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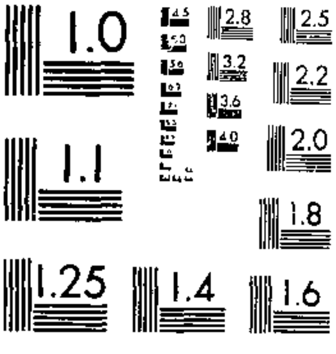

MICROCOPY RESOLUTION TEST CHART
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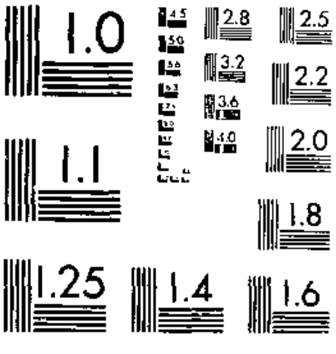

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RAPID DETERMINATION OF OIL CONTENT AND OIL QUALITY IN FLAXSEED

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ORGANIZATION OF THE RESEARCH

The flaxseed industry has long needed prompt and accurate methods for evaluating flaxseed in terms of the quantity and quality of its oil. The methods of analysis in common use, although accurate enough when properly employed, are too time-consuming to be of practical value in the commercial inspection of flaxseed.

To discuss the problem of oil tests for flaxseed a nationally representative conference was held in 1934. The linseed-oil manufacturing industry, the Flax Institute of the United States, the State agricultural experiment stations in the leading flax-producing States, the Agricultural Adjustment Administration, and the Bureaus of Plant Industry and Agricultural Economics of the United States Department of Agriculture took part.

Resolutions were passed at the conference petitioning the Secretary of Agriculture to sponsor a research activity having as its objective the development of rapid, accurate, and simple commercial methods for the determination of the quantity and quality of oil in flaxseed. This research project was assigned to the Bureau of Agricultural

[1] This investigation was made in cooperation with the Division of Cereal Crops and Diseases, Bureau of Plant Industry; and the Oil, Fat, and Wax Section, Carbohydrate Research Division, Bureau of Chemistry and Soils, U. S. Department of Agriculture.

[2] The authors gratefully acknowledge the cooperation of the chemists at the following institutions and laboratories who assisted in the collaborative phases of this work: Minnesota Agricultural Experiment Station, division of agricultural biochemistry; North Dakota Agricultural Experiment Station, department of agricultural chemistry; South Dakota Agricultural Experiment Station, department of agronomy; the Grain Research Laboratory of the Winnipeg Board of Grain Commissioners; Archer-Daniels-Midland Co., Minneapolis, Minn.; Empire Oil & Food Products Co., Portland, Oreg.; Minnesota Linseed Oil Co., Minneapolis, Minn.; National Lead Co., Brooklyn, N. Y.; Pittsburgh Plate Glass Co., Red Wing, Minn.; Producers Cotton Oil Co., Fresno, Calif.; and Spencer Kellogg & Sons, Inc., Buffalo, N. Y.

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Economics. Its experiences and responsibilities in grain inspection and its previous research pertaining to rapid methods for determining the oil content of flaxseed influenced this assignment. With the assistance of the Bureau of Chemistry and Soils and the Bureau of Plant Industry a collaborative research program was begun with interested members of the linseed-oil trade and the State agricultural experiment stations.

The project was inaugurated in April 1935 with the active cooperation of 11 chemical laboratories in the collaborative phases of the work. As a result the Bureau of Agricultural Economics is now recommending a method by which the oil content of flaxseed as well as the iodine number (the principal quality factor) of its oil can be determined accurately in a half hour as compared with the 16 to 24 hours required by the standard analytical procedures. Using this method, one analyst with two nontechnical assistants can make as many as 100 complete analyses for oil content and iodine number in an 8-hour day. This bulletin portrays the problems and describes the methods for making the tests.

NEED FOR SUITABLE OIL-TESTING METHODS

Flaxseed is grown in the United States chiefly in Minnesota, North Dakota, South Dakota, and Montana. It also is produced to a limited extent in Kansas, Iowa, Missouri, Nebraska, Colorado, Michigan, and Oregon. Since 1933 flaxseed has become an increasingly important crop in California, where its production has increased from 11,000 bushels in 1933 to 570,000 bushels in 1935. The average annual production of flaxseed in this country over the 10-year period 1926–35 was 15,066,000 bushels.

The flaxseed crop is used primarily for the manufacture of linseed oil, which in turn is used principally in the preparation of paints, varnishes, and linoleum. Linseed cake, the byproduct of flaxseed processing, is a valuable feed for livestock. At average prices for linseed oil and linseed cake, the oil represents about 70 percent and the cake about 30 percent of the market value of flaxseed products.

It is obvious that the value of any lot of flaxseed depends primarily upon the quantity and quality of linseed oil it will yield. Oil quality is concerned chiefly with the "drying" properties of the oil, that is, the rapidity with which the oil will oxidize to a solid film, and the character of the film produced. In commercial practice, quality is generally expressed in terms of iodine number, which is a quantitative measure of the total amount of oxygen the oil is potentially able to absorb.

