

A Study of the Methods of Determining Boron Compounds in Food and Drugs.

By A. SCOTT DODD, B.Sc., Ph.D., F.I.C.

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PART II. EXPERIMENTAL: EFFECT OF FATS AND OTHER ORGANIC SUBSTANCES ON THE DETERMINATION.

PRACTICAL experience has shown that substances containing large percentages of fat cannot be ignited, even in presence of excess of alkali, without considerable loss of boron compounds resulting. Special methods, such as that of Richmond and Harrison (ANALYST, 1902, **27**, 179) have been devised to get over the difficulty, but their application is limited to substances like butter, margarine, or egg melanges, which can readily be transformed into a homogeneous fluid condition. Substances such as ham, sausages, cakes and fruits, however, apparently cause considerable difficulty, as none of these can be rendered homogeneous by means of ordinary solvents. Since the presence of fat or oil is the chief source of trouble, the following series of experiments was tried with a view to discovering if, under any condition, the fat can readily be removed without any loss of boric acid.

SOLUBILITY OF BORIC ACID IN ORGANIC SOLVENTS AT 60° F.—Weighed quantities of pure dry boric acid were stirred for 5 minutes in a dry beaker with 20 c.c. of methylated ether, petroleum spirit, benzene, chloroform and carbon tetrachloride, respectively, the temperature being maintained at 60° F. In a second series of experiments 5 c.c. of pure olive oil were added in each case. The contents of the beaker were then filtered through a Swedish filter paper into a dry beaker, and 10 c.c. of each filtrate were evaporated to dryness in a porcelain basin (care being taken to keep the solvent below its boiling point during the evaporation), the residue was taken up with 10 to 15 c.c. of distilled water, and

the solution titrated after the addition of 0.5 gm. of mannitol and phenolphthalein. (In each case the solution was tested and found to be neutral before the mannitol was added.)

The remaining portion of the filtrates was tested qualitatively with turmeric paper for the presence of boric acid.

In the case of the filtrates containing oil, 10 c.c. were extracted with 5 c.c. of 0.5 *N* sodium hydroxide solution and about 30 c.c. of water. The extracts (about 50 c.c. in volume) were acidified, boiled for 5 minutes, cooled, neutralised with sodium hydroxide solution (with Sofnol Indicator No. 1), and finally titrated after the addition of 0.5 gm. of mannitol and six drops of phenolphthalein solution.

TABLE I.

Solvent 20 c.c.	Quantity of boric acid taken. Grms.	Final titration.		Reaction.
		0.1 <i>N</i> NaOH c.c.	= H_3BO_3 (actual). Grm.	
Ether (dry)	0.051	0.64	0.0040	Very distinct
"Petroleum" spirit	0.052	0.40	0.0025	"Practically" nil
Chloroform	0.051	<0.01	—	Distinct trace
Benzene	0.052	0.01	0.0006	Distinct trace
Benzene + trace of alcohol	0.049	<0.01	—	Practically nil
Carbon tetrachloride	0.050	0.01	—	Distinct trace
15 c.c. ether + 5 c.c. oil	0.051	<0.01	—	Practically nil
15 c.c. ether + 5 c.c. oil	0.051	<0.01	—	Practically nil
Petroleum spirit + 5 c.c. oil	0.052	0.75	0.0047	Very distinct
Chloroform + 5 c.c. oil	0.051	1.00	0.0062	Very distinct
Benzene + 5 c.c. oil	0.053	0.07	0.0043	Very distinct
Carbon tetrachloride + 5 c.c. oil	0.050	<0.01	—	Practically nil
	0.052	<0.01	—	Practically nil
	0.051	0.28	0.0017	Very distinct

The above results show that solvents, such as petroleum spirit, benzene and carbon tetrachloride, which do not readily mix with water, dissolve practically no boric acid. Ether and chloroform, on the other hand, dissolve a small quantity at 60° F. In the above experiments the solubility, except with ether, was found to be less than 1 part in 10,000 of solvent, but the proportion in all cases probably depends upon the amount of moisture the solvent has taken up during the course of the test. The presence of a minute trace of alcohol mixed with benzene was found to render boric acid more soluble than in the pure dry benzene.

