reputable firm in whose currant jelly we reported salicylic acid but which was present in no greater quantity than we have since found it in the fresh currants. A similar experience was lately had in one of the state laboratories for food control.

In addition to the above work we are studying the distribution of benzoic acid in fruits and vegetables, and hope to be able to publish our results within the year.

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IODINE ABSORPTION OF OILS AND FATS.

A COMPARISON OF METHODS.

BY L. M. TOLMAN AND L. S. MUNSON.

THE Hübl method has been used almost entirely for this determination but it has several faults. First, the solution rapidly loses in strength so that it will change materially during a determination, and after standing a week or so becomes too weak for use. Second, it is so slow in its reaction with some of the oils, such as linseed, that a very serious error is brought about by the change in the strength of the solution during the time of reacting.

Wijs[1] showed how considerable this error might be, and the following table taken from his work shows how much difference the time of titrating the blank makes in the iodine number.

TABLE I.--IODINE NUMBERS OF LINSEED OIL BY HÜBL METHOD.

Time of absorption. Hours.	Blank titrated at beginning.	Blank titrated at end.
2	173.74	. . . .
7	177.65	170.39
24	181.89	163.16

These figures show a decrease in the iodine number after seven hours, if the blank is titrated at the end of the determination. Wijs considers that the true iodine number in this case lies between 173.7 and 181.89. This variation is a very serious objection

[1] Chem. Rev. Fett. u. Hartz. Ind., 6, 6 (1899).

to the Hübl method, and, on such oils as have a high number, greatly reduces the value of the determination and probably accounts for the wide range of figures obtained by different analysts.

A solution that would remain permanent would completely remove this source of error.   Two new solutions have been proposed as substitutes for the Hübl, and it is claimed for both that they change but little with age, and are also more rapid in their action than the Hübl solution, yet give results agreeing very closely with those obtained by the Hübl method.

The Wijs[1] solution is iodine chloride dissolved in glacial acetic acid.   The Hanus[2] solution is iodine bromide dissolved in glacial acetic acid.   Wijs[3] claims that a solution, made up with acetic acid (99 per cent.) showing no reduction with bichromate and sulphuric acid and containing a very slight excess of iodine in order to prevent the presence of any iodine trichloride, remained practically constant for eighteen months.   Twenty-five cc. which were neutralized by 47.3 cc. N/10 thiosulphate at the beginning, at the end required 46.9 cc. N/10 thiosulphate.   Lewkowitsch[4] found that after five months the strength of such a solution was practically unchanged.   Our experience has been the same.   A solution remained practically without change for a month.

Hanus[5] showed that a solution of iodine bromide in glacial acetic acid changes only very slightly in three months, and our experience has corroborated this claim.   The Hanus solution was prepared as suggested by Hunt;[6]   13.2 grams iodine were dissolved in 1000 cc. glacial acetic acid (99.5 per cent.), which gave no reduction with bichromate and sulphuric acid, and then enough bromine was added to double the halogen content of the solution. This takes approximately 3 cc. of bromine.   The iodine solution should be cold when the bromine is added.   Forty cc. of a solution prepared as directed above contained 1.0300 grams of iodine: a month later it contained 1.02791 grams.   This difference might easily be due to the difference in temperature at the time the 40 cc. were measured.   In order to get strictly accurate results a

[1] *Ber. d. chem. Ges.*, **31**, 750 (1898).
[2] *Ztschr. Nahr. u. Genus.*, **4**, 913 (1901).
[3] *Ibid.*, **5**, 499 (1902).
[4] *Analyst*, **24**, 257 (1899).
[5] *Ztschr. Nahr. u. Genus.*, **4**, 916 (1901).
[6] *J. Soc. Chem. Ind.*, **21**, 454 (1902).

number of 20 cc. portions were measured into stoppered flasks and titrated from day to day. The following table shows that the solution is constant:

TABLE II.