Both the quantity and the quality of oil in flaxseed vary over wide ranges. In commercial seed the oil content ranges from about 32 to about 45 percent on a dry-weight basis, and the iodine number (Wijs) varies from 155 to 200. In tables 1 and 2 are shown the average values for oil content and iodine number for various classes of commercial flaxseed over a period of years.

Under present practices tests for oil content and oil quality are not used officially in the commercial inspection of flaxseed. The official standards of the United States for flaxseed are based on such quality factors as test weight per bushel, percentage of damaged seeds, dockage, moisture content, and "condition." Although these factors are of value in measuring the net weight, soundness, and storage qualities of flaxseed, they bear no significant relationship to either the quantity or the quality of the oil.

TABLE 1.—*Oil content of different classes of commercial flaxseed calculated on 8-percent moisture basis*

Item	1930	1931	1932	1933	1934	1935
Northwestern:[1]						
Maximum ... percent	39.0	39.5	39.0	40.0	40.5	...
Minimum ... do	31.0	31.5	30.0	32.0	33.5	...
Average ... do	35.2	35.8	35.7	36.4	37.0	...
Samples analyzed ... number	164	732	846	694	644	...
Canadian:[2]						
Maximum ... percent	...	...	...	...	40.6	41.6
Minimum ... do	...	...	...	...	34.0	35.2
Average ... do	...	...	...	...	38.0	38.7
Samples analyzed ... number	...	...	...	...	148	210
California:						
Maximum ... percent	...	...	...	...	40.9	40.2
Minimum ... do	...	...	...	...	36.9	34.6
Average ... do	...	...	...	...	39.7	38.0
Samples analyzed ... number	...	...	...	...	7	38

[1] Reported by T. H. Hopper, North Dakota Agricultural Experiment Station.
[2] Reported by W. F. Geddes and F. H. Lehberg, Board of Grain Commissioners, Winnipeg, Manitoba, Canada.

TABLE 2.—*Iodine number (Wijs) of different classes of flaxseed*

Item	1930	1931	1932	1933	1934	1935
Northwestern:[1]						
Maximum ... percent	191	191	192	188	188	...
Minimum ... do	154	154	152	152	159	...
Average ... do	176	178	176	175	174	...
Samples analyzed ... number	164	732	846	694	644	...
Canadian:[2]						
Maximum ... percent	...	...	...	...	196	202
Minimum ... do	...	...	...	...	173	172
Average ... do	...	...	...	...	184	190
Samples analyzed ... number	...	...	...	...	148	210
California:						
Maximum ... percent	...	...	...	...	191	197
Minimum ... do	...	...	...	...	175	176
Average ... do	...	...	...	...	182	184
Samples analyzed ... number	...	...	...	...	7	38

[1] See footnote 1, table 1.
[2] See footnote 2, table 1.

This failure to utilize data on oil content and oil quality in the commercial inspection of flaxseed has been due in part at least to the fact that the methods heretofore accepted for making these determinations are too time consuming for such purposes. Various more or less promising rapid analytical methods have been proposed from time to time but none has been sufficiently perfected or tested to meet with complete approval by the linseed oil and flaxseed industries.

The linseed-oil industry has been greatly concerned by the abnormally large proportion of low-quality flaxseed that has been marketed during recent years. The principal factors that have contributed to this production of low-quality flaxseed are:

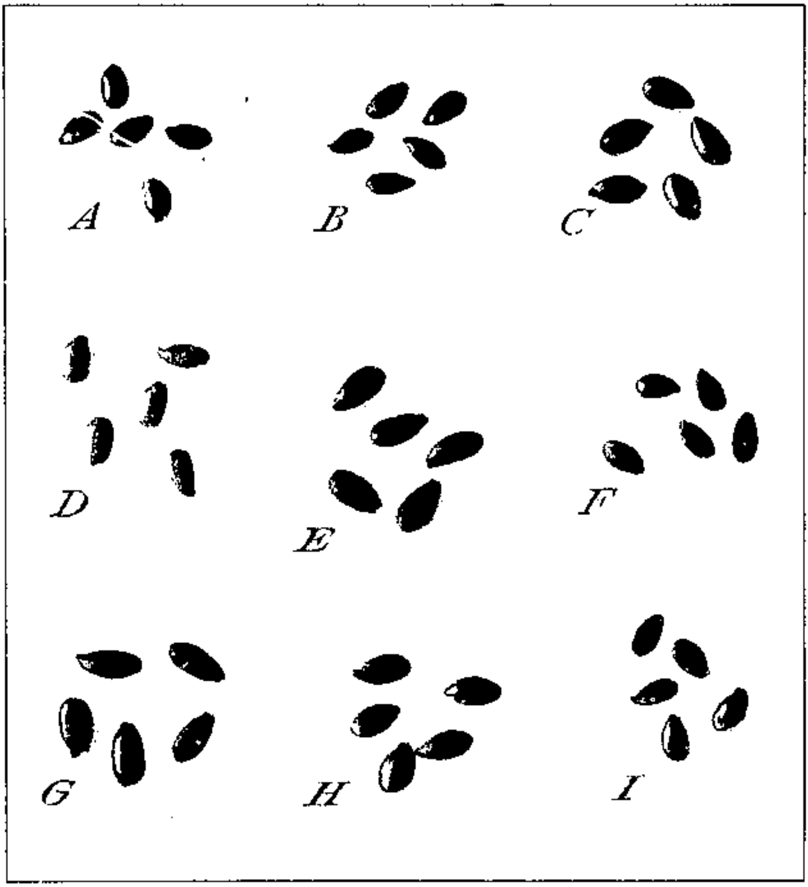

FIGURE 1.—Principal commercial varieties of flaxseed: *A*, Linota; *B*, Redwing; *C*, Bison; *D*, Bolley Golden; *E*, Argentine; *F*, Buda; *G*, Punjab; *H*, Abyssinian; *I*, N. D. R. 114.

