large influence on the biological quality of the oil meal produced.

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THE ESTIMATION OF WATER IN SALAD OIL AND DETERMINATION OF ITS SOLUBILITY AT ORDINARY TEMPERATURES*

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Method for Moisture Determination:

The common methods of drying in a vacuum oven, or heating fat or oil in an aluminum dish on a hot plate to the smoking point are not particularly suited to the accurate determination of small amounts of moisture in salad oils, or even in other fats. The objections to these methods do not require comment here. It seemed that a distinct need existed for a method which would permit the determination of small amounts of water in oils preferably by weighing the water evolved after absorption in some suitable medium. A simple apparatus was devised in which 10 to 50 gram samples of the fat or oil were dried in a current of inert gas at elevated temperatures, the moisture being subsequently absorbed in a neutral medium such as fused calcium chloride. In Figure I the details of the apparatus are illustrated.

Ten to fifty grams of sample, depending on the moisture content are weighed into dry evolution tubes "T" (1 by 8 in. Pyrex). These are attached to the blowing system by means of a tight fitting rubber stopper carrying inlet and outlet tubes. Hydrogen from a cylinder is passed through the fused calcium chloride in drying tube "D" and thence through the flow control capillary "F" and then through the oil. The effluent hydrogen is passed through absorbent cotton in a small bulb "G" to remove any entrained oil. Moisture is absorbed in U-tube "A" with "B" serving as an auxillary to indicate that absorption is complete. "C" merely serves as a guard tube to the train. Fused calcium chloride was used as the absorbent although any other absorbent should be satisfactory. After enough hydrogen has been passed to sweep the air out of the oil, the temperature of the oil bath is raised to 130° to 140° C. and held there until the oil is dry. Tubes "A" and "B" are disconnected from the system and weighed. The increment in weight of the two tubes corresponds to the moisture in the sample. The tubes are replaced in the system and the train is ready for the next determination.

TABLE I.—RATE OF MOISTURE REMOVAL FROM COTTONSEED OIL SALAD OIL

25.7 gm. Sample, 0.062% Moisture.

Run Mins.	Actual Wts. Gms. Tube "A"	Actual Wts. Gms. Tube "B"	Mgms. Change Tube "A"	Mgms. Change Tube "B"	Total Moisture Obtained Mgms.	% Total Obtained
0.....	42.3660	50.1730	...	...	...	....
5.....	42.3750	50.1730	9.0	0	9.0	56.3
10.....	42.3785	50.1735	12.5	0.5	13.0	81.4
20.....	42.3800	50.1735	14.0	0.5	14.5	90.8
30.....	42.3810	50.1735	15.0	0.5	15.5	97.2
40.....	42.3810	50.1740	15.0	1.0	16.0	100.
60.....	42.3810	50.1740	15.0	1.0	16.0	100.

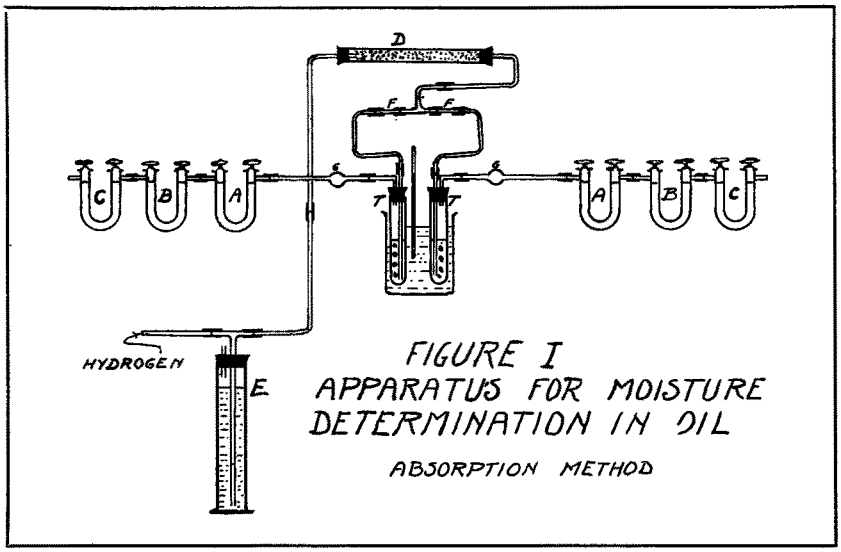

FIGURE I
APPARATUS FOR MOISTURE DETERMINATION IN OIL
ABSORPTION METHOD

Time Required for Evolution of Moisture:

