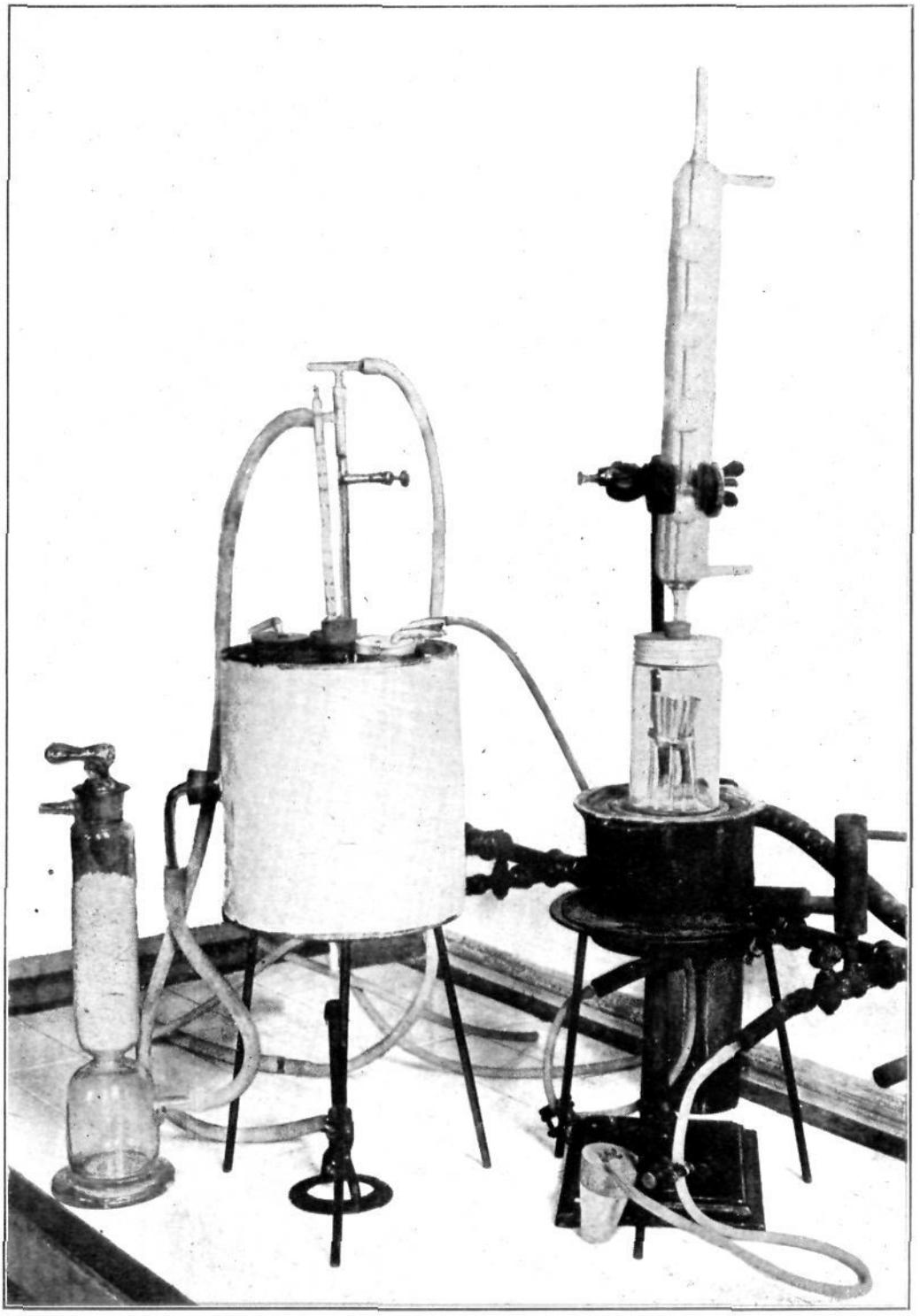

Fig. 1.

samples of butter, and a method was sought which would do away with the transferring of the butter from the drying dish to the extraction apparatus and the subsequent operation with the ether extract. The method herein described proved to be so convenient and expeditious and the apparatus so easily made that it is suggested to those who have much work of this kind to do. It makes use of a rapid current of hot, dry air, which removes the water very quickly.