(1) Drought conditions accompanied by abnormally high temperatures during the growing season in the flax-growing areas.

(2) The introduction of new varieties of flax which have been bred for their resistance to disease and for their large yields of seed, but which inherently tend to produce a low quality oil. Figure 1 shows typical seeds of the principal commercial varieties of seed flax.

(3) The growing of flax in areas that are unsuitable for the production of high-quality flaxseed.

On the other hand, new varieties are gradually being developed and introduced which under similar growing conditions produce a higher quality of seed than do the present principal varieties. Then flax is being raised to a limited extent in new areas where the climatic conditions are unusually favorable to the production of high-quality seed.

All of these factors have had the effect in recent years of decreasing the uniformity of the domestic flaxseed crop, and have thus emphasized the need for reliable and rapid methods for testing flaxseed for oil content and oil quality.

DETERMINATION OF OIL CONTENT OF FLAXSEED

Before attempting to develop a suitable rapid method for determining the oil content of flaxseed, it was found necessary to establish an acceptable standard method against which the results of any new method could be checked. A survey was therefore made of the methods used for the oil-content analysis of flaxseed by various commercial and State agricultural experiment station laboratories.

THE EXTRACTION METHOD

The ether-extraction method of the Association of Official Agricultural Chemists (*2, pp. 279–280*) [3] or some modification thereof was found to be in general use among the laboratories investigated. The outstanding differences in the technique of the extraction method as practiced by 11 oil-testing laboratories are recorded in table 3.

TABLE 3.—*Different modifications of the extraction method for determining the oil content of flaxseed as practiced by 11 laboratories*

Laboratory	Apparatus used for grinding sample	Ground sample dried before extraction	Weight of sample extracted	Type of extraction apparatus	Solvent	Sample reground after partial extraction	Method of driving off last traces of solvent
			Grams				
A	Roller mill	No	2	Pickel	Ethyl ether	No	Air dried.
B	Mortar with sand.	No	3	Soxhlet	Petroleum ether	No	Air oven, 105° C., 20 minutes.
C	Roller mill	No	2½	do	Carbon tetrachloride.	No	Air oven, 105° C., 3 minutes.
D	Attrition mill.	No	5	Bailey-Walker	Petroleum ether	Yes	Air oven 100°–105° C., 30 minutes.
E	Roller mill	No	2	do	Ethyl ether	Yes	Air oven, 80° C.
F	do	Yes		Soxhlet	do	No	Vacuum oven, 105° C., 8 hours.
G	Mortar	No	4	Reflux	Ethylene dichloride.	No	
H	Roller mill	No	4–5	Pickel	Ethyl ether	Yes	Vacuum oven, 80° C., 30 minutes.
I	Attrition mill.	Yes	3	Bailey-Walker	do	No	Vacuum oven (CO_2), 65° C., 8 hours.
J	Burr mill	Yes	5	Soxhlet	do	No	
K				Butt tube			

Four types of grinding equipment; five types of extraction apparatus, and four different solvents were found in use among these laboratories. In some laboratories the ground samples were dried

[3] Italic numbers in parentheses refer to Literature Cited, p. 37.

before extraction whereas in others they were not. Regrinding of the sample with abrasive material after partial extraction was practiced by three of the laboratories. There were marked differences in the size of samples used for extraction and in the methods employed for driving off the solvent from the extract.

To determine the extent of the variations in analytical results obtained by the different laboratories as a result of the differences in the method of analysis used, subsamples from 11 thoroughly mixed samples of flaxseed were distributed to 9 laboratories for oil-content analysis, each laboratory being instructed to analyze the samples by the method it customarily employed. The reports of these laboratories, as recorded in table 4, show wide divergences in analytical results, thus indicating the necessity for establishing a standardized "reference" procedure.