Time of standing. Days.	N/10 thiosulphate = 20 cc. iodine. Cc.
1	45.50
2	45.50
3	45.55
5	45.50
6	45.55
8	45.60

This is enough to show that either of the solutions is satisfactory and very much better than the Hübl in retaining its strength.

The second objection to the Hübl solution is slowness of reaction. The time recommended to be allowed in order to complete the reaction is variously given by different authors from two to twenty-four hours. Allen[1] recommends two hours. The official method of the Association of Official Agricultural Chemists requires three hours. Lewkowitsch[2] recommends four hours. Wijs[3] showed that with linseed oil the absorption was not complete in seven hours and perhaps longer. These different methods will give varying results. Wijs recommends, when his solution is used, fifteen minutes for non-drying fats and oils, thirty minutes for semidrying oils, and one hour for drying oils.

The following table showing results obtained with both Hanus' and Wijs' solutions substantiates these claims.

TABLE III.—TIME NECESSARY FOR COMPLETE ABSORPTION OF IODINE.

Lab. No.	Kind of oil.	Iodine numbers by Hanus' method.			Iodine numbers by Wijs' method.		
		15 min.	30 min.	1 hr.	15 min.	30 min.	1 hr.
773	Cocoanut	8.7	8.6	....	....	....	....
22,077	Konut......... ...	6.4	6.4	....	....	....	....
1,170	Butter	35.2	35.4	....	....	....	....
1,168	"	35.4	35.5	....	35.8	35.9	36.0
	Oleo oil	43.2	43.4	....	....	....	....
4	Oleomargarine .	52.4	51.8	....	....	....	....
5	" ·	51.9	52.1	....	52.9	....	53.0
9	" ·	64.6	65.1	....	....	....	....
1	" ·	52.2	52.4	....	....	....	....

1 "Commercial Organic Analysis," Vol. II, Part 1, p. 64.
2 "Chemical Analysis of Oils, Fats and Waxes," 1898, p. 173.
3 *Chem. Rev. Fett. u. Hartz. Ind.*, 6, 7 (1899).

Lab. No.	Kind of oil.	Iodine numbers by Hanus' method.			Iodine numbers by Wijs' method.		
		15 min.	30 min.	1 hr.	15 min.	30 min.	1 hr.
	Oleomargarine .	80.4	80.5	....	....	....	....
23,606	Lard oil	70.0	69.7	....	....	....	....
798	Olive oil.......	90.1	90.4	90.3	90.0	91.2	90.9
960	" "	82.1	81.7	82.2	82.6	82.6	82.5
833	" "	86.5	86.5	86.5	86.5	86.7	87.2
772	Peanut	96.0	97.4	97.7	98.8	99.0	99.0
777	Mustard	125.0	126.0	126.0	126.5	126.5	128.0
771	"	....	119.4	120.0	....	118.0	118.2
776	"	....	105.0	105.0	....	104.3	104.6
775	Rape..........	....	107.4	107.5	....	105.8	105.7
770	Corn	....	116.8	117.6	117.1	116.1	118.5
774	Poppy.........	136.8	137.4	138.4	138.8	138.9	139.1
1,162	Linseed	....	186.3	186.2	183.8	190.2	188.7

For butter, lard, oleomargarine, and olive oil, the reaction is practically complete in fifteen minutes. With cottonseed, sesame, and mustard, thirty minutes are necessary, while with poppy and linseed one hour gives the best results. There seems to be practically no difference between the two solutions in respect to speed of action. Tables IV and V give the results obtained by ourselves on a number of oils by all three methods. Table VI is a compilation of the data available on the three methods.

TABLE IV.—IODINE NUMBERS OF OILS AND FATS.