Apparatus.—A hot-air bath, arranged to determine five samples at a time, is shown in Fig. 1 and in cross-section in Fig. 2. It is made from a gallon "friction top" can such as is used for "wood filler," syrups, etc. The flanges for the stoppers are soldered on, as shown in Fig. 2. The supports, *d d*, hold up a wire gauze shelf, *S*, which is about an eighth of an inch below the bottom of the crucibles, and catches them should they fall off the stopper while being removed from the bath. A rubber band is placed around the top *T*, between *a* and *b* and makes an air-tight joint. Tube *A* is of glass, and a fine-mesh wire gauze, *W*, rests on the bottom of the bath around its end. Only rubber stoppers are used and the system is air-tight except for the inlet and outlet tubes. The temperature of the bath is maintained at 98°-100° C., and dry air is drawn in through tube *A* and out through the crucible and tube *B* by means of a suction-pump or aspirating bottle. A wide-mouthed bottle, with six tubes through its stopper, will permit the draft for five determinations to be generated by a single filter-pump. The thermostat is not necessary, as the temperature can be easily regulated with a screw pinch-cock on the burner tubing.

Determination of Butter.—A platinum or porcelain Gooch crucible is filled about two-thirds full with loosely packed asbestos fiber,[1] on top of which is placed a Witt plate. It is dried in the apparatus for about five minutes and counterbalanced on the balance. One and a half to two grams of butter are delivered from a pipette on to the Witt plate,[2] the crucible is placed in the bath, as shown in Figs. 1 and 2, and a rapid current of air drawn through it for twenty to thirty minutes. The loss in weight represents the water. Our practice is to pass air through a

[1] The asbestos is previously extracted with ether and dried.

[2] The butter will usually solidify on the plate if it is about the consistency of thick cream when drawn from the sample—the best condition for accurate work.

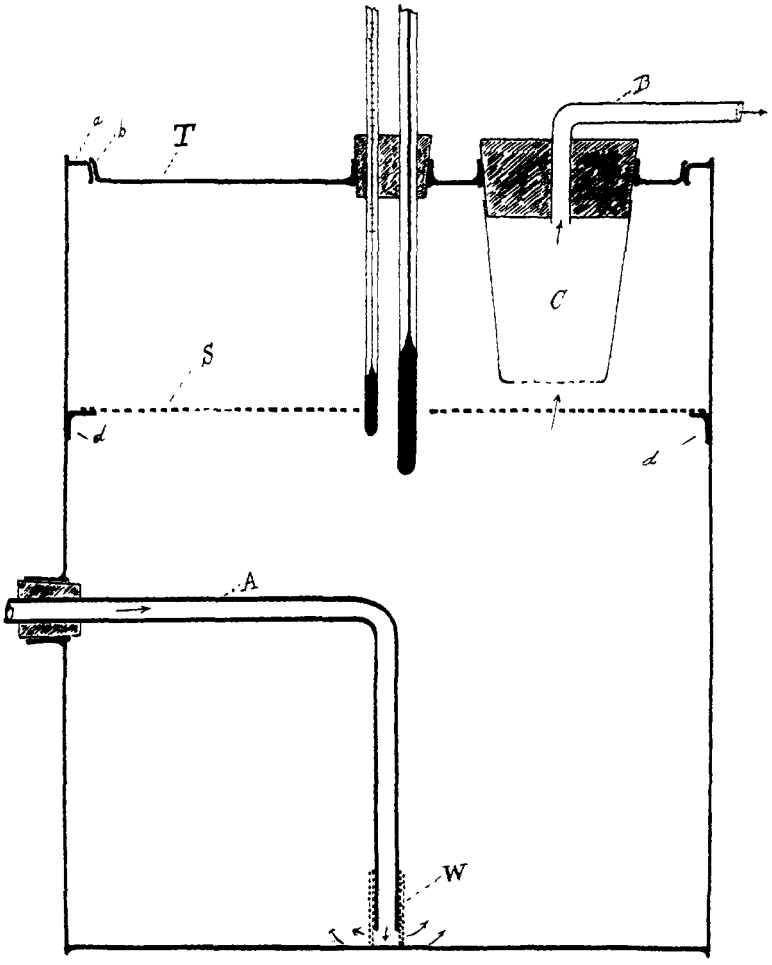

Fig. 2

second time for five or ten minutes and reweigh, although there is rarely any material change after drying twenty minutes.