TABLE 4.—*Percentage of oil in 11 samples of flaxseed as determined by 9 laboratories, each laboratory using its own method of analysis*

[All data are averages of duplicate determinations]

Sample no.	Laboratory 1	Laboratory 2	Laboratory 3	Laboratory 4	Laboratory 5	Laboratory 6	Laboratory 7	Laboratory 8	Laboratory 9	Average
	Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*
1	37.40	36.95	37.87	33.50	35.85	36.06	36.94	38.04	37.12	36.74
2	38.52	38.36	39.79	34.36	40.88	38.58	39.00	39.03	38.35	38.61
3	37.17	38.18	39.14	37.76	37.22	37.84	38.34	39.12	37.99	38.08
4	39.95	40.21	40.18	39.50	39.95	39.82	39.70	41.19	39.70	40.02
5	36.82	36.12	37.08	35.50	37.36	37.32	37.78	38.02	36.08	37.10
6	37.33	37.58	37.04	34.30	37.05	37.53	38.71	38.58	37.50	37.29
7	37.08	36.25	37.76	33.86	36.21	36.70	37.01	37.40	36.25	36.51
8	38.26	37.89	37.71	37.14	37.82	38.16	38.49	38.41	38.34	38.02
10	36.05	35.67	37.73	35.58	36.80	36.66	37.84	38.06	36.53	36.81
11	41.65	43.68	43.12	41.76	41.02	42.71	44.37	43.86	43.34	42.83
12	39.38	38.60	40.04	37.24	39.13	39.04	39.38	40.26	39.05	39.13

Standard deviation between laboratories: 2.73 percent oil. Experimental error: ±0.71 percent oil.

SOURCES OF ANALYTICAL DIFFERENCES IN THE EXTRACTION METHOD

A careful study was made of the various steps in the oil-determination procedure used in the various collaborating laboratories to discover, if possible, the chief causes of these divergences, and to develop a method that would be capable of producing reasonably concordant results between different laboratories. From a study of the data the following possible sources of error were investigated and recommendations were made accordingly: (1) Sampling, (2) grinding, (3) oxidation of ground sample, (4) extraction apparatus, (5) solvent, and (6) method of removing solvent from extract.

SAMPLING

The importance of obtaining a representative sample for analysis from a carload lot or other large quantity of seed is well known. The present problem, however, concerns itself with the procuring of a representative aliquot of the laboratory sample for analysis. A study of aliquots taken from different parts of a quart container of clean flaxseed does not indicate any appreciable difference in oil content. But as a precautionary measure it is recommended that aliquots for analysis be obtained either by hand quartering the laboratory sample or by the use of a mechanical sampling device.

GRINDING

Improper grinding of flaxseed may result in three different sources of error.

Incomplete Extraction.—Unless the seed is in a very fine state of subdivision it is not possible to extract the oil quantitatively.

Subsamples of a well-mixed lot of flaxseed were ground by six laboratories and returned in sealed containers to the Grain Division laboratory. These ground samples were extracted for 17 hours with petroleum ether and determinations were made of the quantities of oil extracted. The extracted residues were sifted through a 40-mesh gauze sieve and the percentages of material retained on the sieve were determined. The residues were then reground in a mortar with carborundum and were re-extracted for 17 hours with petroleum ether. Table 5 shows the quantity of oil that failed to be extracted by petroleum ether in the first 17-hour extraction as related to the fineness of grind.

TABLE 5.—*Relationship between fineness of grind and extractability of oil in flaxseed*

Extracted material retained by 40-mesh gauze (percent)	Oil not extracted after 17 hours		Extracted material retained by 40-mesh gauze (percent)	Oil not extracted after 17 hours	
	Percent	Percent of total oil in sample		Percent	Percent of total oil in sample
3	0.11	0.3	13	0.40	1.1
11	.26	.7	18	.60	1.0
13	.37	1.0	29	.93	2.5

These data indicate a close relationship between the fineness of grinding and the extractability of the oil. It is therefore recommended that all samples be reground with abrasive material after a few hours of preliminary extraction, unless it has been conclusively demonstrated that the method of grinding used produces a meal that will not yield an additional significant quantity of oil by extraction, when the sample is reground after a 16- to 24-hour preliminary extraction.

Segregation.—In flaxseed that is too coarsely ground, there is a considerable tendency for the finer particles, consisting chiefly of endosperm material, to become separated from the coarser seed-coat particles. When this condition exists, it is not possible to weigh out aliquots that are truly representative of the entire ground sample, hence duplicate determinations will fail to check, even though the weighed portions are reground with an abrasive.

Adherence of Oily Material to the Mill.—When flaxseed is ground in any type of mill, a certain quantity of the ground seed will adhere to the rolls or other grinding surfaces. In general, this material will be of higher oil content than the bulk of the ground seed. The complete removal of this adhering material and its mixture with the bulk of the sample is a tedious procedure. It is therefore recommended that in grinding a sample for analysis, a preliminary portion of the sample be ground and discarded before the main sample is ground.

This will allow the moving parts of the mill to become saturated with the oily material and thus prevent the loss of oil from the part of the sample to be used for analysis.

OXIDATION OF GROUND SAMPLE

As linseed oil is highly unsaturated chemically, it absorbs oxygen readily from the air, forming oxidation products that are relatively insoluble in the ordinary fat solvents. Care must be taken to avoid any treatment of the ground sample that would tend to cause excessive oxidation.

Portions of two samples of ground flaxseed were analyzed by petroleum-ether extraction at varying intervals after they were ground. The samples were kept in tightly stoppered glass bottles at room temperature and away from direct sunlight. The analyses (table 6) indicate no appreciable change in the quantity of extractable oil when ground samples are stored in this way for 28 days.