The solubility of boric acid in mixtures of oil and benzene or petroleum spirit at 60° F. is practically negligible. Mixtures of pure olive oil and methylated ether, chloroform or carbon tetrachloride, on the other hand, dissolve small, but quite appreciable, quantities of boric acid. It is, however, quite possible that different oils and fats might behave differently from olive oil, but this may readily be verified by subsequent tests.

In view of the fact that some products are more or less acid, and that some of their boric acid content may be lost if they are dried without previous neutralising with caustic soda, another series of experiments was tried, in which about 0.5 gm. of boric acid and 10 c.c. of *N* sodium hydroxide solution were evaporated to dryness before stirring up with the solvents. The results are shown below:

TABLE II.
SOLUBILITY OF SODIUM BORATE IN ORGANIC SOLVENTS AT 60° F.

Oil or fat.	Solvent.	Boric acid reaction of filtrate.
Olive oil	Ether	Distinct
" "	Petroleum spirit	Practically nil
" "	Benzene	Practically nil
" "	Chloroform	Distinct
" "	Carbon tetrachloride	Distinct trace
Cocoa butter	Ether	Distinct
" "	Petroleum spirit	Practically nil
" "	Benzene	Practically nil
" "	Chloroform	Distinct
" "	Carbon tetrachloride	Distinct trace

As shown by the foregoing experimental results, benzene and petroleum spirit can be used to eliminate fats and oils from vegetable products by merely washing out at room temperature and rejecting. The quantity of boric acid dissolved thereby is practically negligible if the product is first dried, either alone or after being rendered slightly alkaline with caustic soda. This has an important practical bearing, as much time might thus be saved. Perfect dryness, however, is essential, and it is safer to determine the quantity of boric acid in the extract as shown below.

The following experiment was tried to ascertain whether, instead of rejecting the filtrate containing the excess of fat, the quantity of boric acid could be directly titrated and allowed for :

TABLE III.

Fat or oil.	Solvent.	Boric acid added. Grm.	Titration.	
			0.1 N NaOH c.c.	= H ₃ BO ₃ . Grm.
Olive oil	Ether	0.0195	3.14	0.0195
" "	Petroleum spirit	0.0195	3.14	0.0195
" "	Benzene	0.0195	3.14	0.0195
" "	Chloroform	0.0195	3.14	0.0195
" "	Carbon tetrachloride	0.0195	3.14	0.0195

As shown in Table III, the presence of oil, together with methylated ether, petroleum spirit, benzene, chloroform or carbon tetrachloride, does not affect the accuracy with which boric acid solutions can be titrated. In these experiments a known quantity (3 c.c.) of boric acid solution was titrated direct, with the use of 0.5 gm. of mannitol and phenolphthalein. Similar quantities were placed in a porcelain basin, together with 5 c.c. of olive oil and 15 c.c. of the above organic solvents, in turn. About 30 c.c. of water were added, and the contents of the basin were heated for 5 minutes on a water-bath, with constant stirring, and then cooled. The contents were then titrated with 0.1 N sodium hydroxide solution, after the addition of 0.5 gm. of mannitol and phenolphthalein, and the results were recorded as shown in the Table.

These results are of importance in so far as they show how the usual lengthy determination of boric acid in products containing a large percentage of fat may be

shortened considerably. Now that it is known that the determination of boric acid may be made direct on the fat-solvent portion without first extracting with alkali, the product under examination can be dried, washed with a solvent into a titrating basin, and the extracted boric acid determined and added to the result obtained in the main determination.

One would, however, require to select a solvent and carry out the extraction of the oil or fat under such conditions as would prevent the extraction of any phosphates along with the boric acid. A further investigation would, therefore, require to be made to ascertain under what conditions the phosphates would be excluded.