The crucible is now placed in the extraction apparatus, shown on the right of Fig. 1, and extracted with ether until its weight is constant after drying five to ten minutes in the hot-draft bath; the loss represents the fat. If the ether is boiled so as to flow from the condenser in a stream the extraction usually takes from thirty to fifty minutes.

A convenient extraction apparatus is made from a glass sample jar with a metal screw top. A hole is made in the top for a cork, through which passes the condenser tube. The crucible is supported on a glass tripod. A jar and condenser arranged for a mercury seal is rather more economical with ether, but hardly more convenient. The ether may be recovered from the extract.

Discussion.—As will be seen, the method is a convenient and rapid one—counterbalancing the prepared crucible, taking weight with butter in it, passing current of air for twenty to thirty minutes, taking weight, extracting for about forty-five minutes, drying for five minutes, taking weight. It is more convenient to determine five samples at once and weigh by method of differences against a constant counterbalance.

As the hot, dry air comes in contact with the solid butter it absorbs the water from the thin film formed on the surface almost immediately, and the capillary action of the fiber draws away the butter as fast as melted.

The accuracy of the method is equal to that of the official method, as will be seen by reference to Table I, and the saving in time is very great. The results agree entirely with those obtained by the official method. Unusual care was taken in making the determinations recorded here, especially in drawing butter from the sample, which was generally made to the consistency of thick cream so as to avoid the least difference of water in different parts while taking amounts for check analyses. The percentages of water, shown in Table I, are based upon the first reweighing which showed a minus change of less than one-tenth of one per cent.

TABLE I.—WATER IN BUTTER.

		A. O. A. C. official method.				Current of dry, hot air.		
		Time			Mean.			Mean.
No.	Kind.	Hrs.	Min.	Per cent.	Per cent.	Time. Min.	Per cent.	Per cent.
3 to 10	Creamery[1]	3		14.68[2]		32	15.30	
			30	15.74[3]		20	15.04	
		1		15.26[3]		20	14.98	
		1	19	15.35[4]				15.11
		1	19	15.75[4]				
					15.36			

[1] Additional butter was worked into sample.
[2] Dried in flat-bottomed dish, surface 72 sq. cm., nothing in dish.
[3] " " " " " 43 " sand " "
[4] " " " " " 43 " asbestos " "

TABLE I.—(*Continued*).

No.	Kind.	A. O. A. C. official method. Time. Hrs.	Min.	Per cent.	Mean. Per cent.	Current of dry, hot air. Time. Min.	Per cent.	Mean. Per cent.
11 to 16*d*	"	4	30	12.67[1]		21[2]	12.54	
		4		12.79[1]		21	12.22	
					12.73			12.38
17 to 20*e*	"	4		13.20[3]		20	13.30	
		4		13.58[3]		20	13.90	
					13.39			13.60
23 to 28*f*	"	2		8.44[3]		15	8.43	
		2		8.54[4]		15	8.45	
					8.49			8.44
29 to 30*g*	Butterine	2	05	8.74	8.74	30	8.81	8.81
31 to 32*h*	Poor country	2	05	13.07[4]	13.07	30	13.03	13.03
33 to 34*i*	Rancid creamery	2	05	14.28[4]	14.28	30	14.62	14.62
35 to 36*k*	Renovated butter	2	05	14.11[4]	14.11	30	13.02	13.02
37 to 38*m*	Creamery[5]	2	05	15.63[4]	15.63	30	15.85	15.85

TABLE II.—CHANGES IN WEIGHT OF BUTTER WHEN DRIED IN DIFFERENT WAYS.

No.	Kind.	Quantity taken. Grams.	Periods of heating. Hrs.	Min.	Changes in weight of sample. Per cent.	Water. Per cent.	Method.
1*a*	Creamery	1.7396		30	— 16.458	16.57	Current of
				15	— 0.115		hot, dry air.
				15	+ 0.040		
				15	— 0.057		
2*b*	"	1.7637		10	— 12.406		
				5	— 0.334	12.74	"
				5	+ 0.017		
				5	— 0.005		
3*c*	"	1.4076		32	— 15.295	15.30	
				15	+ 0.007		
				17	+ 0.007		"
				15	— 0.014		

[1] Dried in flat-bottomed dish, surface 72 sq. cm., nothing in dish.
[2] Water was worked out of sample.
[3] Dreid in flat-bottomed dish, surface 43 " sand " "
[4] " " " " " " 43 " nothing " "
[5] Additional butter was worked into sample.