TABLE 6.—*Oil content of flaxseed samples as determined by petroleum-ether extraction at different intervals after grinding*

[Averages of duplicate determinations]

Period after grinding (days)	Sample A	Sample B	Period after grinding (days)	Sample A	Sample B
	Percent	*Percent*		*Percent*	*Percent*
0	34.91	42.70	13	34.98	42.63
1	35.02	42.01	28	35.02	42.83
3	34.88	42.61			

It is possible that in these experiments a significant amount of oxidation may have occurred, but if so it did not affect the results appreciably because the insoluble oxidized fraction of the oil was compensated for by the increase in weight of the soluble partially oxidized fraction. The best practice is to analyze the samples as soon as possible after they are ground.

Drying Sample Previous to Extraction.—Drying the ground sample before extraction is sometimes practiced, and appears to be necessary when anhydrous ethyl ether is used as the solvent. Table 7 records a comparison of the quantity of oil extracted from a series of samples before drying and after drying by two different methods.

TABLE 7.—*Oil content of flaxseed samples as determined by 24-hour petroleum ether extraction of undried and dried samples*

[Averages of duplicate determinations]

Sample	Undried	Dried[1] 6½ hours in vacuum oven at 100° C.	Dried[1] 17 hours in air oven at 100° C.	Sample	Undried	Dried[1] 6½ hours in vacuum oven at 100° C.	Dried[1] 17 hours in air oven at 100° C.
	Percent	*Percent*	*Percent*		*Percent*	*Percent*	*Percent*
C	37.99	37.72	27.17	F	37.39	36.24	----
D	37.40	36.73	31.60	G	36.46	36.07	----
E	42.07	42.24	21.28	H	43.03	39.97	----

[1] Samples dried after being weighed.

Drying the samples reduced the quantity of oil extractable by petroleum ether, probably because of partial oxidation of the oil. The extracts of the undried samples were not contaminated by water-soluble products since water is nearly insoluble in petroleum ether and since the extracts in all cases were perfectly clear. It is therefore recommended that drying of the ground sample before extraction be avoided.

EXTRACTION APPARATUS

Six replicate samples of ground flaxseed were extracted with petroleum ether for 21 hours on four types of extraction apparatus. The results obtained are given in table 8.

TABLE 8.—*Oil content of replicate samples of flaxseed as determined by 21-hour petroleum-ether extraction using different types of extraction apparatus, and as determined by the modified refractometric method*[1]

Goldfisch extractor	Soxhlet extractor	Butt tube extractor	Bailey-Walker extractor	Modified refractometric method
Percent	*Percent*	*Percent*	*Percent*	*Percent*
36.45	36.47	36.42	36.29	36.33
36.32	36.30	36.37	36.41	36.23
36.17	36.33	36.46	36.20	36.53
36.21	36.35	36.27	36.29	36.26
36.32	36.31	36.28	36.36	36.43
36.38	36.37	36.40	36.31	36.43
[2] 36.31	[2] 36.36	[2] 36.38	[2] 36.32	[2] 36.37

[1] See p. 29. [2] Average.

The data given in table 8 indicate that equally satisfactory results may be obtained with the Goldfisch, Soxhlet, Butt tube, or Bailey-Walker types of extraction apparatus. Various other types of equipment are probably also entirely satisfactory for the quantitative extraction of oil from flaxseed. Different types of extraction apparatus in common use are shown in figures 2 to 6.

SOLVENT

The solvents in most common use for determining the oil content of flaxseed by extraction are anhydrous diethyl ether and petroleum ether.

As water is appreciably soluble in diethyl ether, an extract made with this solvent is likely to be contaminated with water-soluble products unless the sample is thoroughly dried before extraction. It has already been shown that the process of drying the finely ground flaxseed sample is likely to cause sufficient oxidation to lower the solubility of the oil appreciably.

Evidence has also been obtained to show that diethyl ether extracts a certain quantity of nonoily constituents from flaxseed which are insoluble in petroleum ether. Three samples of freshly ground undried flaxseed were extracted for 17 hours with petroleum ether. The residues in each case were dried and reground in a mortar with carborundum after which they were re-extracted for 17 hours with petroleum ether. The residues were again dried and then extracted for an additional 17 hours with anhydrous diethyl ether. The results of these analyses are listed in table 9.