EFFECT OF CONCENTRATION OF FAT ON LOSS OF BORIC ACID WHEN IGNITED WITH EXCESS OF ALKALI.—Three different quantities of boric acid solution containing 0.0124 grm., 0.0186 grm., and 0.0310 grm. of boric acid, respectively, were placed in a platinum basin together with 3 c.c. of *N*-sodium hydroxide solution. The contents of the basin were evaporated to dryness on a water-bath, and different weights of olive oil were added. The oil, borate and excess of alkali were then heated gradually, ignited and burned off at a dull red temperature. The ash was boiled with water containing a slight excess of *N*-sulphuric acid, filtered, and washed into a flat titrating basin. The contents of the basin were boiled for 5 minutes to ensure the complete expulsion of carbonic anhydride and then cooled. One drop of Sofnol Indicator No. 1 was added, and 0.1 *N* sodium hydroxide solution was carefully run in from a burette until the liquid was just neutral. The burette was now filled up and carefully adjusted for the final titration of the boric acid. Then 0.1 grm. of mannitol and about 6 to 8 drops of phenolphthalein solution were added, and the contents were titrated.

In order to determine more accurately the loss due to the oil the following experiment was tried:—Three platinum basins, each containing 0.0186 grm. of boric acid and 3 c.c. of *N*-sodium hydroxide solution, were heated on a water-bath until complete evaporation of the contents had taken place. Two were then heated alone, one at a dull red heat, and the other at a high temperature. The former was found to contain 0.0183 grm. of boric acid, and the latter 0.0174 grm. That is, the ignition of 0.0186 grm. of boric acid in the presence of excess of alkali caused a loss of 0.0003 grm. of boric acid at a dull red heat, and of 0.0012 grm. of boric acid at a high temperature.

The contents of the third platinum basin were ignited with 10 grm. of stearic acid and treated as in the experiments with the olive oil. The ash, when finally titrated, was found to contain 0.0178 grm. of boric acid.

This experiment showed that if one took into account the difficulty of igniting a highly carbonaceous substance like stearic acid, the loss due to the presence of the stearic acid was very similar to that of the borate when ignited alone. In other words, the presence of a fatty acid causes little loss of boric acid when ignited with excess of alkali.

The following table shows the losses of boric acid which occurred on igniting olive oil with borate and excess of alkali:

TABLE IV.
EFFECT OF CONCENTRATION OF FAT ON LOSS OF BORIC ACID WHEN
IGNITED WITH EXCESS OF ALKALI.

Quantity of fat taken. Grms.	Quantity of boric acid added.		Final titration.	
	Grms.	Percentage in fat.	0.1 N NaOH c.c.	= H_3BO_3 Grm.
1	0.0124	1.24	1.60	0.0099
1	0.0186	1.86	2.22	0.0138
1	0.0310	3.10	4.42	0.0274
5	0.0124	0.248	0.63	0.0039
5	0.0186	0.372	0.94	0.0058
5	0.0310	0.620	1.50	0.0093
10	0.0124	0.124	0.45	0.0028
10	0.0186	0.186	0.72	0.0045
10	0.0310	0.310	1.40	0.0087
15	0.0124	0.083	0.65	0.0040
15	0.0186	0.124	0.63	0.0039
15	0.0310	0.207	0.68	0.0042
15	0.0310	0.207	1.30	0.0081
20	0.0124	0.062	0.50	0.0031
20	0.0186	0.093	0.75	0.0046
20	0.0310	0.155	1.40	0.0087

In each test 3 c.c. of N sodium hydroxide were used for fixing the boric acid during ignition.

These results show that when 1 gram. of oil was used the loss, though considerable, was distinctly irregular. Greater regularity was observed when 5 grms. and upwards of oil were used. This is shown by the following tabulated results, which were calculated from the figures found in Table IV.:

TABLE V.

H_3BO_3 present. Per Cent.	H_3BO_3 found. Per Cent.	H_3BO_3 lost. Per Cent.	Percentage of total H_3BO_3 lost.
0.062	0.016	0.046	74.2
0.083	0.026	0.057	69.4
0.083	0.027	0.056	68.7
0.093	0.023	0.070	75.3
0.124	0.028	0.096	77.4
0.124	0.028	0.096	77.4
0.155	0.044	0.111	71.6
0.186	0.045	0.141	75.7
0.207	0.054	0.153	73.9
0.248	0.085	0.163	69.4
0.310	0.087	0.223	71.9
0.372	0.116	0.256	68.8
0.620	0.186	0.434	70.0

It will be observed that the percentage of boric acid lost under the above conditions is very large, and, although not very constant, yet possesses a degree of constancy which suggests that if the conditions of ignition were more uniform a fairly constant percentage loss would result.