Lab. No.	Oils.	Hübl's number, 3 hrs.	Wijs's number, 30 min.	Hanus's number, 30 min.	Difference between Wijs and Hübl.	Difference between Hanus and Hübl.
	Non-drying.					
	Cocoanut......	8.93	9.05	8.60	+ 0.12	— 0.33
	Konut	6.09	6.43	6.40	+ 0.34	+ 0.31[1]
1,170	Butter	35.3	36.2	35.3	+ 0.90	+ 0.00
1,168	Butter	34.8	35.9	35.4	+ 1.10	+ 0.60
	Oleo oil	42.6	43.5	43.3	+ 0.90	+ 0.70
1	Oleomargarine	53.6	53.5	52.3	— 0.10	— 1.30
4	"	52.8	53.7	52.2	+ 0.90	— 0.60
5	"	52.5	52.9	52.0	+ 0.40	— 0.50[1]
9	"	66.3	66.0	64.8	— 0.30	— 1.50
23,606	Lard oil......	69.3	70.5	69.8	+ 1.20	+ 0.50
487	" "	73.7	74.5	73.9	+ 0.70	+ 0.20[1]
1,181	Magnolia oil ..	81.7	79.4	78.9	— 2.30	— 2.80
1,182	" " ..	76.1	75.6	74.0	— 0.50	— 2.10
772	Peanut " ..	96.3	99.0	97.4	+ 3.00	+ 1.10
1,149	" " ..	94.5	95.2	94.1	+ 0 70	— 0.10
492	" " ..	107.7	109.5	107.7	+ 1.80	+ 0.00[2]

[1] Commercial oils.
[2] Adulterated with cottonseed.

Lab. No.	Oils.	Hübl's number, 3 hrs.	Wijs's number, 30 min.	Hanus's number, 30 min.	Difference between Wijs and Hübl.	Difference between Hanus and Hübl.
770	Mustard oil ..	110.4	118.5	115.5	+ 8.10	+ 5.10
771	" " ..	113.0	118.2	116.8	+ 5.20	+ 3.80
776	" " ..	98.4	104.3	103.8	+ 5.90	+ 5.40
486	" " ..	103.5	112.5	110.2	+ 9.00	+ 6.70[1]
495	" " ..	106.4	117.3	114.8	+10.90	+ 8.40[1]
775	Rape oil	101.3	105.7	105.2	- 4.40	+ 3.80
490	" "	100.2	104.1	102.8	+ 3.90	+ 2.60[1]
	Semi-drying and drying.					
	Sunflower.....	106.4	109.2	107.2	+ 2.80	+ 0.80
1,159	Cottonseed oil .	103.8	105.3	105.2	+ 1.50	+ 1.40
1,160	" " .	106.2	107.3	107.8	+ 1.10	- 1.60
1,161	" " .	104.8	106.2	106.7	+ 1.40	+ 1.90
489	Sesame oil	106.4	107.0	106.5	+ 0.60	+ 0.10[1]
444	Corn "	119.0	123.2	120.2	+ 4.20	+ 1.20[1]
491	" "	119.0	122.2	119.6	+ 3.00	+ 0.40[1]
777	" "	123.3	129.2	126.0	+ 5.80	- 2.70
493	Poppy "	133.4	135.2	132.9	+ 1.80	- 0.50[1]
774	" "	134.9	139.1	138.4	+ 4.20	+ 3.50
1,162	Linseed "	169.8	186.5	184.5	+16.70	+14.70[1]
1,188	" " ..	179.5[2]	188.7	183.7	- 9.20	+ 4.20

It will be seen from these results that for oils and fats with an iodine number under 100 there is little difference between the three methods. For practical purposes they are the same. But this is not so true for the oils with higher iodine numbers. With mustard and rape oil there is a marked difference, 8.4 numbers higher with Hanus's and 10.9 numbers higher with Wijs's method. It is with these oils and linseed oil that there is the widest variation, but there can be little doubt that the higher numbers obtained by the Hanus and Wijs methods are the more correct.

TABLE V.—IODINE NUMBERS OF OLIVE OILS.