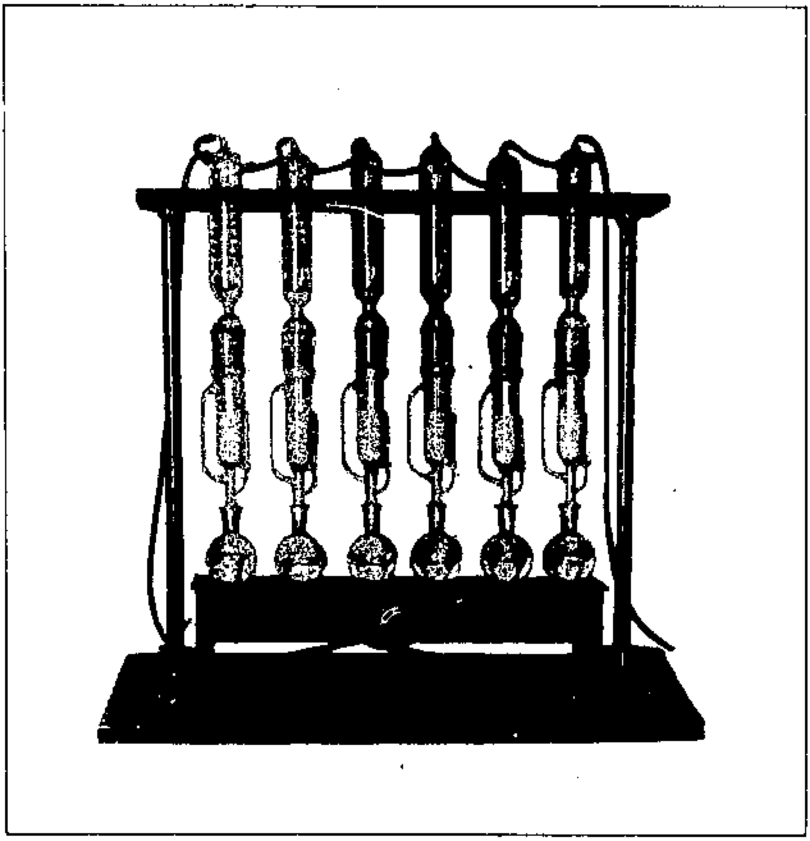

FIGURE 2.—Soxhlet extraction apparatus.

TABLE 9.—*Fraction of flaxseed soluble in anhydrous diethyl ether and insoluble in petroleum ether*

[All data are averages of duplicate determinations]

Sample no.	Extract A [1]	Extract B [2]	Extract C [3]
	Percent	*Percent*	*Percent*
1	37.39	0.11	0.44
2	30.46	.12	.51
3	43.03	.10	.42

[1] Extract A, petroleum ether extract.
[2] Extract B, petroleum ether extract of residue from A, after drying and regrinding with carborundum.
[3] Extract C, anhydrous diethyl ether extract of dry residue from B.

The material extracted by the diethyl ether (extract C) was of a solid nonoily, and partly crystalline composition, and was insoluble in water. Anhydrous diethyl ether, therefore, extracts from 0.4 to

0.5 percent of material from flaxseed which is not extracted by petroleum ether and which should not properly be considered as a part of the oil content of the flaxseed.

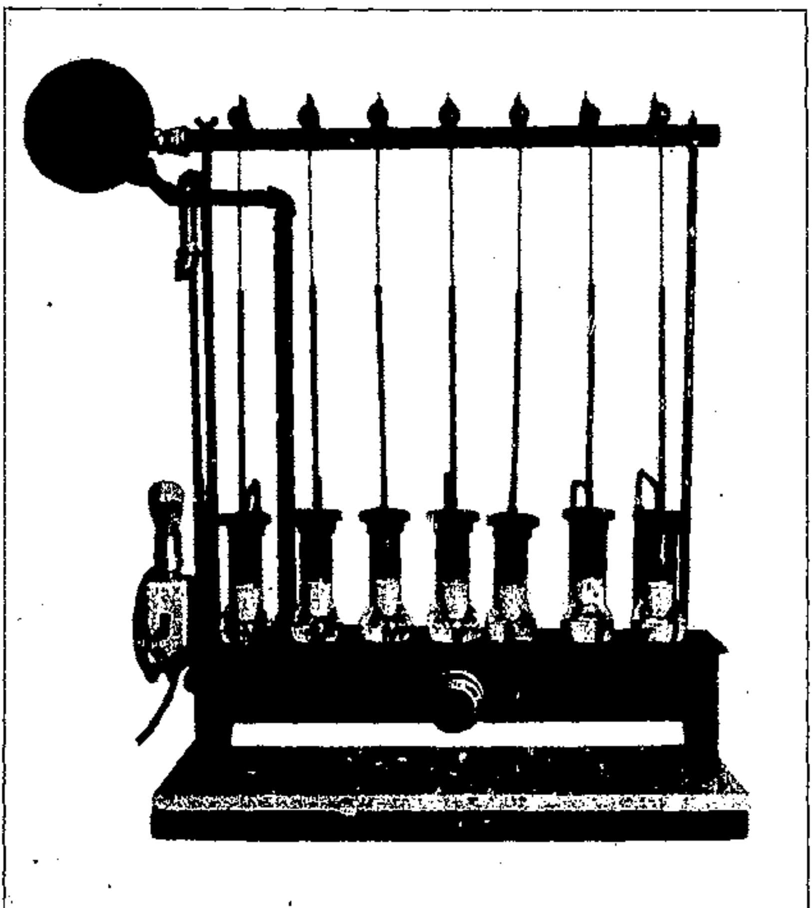

FIGURE 3.—Bailey-Walker extraction apparatus.

Although the use of anhydrous diethyl ether as an extractive for flaxseed introduces two important sources of error in the determination of oil content, analyses made using this solvent agree very well, in general, with analyses made with the use of petroleum ether as a solvent. This is probably because of an approximate compensation of the two errors involved.