It seemed advisable to determine the rate at which moisture was removed from the sample under the conditions of the method using about 100 cc. of hydrogen per minute through the system. In Table I the data is given for a typical run designed to measure the rate of moisture removal. It will be seen that the water is removed to within the accuracy of the weighing (± 0.5 mgm.) within 30 minutes. As a factor of safety, the evolution period was usually set at one hour.

Results Obtained With the Method:

In Table II a series of analyses representing samples of various types of salad oils selected at ran-

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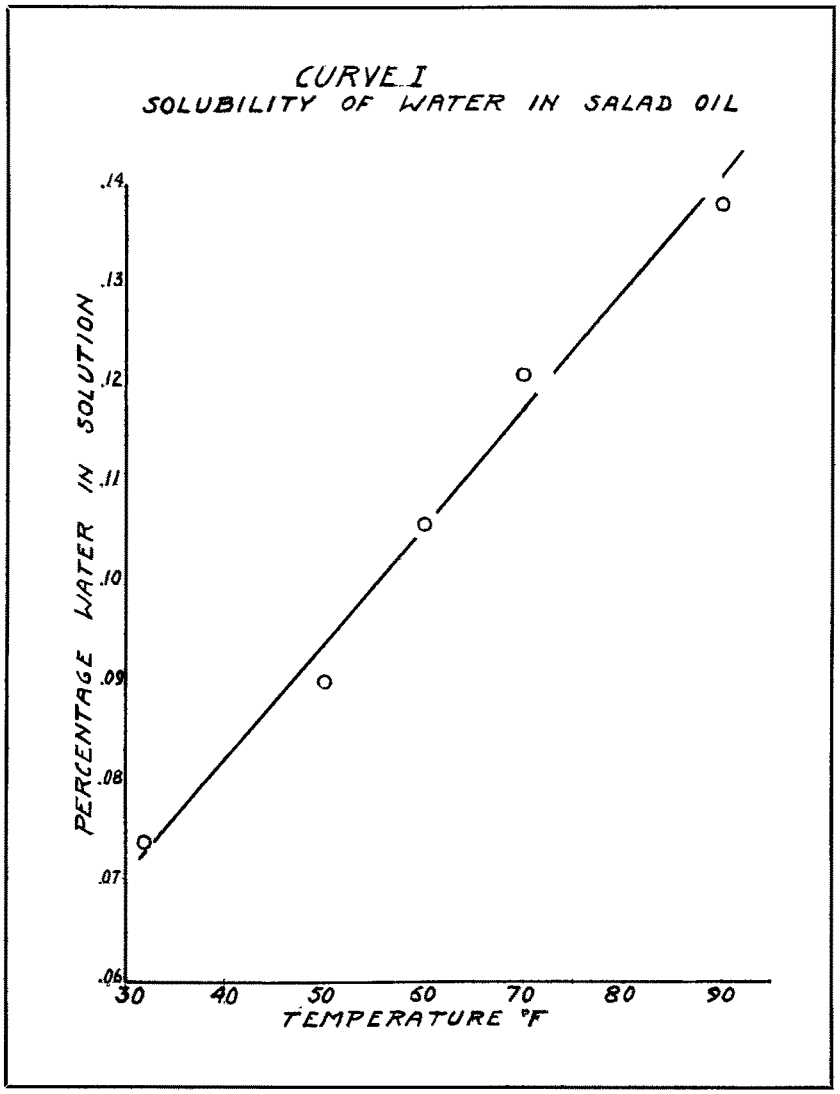
TABLE II.—MOISTURE ANALYSES ON SALAD OILS