The losses shown in Table V fluctuate between 68.7 and 77.4 per cent., and have a mean value of 72.6 per cent.

In view of the fact that a very small percentage only of boric acid is lost when ignited, either with alkali alone or with alkali and a fatty acid, it is evident that

the loss must be due to the glyceride present when an oil is used. The apparent constancy of loss appears to suggest that, when excess of oil and caustic alkali is present, a complex consisting of a sodium salt, borate and glycerol is formed in course of ignition, and that this complex splits up into sodium borate and glyceroborate. This contains about 70 per cent. of the original borate, and is volatilised in the course of ignition, leaving sodium borate.

In order to ascertain whether the amount of alkali in excess of that required to neutralise the boric acid was an important factor, different quantities of caustic soda were added to the same quantities of boric acid and olive oil prior to ignition. It was found, however, that the results obtained were so similar as to admit of the conclusion being drawn that, so long as the sodium hydroxide was in excess, the amount was immaterial.

It is also quite evident from the above experiments that, so long as there is sufficient oil or fat present to provide the necessary glycerol for the boric acid present, the quantity of oil or fat is also immaterial.

VARIATION IN THE LOSS OF BORIC ACID CAUSED BY IGNITING DIFFERENT OILS IN PRESENCE OF BORIC ACID AND EXCESS OF ALKALI.—Similar weights (5 grms.) of different fats and oils were ignited with known weights of boric acid and excess of sodium hydroxide at a dull red heat. The ash was digested with water and 6 drops of concentrated hydrochloric acid, and filtered into a titrating basin. The filter paper and contents were returned to the platinum basin and fully ignited, and then washed into the basin. The contents of the basin were boiled for 5 minutes, with constant stirring, to expel the carbonic anhydride, cooled, neutralised, and then titrated after the addition of 0.5 gm. of mannitol. The results obtained are given in the following Table:

TABLE VI.

Kind of oil 5 grms.	N-NaOH added. c.c.	Loss of H_3BO_3 . Per Cent.	Final titration.		
			Boric acid H_3BO_3 added. Grm.	0.1 N NaOH. c.c.	$=\text{H}_3\text{BO}_3$. Grm.
Coconut butter	3.0	73.0	0.0189	0.83	0.0051
" "	3.0	73.5	0.0189	0.81	0.0050
Olive oil	3.0	69.0	0.0186	0.94	0.0058
" "	3.0	70.0	0.0186	0.90	0.0056
Almond oil	3.0	58.6	0.0189	1.25	0.0078
" "	3.0	58.2	0.0189	1.28	0.0079
Cacao butter	3.0	61.4	0.0189	1.18	0.0073
" "	3.0	60.3	0.0189	1.21	0.0075
Linseed oil	3.0	56.4	0.0186	1.30	0.0081
" "	3.0	46.4	0.0186	1.30	0.0081
Castor oil	3.0	56.1	0.0189	1.34	0.0083
" "	3.0	54.5	0.0189	1.38	0.0086
Sesame oil	3.0	54.0	0.0199	1.40	0.0087
" "	3.0	54.5	0.0189	1.38	0.0086
Rape oil	3.0	50.8	0.0189	1.50	0.0093
" "	3.0	50.3	0.0189	1.51	0.0094
Cottonseed oil	3.0	49.2	0.0186	1.65	0.0102
" "	3.0	44.0	0.0186	1.70	0.0105
" "	3.0	51.1	0.0186	1.47	0.0091

From these results it will be observed that different kinds of fat or oil give rise to variations in the percentage of boric acid volatilised and lost during the process of ignition. The proportion of loss is fairly constant for the same kind of oil, and appears to have some bearing on the constitution of the oil. As already mentioned, the glycerol content of an oil plays an important part in rendering boric acid volatile, so that there appears to be some possibility of utilising the ignition of boric acid in presence of excess of alkali as a means of identifying a glyceride (fat or oil), and also of determining the amount of glycerol contained in compounds or mixtures. The above results were obtained under ordinary laboratory conditions, but, so far as they go, they afford reason for the belief that, if special precautions were taken, which would keep the conditions constant and exclude any disturbing factors, characteristic results would be found for individual oils and fats.