According to the data in table 9, anhydrous diethyl ether extracts about 0.5 percent more material, on the average, than does petroleum ether. But since, when diethyl ether is used, the ground seed must be vacuum-dried before extraction, its apparent oil content (table 7) is reduced on the average by about 0.5 percent. Thus the errors involved by the use of anhydrous diethyl ether are largely neutralized.

Petroleum ether, however, is recommended for the extraction of flaxseed since its use seems to involve fewer sources of error than does the use of diethyl ether. For the sake of uniformity a petroleum ether conforming to the following specifications,[4] which have been adopted as official for the analysis of cottonseed (*23*) should be used:

Initial boiling temperature, not less than 35° C., nor over 40°.
Dry-flask end point, not over 60° C., nor less than 50°.
Distilling under 55° C., at least 95 percent.
Distilling under 40° C., not over 85 percent.
Specific gravity at 60° F., 0.630 to 0.675.
Color, water white.
Residue on evaporation, not over 0.002 percent by weight.
Doctor test, sweet.
Copper-strip corrosion test, noncorrosive.
Unsaturated compounds, trace only permitted.

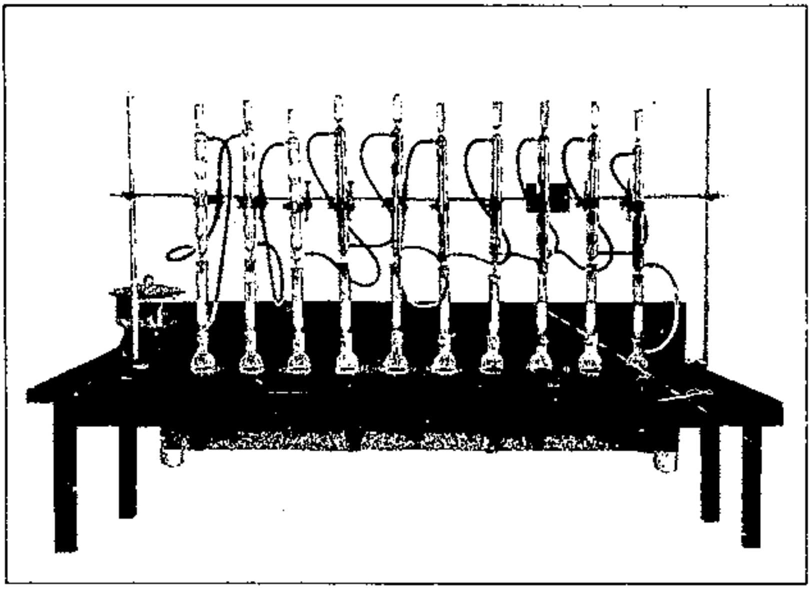

FIGURE 4.—Butt-tube extraction apparatus.

METHOD OF REMOVING SOLVENT FROM EXTRACT

Incomplete evaporation of the solvent from the dissolved extract will naturally result in high analytical results. Prolonged heating of the extract also will lead to high results because of oxidation of the oil. The optimum length of time for heating the extract therefore should be that at which the minimum weight for the extract is obtained. After removal of the bulk of the solvent on the steam bath the following three methods for removing the last traces of solvent have been found to give equally satisfactory results: (1) Heating an additional 1½ hours on the steam bath, (2) heating 1½ hours in an air oven at 100° C., and (3) heating 30 minutes in a vacuum oven at 100°.

[4] See also recommendation of the American Oil Chemists Society (*24*).

The question logically arises here why a 1½-hour heating of the extract at 100° C. in air will not cause sufficient oxidation to produce a significant increase in weight, it having already been shown that drying the ground seed under similar conditions causes rapid oxidation of the oil. The apparent reason is that in the case of the finely ground seed a large surface is exposed to the air, while in the case of the oil in the extraction flask the surface area is relatively insignificant.