Sample	Run No.	Sample Wt. Gms.	Mgms. Moisture Tube "A"	Mgms. Moisture Tube "B"	Total Moisture Mgms.	Total Moisture Percent
1.....	1	25.2	15.5	0	15.5	.062
	2	25.7	15.5	0.5	16.0	.062
2.....	1	27.0	14.0	0	14.0	.052
	2	27.6	14.0	0	14.0	.051
3.....	1	21.9	29.5	0	29.5	.135
	2	22.4	31.0	0	31.0	.138
	3	22.2	30.5	0.5	31.0	.140
	4	22.4	30.0	0.5	30.5	.136
4.....	1	32.8	8.0	0	8.0	.024
	2	30.1	8.0	0	8.0	.027
5.....	1	37.4	2.5	0	2.5	.0067
	2	40.1	2.5	0.5	3.0	.0075
6.....	1	38.4	13.0	0	13.0	.034
	2	40.3	13.0	1.0	14.0	.035
7.....	1	48.5	7.0	0.5	7.5	.015
	2	46.7	6.5	0.5	7.0	.015
8.....	1	51.2	10.0	0.0	10.0	.020
	2	50.5	10.0	1.0	11.0	.022
	3	50 5	12.5	0.0	12.5	.025

dom from laboratory records are tabulated. Results usually do not differ by more than .004%. The percentage variation is dependent on the moisture content of the oil and size of sample, the accuracy of weighing being the limiting factor on samples of low moisture. No statement can be made concerning the absolute accuracy of the method, and it has not been considered worthwhile to go to any great lengths to determine recoveries of added water. It can only be stated that rather precise results are obtained in practice.



The Determination of the Solubility of Water In Salad Oil:

Salad oils containing small amounts of water often show a haze when subjected to the cloud test. This may often be confused with the separation of solid glycerides leading to false conclusions about the low temperature stability of the oil. It seemed worthwhile to attempt to measure the temperatures at which oils of known moisture content showed slight clouding and hence to construct from such data the solubility curve for water in salad oil. This is obviously an application of the well-known critical solution temperature method of solubility determination. It is applied here to a rather special case of very slight solubility of one liquid (water) in another liquid (salad oil). The experimental technique consisted in measuring various portions of dry oil and wet oil of known moisture contents into necked down dried test tubes. These were immediately sealed off to prevent loss of water. These tubes were then used for the determination of the critical solution temperatures.

Salad oil was saturated with water by steaming and then allowing it to cool in a closed container kept at 98° F. The oil was decanted from the separated water and centrifuged in a 98° F. room to remove suspended droplets of water. After centrifuging, the oil which was approximately saturated with water at 98° F. was stored in a glass-stoppered bottle at this temperature until used. Another bottle of rather dry salad oil was held at the same temperature, these two serving as the stock solutions from which other mixtures were made. Oils were measured from 50 cc. burettes which were equipped with long slender tips capable of reaching to the bottom of the drawn out 5/8 in. by 6 in. dried test tubes. Immediately after measuring the tubes were sealed off. The oil was transferred from the stock bottles to the burettes and removed therefrom for analysis. Two separate sets of tubes were made covering the range of moisture content from .04 to 0.13%.

The tubes were transferred to a water bath at 98° and allowed to cool through about a 5° F. interval and then held there for ½ hour before examining. Tubes showing cloudiness were recorded, removed, and then the remainder were cooled through another 5° F. interval, held for half an hour and examined.

The complete data together with the approximate cloud points appear in Table III. It should perhaps be pointed out that no attempt was made to make precise measurements of the critical solution temperatures since these are rather time-consuming and were not warranted for our purposes. It was apparent during the experiments that the temperatures could be rather definitely established and could be approached from both directions.

In Curve I solubility of water in cottonseed salad oil is plotted as a function of temperature. It will be observed that the solubility of water in the oil approximately doubles in the range of 32° to 90° F. The practical implication of course is that salad oils must run less than .07% moisture if they are not to haze when cooled to 32° F. Further, the data may be used to determine roughly the percentage of dissolved moisture in salad oils by cooling these slowly and observing the temperature at which haze is first noted. This procedure is obviously limited to oils of from 0.07 to .14% moisture content.