EFFECT OF THE PRESENCE OF ORGANIC MATTER ON THE LOSS OF BORIC ACID WHEN IGNITED WITH EXCESS OF ALKALI.—Twenty grms. each of different organic substances which were free from boric acid, were ignited at a dull red heat in a platinum basin with a known quantity of boric acid and excess of alkali (3 c.c. of 0.1 N H_3BO_3 + 3 c.c. of N-NaOH). The boric acid was then determined in the usual way after lixiviating and eliminating carbonic anhydride and phosphates, if present. When the substance (*e.g.* sugar) contained no phosphates, the ash was boiled with water and a slight excess of hydrochloric acid until most of the carbonic anhydride was expelled, and the solution was then transferred to a 100 c.c. flask, made up to the mark, shaken and filtered. Fifty c.c. of the filtrate were placed in a porcelain titrating basin and boiled for 5 minutes, with constant stirring, to ensure the complete expulsion of carbonic anhydride. After rapid cooling, the contents were neutralised with 0.1 N sodium hydroxide solution, Sofnol Indicator No. 1 being used. The final titration was effected after adding 0.5 gm. of mannitol and phenolphthalein solution. For convenience, these results are shown in the following Table doubled to equal the entire quantity.

The starch was found to contain a small percentage of phosphates, which rendered the above shortened method inapplicable. The boric acid in the ashes obtained from mixtures containing starch was therefore determined by the usual long method.

The following Table, No. VII (see p. 722), shows the results obtained.

These results indicate that substances of the nature of carbohydrates and fatty acids do not have any considerable effect on the loss of boric acid, when ignited in presence of excess of alkali. It will be observed that a slight loss does take place, but it is insignificant when compared with that produced by the ignition of an oil or fat, and is, in reality, not much more than is caused by the ignition of boric acid and excess of alkali alone. There are certainly slight differences with different substances, but these appear to be mainly due to the variation in the inflammability of these substances. The loss of boric acid in these cases would therefore be mechanical rather than chemical, as in the case of substances containing glycerol.

TABLE VII.

EFFECT OF THE PRESENCE OF ORGANIC MATTER ON THE LOSS OF BORIC ACID WHEN
IGNITED WITH EXCESS OF ALKALI.

Substance, 20 Grms.	Boric acid taken.		Final titration.	
	Grm.	Percentage of substance.	0.1 N NaOH.	= H ₃ BO ₃
			c.c.	Grm.
Olive oil	0.0186	0.093	0.76	0.0047
Sugar	0.0186	0.093	2.98	0.0185
Starch	0.0186	0.093	2.96	0.0184
Stearic acid	0.0186	0.093	2.84	0.0176
19 sugar and 1 oil	0.0186	0.093	2.96	0.0184
18 sugar and 2 oil	0.0186	0.093	2.40	0.0149
15 sugar and 5 oil	0.0186	0.093	1.68	0.0104
			1.80	0.0112
19 starch and 1 oil	0.0186	0.093	2.90	0.0180
18 starch and 2 oil	0.0186	0.093	2.38	0.0148
			2.46	0.0153
15 starch and 5 oil	0.0186	0.093	2.38	0.0148
19 starch and sugar and 1 oil	0.0186	0.093	2.94	0.0182
18.8 starch and sugar and 1.2 oil	0.0186	0.093	2.78	0.0172
18.6 starch and sugar and 1.4 oil	0.0186	0.093	2.30	0.0143
18.4 starch and sugar and 1.6 oil	0.0186	0.093	2.00	0.0124
18.2 starch and sugar and 1.8 oil	0.0186	0.093	2.04	0.0126
18 starch and sugar and 2 oil	0.0186	0.093	1.88	0.0117