Lab. No.	Hübl's number, 3 hrs.	Wijs's number, 30 min.	Hanus's number, 30 min.	Difference between Wijs and Hübl.	Difference between Hanus and Hübl.
795	89.7	90.9	90.4	+1.2	+0.7
796	89.7	90.6	90.0	+0.9	+0.3
797	89.8	91.4	90.0	-1.6	+0.2
798	89.7	91.1	90.4	+1.4	+0.7
960	80.9	82.5	81.7	+1.6	+0.8
833	84.8	86.7	86.5	+1.9	+1.7

[1] Commercial oils.
[2] Four hours for Hübl determination.

Lab. No.	Hübl's number, 3 hrs.	Wijs's number, 30 min.	Hanus's number, 30 min.	Difference between Wijs and Hübl.	Difference between Hanus and Hübl.
831	85.2	86.7	85.9	+1.5	+0.7
832	84.9	86.5	85.9	+1.6	+1.0
834	84.5	86.1	85.1	+1.6	+0.6
936	81.8	82.8	82.8	+1.0	+1.0
933	80.6	81.6	81.1	+1.0	+0.5
835	82.7	83.1	82.6	+0.4	−0.1
962	81.2	81.5	80.9	+0.3	−0.3
955	82.6	83.6	83.1	+1.0	+0.5
952	81.3	82.2	81.8	+0.9	+0.5
840	86.3	87.8	86.7	+1.5	+0.4
935	80.5	80.9	81.6	+0.4	+1.1
953	79.2	79.9	80.6	+0.7	+1.4
958	81.8	83.1	81.8	+1.3	0.0
959	81.4	83.4	81.9	+2.0	+0.5
954	86.1	87.7	80.3	+1.6	+0.2
934	81.1	82.4	82.3	+1.3	+1.1
956	84.5	85.9	85.6	+1.4	+1.1
932	80.5	82.0	81.4	+1.5	+0.9
961	80.8	82.3	81.1	+1.5	+0.3
836	86.0	87.7	86.6	+1.7	+0.6
931.	80.7	82.2	81.5	+1.5	+0.8
838	86.1	87.9	87.1	+1.8	+1.0
839	89.0	91.3	89.9	+2.3	+0.9
837	84.0	...	84.9	...	+0.9
673	83.3	84.1	82.6	+0.8	−0.7
841	86.9	88.0	87.3	+1.1	+0.4
842	87.2	88.1	87.1	+0.9	+0.1
843	85.1	86.6	85.6	+1.5	+0.5
844	84.2	85.0	84.2	+0.8	0.0
1,091	81.9	83.4	82.0	+1.5	+1.10
Average, 36 samples,				+1.2	+0.59

The iodine numbers of the thirty-six samples of olive oils of known purity obtained by the three methods are given in this table. The maximum difference between Wijs and Hübl is 2, the Wijs method always giving higher results. On the thirty-six samples the Wijs method gave an average of 1.2 numbers higher.

The Hanus method gave a maximum of 1.4 numbers higher than Hübl, with an average of 0.59 number higher. This gives the Hanus solution a very slight advantage over the Wijs and this difference seems to hold good on all the oils—the Hanus being slightly nearer the Hübl than is the Wijs.

TABLE VI.—IODINE NUMBERS BY DIFFERENT METHODS.

Variety of oil.	Hübl's number.	Wijs's number.	Hanus's number.	Difference between Hübl and Wijs.	Difference between Hübl and Hanus.	Analyst.
Cocoanut..	9.03	....	9.03	....	0.0	Hanus.
Butter	30.7	....	30.6	....	--0.1	"
Lard	56.4	....	56.9	....	--0.5	"
Peanut	88.3	....	88.4	....	+0.1	"
"	87.2	87.2	...	0.0	....	Wijs.
"	91.8	93.4	91.6	--1.6	--0.2	Hunt.[2]
Rape......	99.3	...	98.8	....	0.5	Hanus.
"	102.9	103.3	....	--0.4	....	Wijs.[1]
"	103.0	102.1	101.9	0.9	--1.1	Hunt.[2]
Sunflower ·	117.8	119.0	....	+1.2	....	Wijs.[1]
Sesame ...	110.3	111.7	....	--1.7	....	"
" ...	107.1	....	107.5	....	+0.4	Hanus.
Corn......	124.8	128.4	....	--3.6	....	Wijs.[1]
Poppy	122.4	....	122.6	....	--0.2	Hanus.
"	134.6	....	135.2	....	--0.6	"
"	119.6	119.6	....	0.0	....	Wijs.[1]
Linseed ...	174.8	177.3	174.5	--2.5	0.3	Hunt.[2]
" ...	180.9	182.1	....	--1.2	....	Wijs.[1]
" ...	170.2	....	171.0	....	0.8	Hanus.