TABLE II.—(*Continued*).

No.	Kind.	Quantity taken. Grams.	Periods of heating. Hrs.	Periods of heating. Min.	Changes in weight of sample. Per cent.	Water. Per cent.	Method.
4*c*	Creamery	1.5055		20	— 15.038	15.04	Current of
			2		+ 0.106		hot, dry air.
5*c*	"	1.4162		20	— 14.977	14.98	
				12	— 0.007		"
6*c*	"	2.4790	3		— 14.68	14.68	Official[1]
			1	30	+ 0.11		
7*c*	"	1.9130		30	— 15.735		" [2]
				30	— 0.052	15.79	
			1		— 0.010		
			2		+ 0.110		
8*c*	"	1.4790		30	— 15.145		"
				30	— 0.115	15.26	
			1		+ 0.027		
			2		— 0.054		
9*c*	"	1.5283		54	— 15.298	15.35	" [3]
				25	— 0.059		
				30	— 0.072		
				41	— 0.033		
			1		+ 0.131		
10*c*	"	1.6077		54	— 15.668	15.75	"
				25	— 0.086		
				30	— 0.044		
				41	— 0.044		
			1		+ 0.056		
11*d*	"	1.6494		21	— 12.411	12.54	Current of
				30	— 0.133		hot, dry air.
				30	— 0.013		
12*d*	"	1.7863		21	— 12.215	12.22	"
				30	— 0.056		
				30	+ 0.006		
13*d*	"	2.0015	2		— 6.124	6.12	Official[3]
			1		+ 0.005		
14*d*	"	1.6650	2		— 7.928	7.93	"
			1		+ 0.012		
15*d*	"	2.8050	4	30	— 12.67	12.67	" [1]
			1	30	+ 0.114		
16*d*	"	2.6610	4		— 12.79	12.79	"
			1	30	+ 0.071		

[1] Flat-bottomed dish 72 cm. surface, nothing in dish.
[2] " " 43 " " sand " "
[3] " " 43 " " asbestos " "

TABLE II.—(*Continued*).

No.	Kind.	Quantity taken. Grams.	Periods of heating. Hrs.	Min.	Changes in weight of sample. Per cent.	Water. Per cent.	Method.
17*e*	Creamery	1.6312		20	— 13.297	13.30	Current of hot, dry air.
				4	— 0.018		
				30	— 0.036		
18*e*	"	1.5982		20	— 13.898	13.90	"
				4	— 0.063		
				30	— 0.081		
19*e*	"	2.3920	4	30	— 13.20	13.20	Official[1]
			1	30	+ 0.156		
20*e*	"	2.4397	4		— 13.58	13.58	"
			1	30	+ 0.156		
21*e*	"	2.2678	2		— 9.185		" [2]
			1	30	— 0.031	9.22	
22*e*	"	1.8733	2		— 8.552		"
			1		— 0.060	8.61	
23*f*	"	1.6126		15	— 8.427	8.43	Current of hot, dry air.
				45	— 0.081		
24*f*	"	1.6116		15	— 8.445	8.45	"
				45	— 0.074		
25*f*	"	1.4983	2		— 8.436	8.44	Official[2]
26*f*	"	2.2453	2		— 8.538	8.54	"
27*f*	"	2.7117	4		— 8.013	8.01	" [1]
			1	30	+ 0.151		
28*f*	"	2.8730	3	30	— 8.447	8.45	
			1	30	+ 0.160		
29*g*	Butterine	1.9292		30	— 8.807	8.81	Current of hot, dry air.
				20	— 0.040		
				10	+ 0.010		
30*g*	"	2.2773	2	5	— 8.739	8.74	Official.[3]
			1		— 0.022		
				30	+ 0.026		
31*h*	Poor country butter.	1.5079		30	— 13.025	13.03	Current of hot, dry air.
				20	— 0.080		
				10	— 0.020		
32*h*	"	1.7538	2	5	— 13.073	13.07	Official.[3]
			1		— 0.070		
				30	+ 0.040		