A comparison of the results shown in Table VII with those in Table IV shows that the presence of carbohydrates with fat tends to diminish the loss of boric acid which would have been caused by the presence of the same amount of fat by itself. Thus, for example, 1 grm. of olive oil ignited with 0.0186 grm. of boric acid and 3 c.c. of *N* sodium hydroxide causes an actual loss of 0.0048 grm. of boric acid, whilst 1 grm. of olive oil, together with 19 grms. of sugar, ignited with 0.0186 grm. of boric acid and 3 c.c. of *N* sodium hydroxide solution causes an actual loss of merely 0.0002 grm. of boric acid. Also, 5 grms. of olive oil under the above conditions causes an actual loss of 0.0128 grm. of boric acid, whereas 5 grms. of olive oil and 15 grms. of sugar causes a loss of 0.0069 grm. of boric acid; and 5 grms. of olive oil and 15 grms. of starch causes a loss of only 0.004 grm. of boric acid. The difference in these last two results is probably due to the dry nature of the starch affording a better protecting medium between the oil and the sodium borate than sugar, as a mixture of oil and sugar allows a quantity of the oil to come into direct contact with the sodium borate, thereby causing greater loss when the mass is ignited. In some instances the experiments were repeated several times before concordant results were obtained. This difficulty appeared to be caused by the variations in the degree of contact between the oil and the sodium borate.

The conditions of the foregoing experiments are to a large extent artificial, and the oil and the boric acid are by no means as homogeneously mixed with the starch, etc., as they would be in natural vegetable substances. The protective properties of the non-fatty constituents would, therefore, tend to be more variable. So far as the above results go, it would appear that comparatively little loss of boric acid occurred during direct ignition if the percentage of oil or fat in the

sample did not exceed 5 per cent. If that were absolutely correct for all substances, then Thomson's process could be curtailed by directly igniting in presence of excess of alkali any substance which was known to contain less than 5 per cent. of oil.

The following table shows some results obtained by the use of natural vegetable substances in place of artificial mixtures of starch, sugar and oil:

TABLE VIII.

EFFECT OF THE PERCENTAGE OF FAT OR OIL IN VEGETABLE PRODUCTS ON THE LOSS OF BORIC ACID ON IGNITION.

Article, 20 grms.	Containing in 20 grms.		Boric acid added. Grm.	Final Titration.	
	H ₃ BO ₃ .	Oil.		For ½ quantity. c.c.	= H ₃ BO ₃ . Grm.
	Grm.	Grm.			
Dairy meal ..	0.0001	1.30	0.0188	1.45	0.0090
" " ..	0.0001	1.30	0.0310	2.43	0.0151
Soya meal ..	0.0005	0.14	0.0188	1.46	0.0091
" " ..	0.0005	0.14	0.0310	2.41	0.0149
Dried grains ..	Nil	1.43	0.0188	1.48	0.0092
" " ..	—	1.43	0.0310	2.43	0.0151
Kardi seed cake ..	0.0003	1.73	0.0189	1.48	0.0092
" " ..	0.0003	1.73	0.0313	2.48	0.0154
Calf meal ..	0.0003	1.69	0.0310	2.45	0.0152

The loss of boric acid is shown in the following table:

TABLE VIIIa.

Article, 20 grms.	Oil. Per Cent.	Loss of boric acid H_3BO_3 .	
		Percentage of total boric acid.	
		Grm.	
Dairy meal ..	6.50	0.0009	4.75
" " ..	6.50	0.0009	2.90
Soya meal ..	0.70	0.0011	5.70
" " ..	0.70	0.0017	5.08
Dried grains ..	7.15	0.0004	2.13
" " ..	7.15	0.0008	2.58
Kardi seed cake ..	8.65	0.0008	4.16
" " ..	8.65	0.0008	2.53
Calf meal ..	8.45	0.0009	2.88