These figures show practically the same results as the previous tables although not quite so wide a variation, which may be somewhat accounted for by the fact that four hours were used in the Hübl determinations, which gives a little higher figure than the three hours on the higher-absorbing oils. These results show very little choice between the two proposed substitutes for the Hübl method. The Hanus gives results a little closer to the Hübl figures but the difference is of practically no consequence. Its chief advantage over the Wijs is in the ease of preparation. Iodine trichloride is a rather rare reagent and the preparation of the iodine chloride by passing chlorine gas into the acetic acid solution of iodine until the halogen content is doubled is a somewhat tedious operation compared with the addition of bromine. On this account we favor the Hanus solution.

With this change of method, a new set of figures will have to be obtained for the semidrying and drying oils, and such oils as mustard and rape with high iodine absorption. For the butters, lard and olive oil the Hanus method compares satisfactorily with the Hübl. The stability of the solution does not lessen the need

[1] Seven minutes used in determination.
[2] One hour used in determination.

of blanks for each set of determinations.   The high coefficient of expansion of glacial acetic acid, 0.00115 for 1° C., makes an appreciable error if a slight change of temperature takes place, as will be seen from the following table.

TABLE VII.

Temperature. °C.	40 cc. iodine monobromide in N/10 thiosulphate.
16.0	92.05
17.5	91.85
18.5	91.80
21.5	91.35
24.5	91.10
27.0	90.80
11°	1.25 cc.
change	decrease.

A change of ± 1° C. gives a change of ∓ 0.11 cc. in the amount of N/10 thiosulphate necessary to neutralize 40 cc. of the iodine solution, so that a blank titration of the iodine solution could easily vary 1 cc. N/10 thiosulphate in a day, which would completely vitiate the results.

DOES CHOLESTEROL OCCUR IN MAIZE OIL?

BY AUGUSTUS H. GILL AND CHARLES G. TUFTS.

ACCORDING to Hoppe-Seyler[1] and to Hopkins,[2] cholesterol occurs in maize oil.   As there is evidence of the occurrence of this substance in no other vegetable oil, except olive oil, this is interesting, and of possible importance as a means of detecting maize oil in mixtures with other oils.   The statement of Hoppe-Seyler was made before this group of bodies was clearly differentiated, however, and the melting-point ascribed by Hopkins to the alcohol found by him in maize oil is not the true melting-point of cholesterol.   The difference in the melting-points is shown below. Cholesterol, 146°-147° ;[3] "cholesterol" from maize oil, 137°-137.5°.[3] Hopkins used no other means of identification except the color

[1] *Bull. Soc. Chim* [2], **6**, 342 (1866); *Medicin.-chem. Untersuch.*, 1, 162.

[2] This Journal, **20**, 948 (1898).

[3] Wislicenus and Moldenhauer : *Ann. Chem.* (Liebig), **146**, 179 (m. p. 147°); Reinitzer: *Monatsh. Chem.*, **9**, 422 (m p. 147.5°) ; Hesse : *Ann. Chem.* (Liebig), **192**, 177 (1878) (m. p. 145°-146°); Salkowski : *Ztschr. anal. Chem.*, **26**, 567 (m. p. 146°); Bömer: *Ztschr. Untersuch. Nahr. u. Genus.* (1898), p. 81 (average m. p. of fifty-two samples, 146.4°-147.3°).