TABLE III.—DATA ON CRITICAL SOLUTION TEMPERATURES OF VARYING AMOUNTS OF WATER IN SALAD OIL

Exp. No.	Duplicate	cc. into tubes—Oil of 0.138% Moisture	cc. into tubes—Oil of 0.043% Moisture	Calc. % Moisture in Oil Mixture	Critical Temp. of Solution Deg. F.
1	A	10.0	0	0.138	90
	B	10.0	0	0.138	90
2	A	8.3	1.7	0.121	72.5
	B	8.3	1.7	0.121	72.5
3	A	6.7	3.3	0.106	60.
	B	6.7	3.3	0.106	62.5
4	A	5.0	5.0	0.090	50.
	B	5.0	5.0	0.090	50.
5	A	3.3	6.7	0.074	32.
	B	3.3	6.7	0.074	32.
6	A	1.7	8.3	0.059	Clear at 32° F.
	B	1.7	8.3	0.059	
7	A	0	10.0	0.043	Clear at 32° F.
	B	0	10.0	0.043	

THE EFFECT OF FUEL OIL ON COLOR OF REFINED COTTONSEED OIL

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THE fact has been known for years that a minute quantity of the lubricating oil used in a cottonseed oil mill would, if allowed to come in contact with the cottonseed or cottonseed meats, cause the refined oil obtained from the crude cottonseed oil produced from the contaminated seed or meats to be increased in color with a fluorescent appearance. The fluorescent appearance and a portion of the increase in color persists in the bleached oil.

A cottonseed oil mill suddenly commenced producing oil which, by the above criterion, was contaminated with mineral oil. No source of lubricating oil contamination could be found at the mill. On investigation, the contamination was found to be due to fuel oil which became mixed with the cottonseed in transit from the gins to the mill. The mill was furnishing fuel oil to the gins in drums. The trucks going to the gins for seed carried the drums of fuel oil. The trucks on returning to the mill with seed brought back the empty drums on top of the seed, thus contaminating the seed with fuel oil.

Tank cars and tanks on ships which have been used to transport fuel oil are often used, after cleaning, to carry crude or refined cottonseed oil. If the tanks are not thoroughly cleaned of fuel oil before loading with cottonseed oil, the latter will be contaminated, causing an increase in color. The following study was made to determine the effect of fuel oil contamination on the color of refined cottonseed oil.

Three refined cottonseed oils, A, B, and C, were mixed with various proportions of fuel oils, X, Y, and Z, and the color of the yellow and bleached oils determined. The results obtained are given in Tables I, II, and III. In making the color readings, standard Lovibond color glasses were used. All yellow oils

TABLE I.—REFINED COTTONSEED OIL A

Mineral Oil Added	0 Y.O.	0 Bl.	.01% Y.O.	.01% Bl.	.02% Y.O.	.02% Bl.	.03% Y.O.	.03% Bl.
X	4.5	2.2	6.4	2.7	8.7	3.8	11.1	4.3
Y	4.5	2.2	6.6	2.9	9.5	3.8	12.7	4.3
Z	4.5	2.2	5.8	2.5	7.2	3.0	8.7	3.5

TABLE II.—REFINED COTTONSEED OIL B

Mineral Oil Added	0 Y.O.	0 Bl.	.01% Y.O.	.01% Bl.	.02% Y.O.	.02% Bl.	.03% Y.O.	.03% Bl.
X	5.8	2.3	7.8	2.7	9.9	3.6	11.2	4.5
Y	5.8	2.3	8.1	2.9	10.8	3.7	14.3	4.5
Z	5.8	2.3	7.1	2.6	8.5	3.0	9.8	3.5

TABLE III.—REFINED COTTONSEED OIL C

Mineral Oil Added	0 Y.O.	0 Bl.	.01% Y.O.	.01% Bl.	.02% Y.O.	.02% Bl.	.03% Y.O.	.03% Bl.
X	9.1	3.0	11.1	3.4	13.2	4.2	16.8	5.0
Y	9.1	3.0	11.5	3.5	14.6	4.5	19.8	5.3
Z	9.1	3.0	10.2	3.3	11.9	3.8	13.5	4.1