From the above results it will be observed that a slight loss of boric acid has taken place in each determination. It is apparent, however, that the loss bears no relationship to the percentage of oil or fat present in the sample, and is, in fact, greater in the case of the extracted soya meal, which contained little oil, than in any of the other samples. The comparatively low results with this sample may be attributed to losses other than those caused by ignition, as the large percentage of phosphates and other mineral matter rendered the separation of the boric acid extremely difficult. The other actual quantities of boric acid lost are more constant, and are comparable with the loss due to the ignition of fat-free organic matter and boric acid together with excess of alkali. It would appear, therefore, that there is a tendency towards slightly low results. The deficiency may amount to about 0.0008 gm. of boric acid (H_3BO_3), but should be considerably less in the case of substances which do not contain high percentages of phosphates and other mineral constituents.

As already mentioned, the loss of boric acid, due to the oil in a sample, depends upon the degree of contact between the oil and the boric acid. The samples used in this experiment did not appear oily, and, apparently, the contact of the oil and the boric acid is very slight, as even 8.65 per cent. of oil causes no definite loss. In all probability the percentage of oil in some natural vegetable products could be increased still further without causing any appreciable loss of boric acid. As, however, the constitution of some vegetable products may give rise to greater contact between the oil and boric acid present, it would be inadvisable to take too high a percentage of oil as the general margin of safety.

Taking everything into consideration, it would appear that any vegetable substances which contains less than 8 per cent. of oil, may be safely ignited directly without previous extraction of the oil, and that no appreciable loss of boric acid will result therefrom.

By taking 8 per cent. of oil as a general margin of safety, and omitting the preliminary extraction of oil in all samples containing 8 per cent. of oil and under, considerable time and trouble will be saved. It is, however, imperative to mix such samples with a solution of alkali, and to dry them thoroughly at water-bath temperature before attempting to ignite them, as the presence of moisture causes a loss of boric acid if an attempt is made to ignite a moist sample.

LOSS OF BORIC ACID RESULTING FROM BOILING ACIDIFIED BORIC ACID SOLUTIONS.—In keeping with the practice of boiling 50 c.c. of solution to eliminate carbonic anhydride, 50 c.c. of liquid containing sulphuric acid and a known weight of boric acid were boiled for various lengths of time. One series was carried out in a beaker flask covered with a watch glass; the other series was carried out by boiling the solution in an open shallow titrating basin, with constant stirring. The solutions were cooled, neutralised with 0.1 *N* sodium hydroxide solution, with Sofnol Indicator No. 1, and finally titrated after adding 0.5 gm. of mannitol and phenolphthalein. The volume to which the original solution was evaporated during the boiling was ascertained by pouring into a burette and measuring the contents of the vessel after titrating, and deducting the known volume of the solutions added during the neutralising and titrating.

TABLE IX.

Boric acid taken. Grm.	Time of boiling. Minutes.	Final volume. c.c.	Final titration.	
			0.1 N NaOH. c.c.	= H ₃ BO ₃ . Grm.
<i>Open Basin.</i>				
0.0329	5	24.7	5.30	0.0329
0.0329	10	12.2	5.20	0.0322
0.0329	15	2.0	4.58	0.0284
0.0329 to dryness	—	—	4.28	0.0266
<i>Beaker Flask and Watch Glass.</i>				
0.0329	5	47.3	5.30	0.0329
0.0329	10	43.5	5.30	0.0329
0.0329	15	33.1	5.30	0.0329

N.B.—In each of these tests the volume of the original solution was 50 c.c., and the acidity equal to 5 c.c. of 0.1 *N* H_2SO_4 .

From the above results it will be observed that a fair amount of liberty can with safety be taken in the process of expelling carbon dioxide from acidified dilute boric acid solutions. The boiling was found to be done most expeditiously in an open basin; 5 minutes, or even less with constant stirring, expels the carbon dioxide completely. The only likely causes of loss arise from too rapid boiling, which may cause spurting or over-drying at the edges, but this is easily remedied if constant attention is given. It was found that an appreciable loss occurred only when the solution was concentrated to about one-fifth of its original bulk, but such a condition is unlikely to occur in practice.

(To be concluded.)