[1] Flat-bottomed dish, 72 cm. surface, nothing in dish.
[2] " " 43 " " asbestos " "
43 " " nothing " "

TABLE II.—(*Continued.*)

No.	Kind.	Quantity taken. Grams.	Periods of heating. Hrs.	Periods of heating. Min.	Changes in weight of sample. Per cent.	Water. Per cent.	Method.
33*i*	Rancid butter.	1.3670		30	— 14.623	14.62	Current of
				20	— 0.073		hot, dry air.
				10	— 0.036		
34*i*	"	1.5790	2	5	— 14.281	14.28	Official[1].
			1		— 0.000		
35*k*	Renovated butter.	1.5081		30	— 13.017	13.02	Current of
				20	— 0.053		hot, dry air.
				10	— 0.007		
36*k*	"	1.6949	2	5	— 14.113	14.11	Official.[1]
			1		— 0.041		
				30	+ 0.018		
37*m*	Creamery.	1.4704		30	— 15.853	15.85	Current of
				20	— 0.000		hot, dry air.
38*m*	"	1.5613	2	5	— 15.634	15.63	Official[1].
			1		— 0.039		
				30	+ 0.039		

Table II shows, in detail, the changes in weight during varying periods of heating, for both the official method and the method here described. As has been often noticed, there are apparently three changes taking place when butter is being dried (loss of water, loss of other volatile constituents and oxidation which causes an increase in weight) and a sample loses weight rapidly at first, then slowly and finally shows a small increase. In drying in a bath heated by boiling water these changes are somewhat more marked than is the case when dried by the hot air current. The reason why the weight soon comes to a practical standstill in the method proposed is probably because the drying takes place at a temperature too low to volatilize much else besides water or to cause much oxidation. Sample 4*c* (Table II) changed, after twenty minutes, only one-tenth of one per cent. on passing the air through it for two hours; this was a net gain. While it at first seems strange, yet the temperature in the crucible at the surface of the asbestos never rose above 70° C., and rarely above 60° C., although the bath temperature remained at 98°-100° C., the bulb of the bath thermometer being as shown in Fig. 2.

[1] Flat-bottomed dish, 43 cm. surface, nothing in dish.

The results, by the official method, of 13 and 14*d*, and 21 and 22*e* are very singular and inexplicable to us. These four (*d* and *e*, each in duplicate) were weighed out at one sitting and dried in a bath, heated by boiling water, which was in action constantly from morning till night for days before and after this particular lot was dried (alone). The weights came to a standstill and the percentages in one case agreed closely, yet the figures are something like half the true values, They are given here merely as a matter of record, although we were unable to find a cause for the queer conduct.

To show the time required for extraction, four determinations of fat are given in Table III.

TABLE III.—EXTRACTION OF FAT.

No.	Quantity taken. Grams.	Periods of extraction. Minutes.	Change in weight of sample. Per cent.	Fat. Per cent.
47	1.6494	30	—84.92	
		20	— 0.05	
		15	— 0.02	84.99
48	1.6116	20	—88.18	
		30	— 0.61	
		15	— 0.00	88.79
49	1.6312	45	—84.02	
		15	— 0.05	84.07
50	1.4076	45	—82.23	
		15	— 0.04	82.27

The method is applicable to various other substances, the proper gas being aspirated through the apparatus, and the pressure reduced if this is desirable. With finely ground material, a layer of asbestos should cover the bottom of the crucible and a plug of cotton placed in the inner end of the outlet tube, to insure against loss.

UNIVERSITY OF MISSOURI,
COLUMBIA, MO.

[CONTRIBUTION FROM THE FOOD LABORATORY OF THE BUREAU OF CHEMISTRY, U. S. DEPARTMENT OF AGRICULTURE, NO. 1.
SENT BY H. W. WILEY.]

A COMPARISON OF THE HALOGEN ABSORPTION OF OILS BY THE HÜBL, WIJS, HANUS, AND McILHINEY METHODS.

BY L. M. TOLMAN.

Received May 26, 1904.

THE Hübl method for determining the iodine absorption of oils and fats is the official method, but it has several faults which