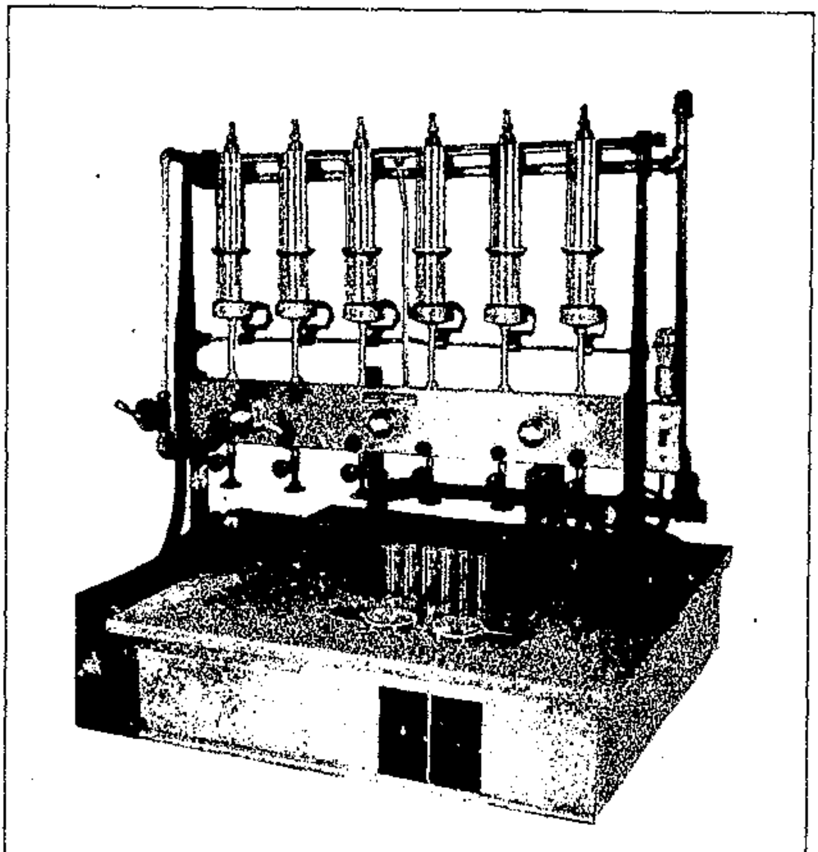

FIGURE 5. Goldfisch extraction apparatus.

STANDARD PETROLEUM-ETHER EXTRACTION METHOD

From a careful consideration of the foregoing study of the extraction method, a procedure was formulated for the determination of oil in flaxseed by extraction, the aim of which was to eliminate as far as possible the errors arising from the various sources studied. This method was subjected to collaborative study by 10 laboratories including the 9 that collaborated in the study previously discussed. Each laboratory analyzed 10 subsamples of flaxseed, representing 10 individual varieties, by the proposed method.

The results of these analyses are listed in table 10. Statistical analysis of these data shows a standard deviation between laboratories of 1.55 percent oil in the case of the standard petroleum-ether extraction method as compared with a standard deviation of 2.73 percent oil when each laboratory used its own individual extraction procedure. The experimental error also is reduced to ±0.39 percent oil for the standard procedure as compared with ±0.71 percent for the individual procedures. The results obtained by the standard petroleum-ether extraction method were considered sufficiently satisfactory to justify the adoption of the procedure as a standard reference method with which to compare the results obtained by other less orthodox methods of analysis.

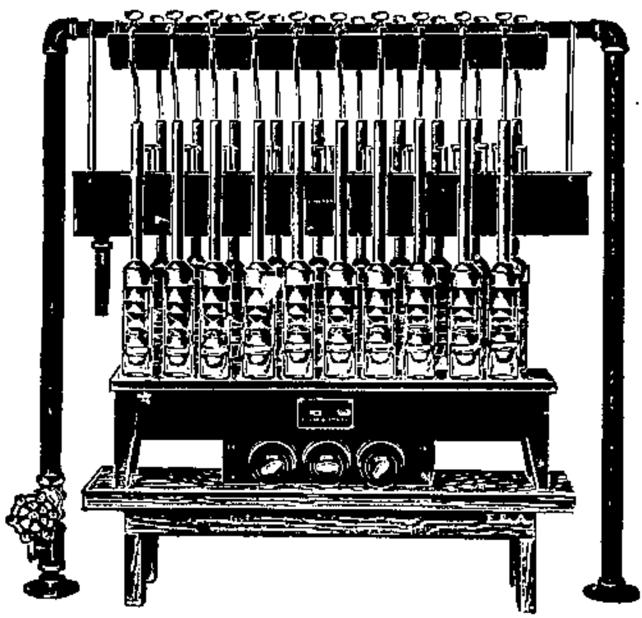

FIGURE 6.—Pickel extraction apparatus.

DETAILS OF STANDARD EXTRACTION METHOD

(1) The clean sample should be reduced to approximately 50 g by use of a mechanical sampler or by hand quartering.

(2) A motor-driven experimental roller flour mill with 6- by 6-inch rolls, 40 corrugations to the inch, is recommended for grinding. The rolls should have a speed differential of 9:7 and the faster roll should have a speed of approximately 900 revolutions per minute. The rolls should be adjusted to the minimum possible clearance but must not make contact. Such a mill is shown in figure 7.

Run about one-half of the 50-g sample through the mill, brush off any loose material from the stationary parts of the mill, and discard the ground material. Then, without cleaning the rolls or other moving parts of the mill, run through the remainder of the sample, again brushing any loose material on the mill into the sample and again not cleaning the rolls or other moving parts.

(3) Samples should be weighed and extraction begun immediately after the grinding. In case any appreciable time elapses between

TABLE 10.—*Oil in 10 samples of flaxseed, representing 10 varieties, as determined by 10 laboratories using the standard petroleum-ether extraction method*

[All data are averages of duplicate determinations]

Sample no.	Variety	Laboratory 1	Laboratory 2	Laboratory 3	Laboratory 4	Laboratory 5	Laboratory 6	Laboratory 7	Laboratory 8	Laboratory 9	Laboratory 10	Average
		Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*
31	New Golden	36.90	37.05	37.09	36.22	37.12	36.40	36.78	37.18	37.93	36.96	36.96
32	N. D. R. 114	34.80	35.06	34.98	34.58	35.09	34.18	34.41	35.02	35.77	34.58	34.85
33	Buda	35.40	35.87	35.71	34.45	35.72	35.21	35.26	35.37	36.78	35.55	35.53
34	Walsh	37.73	38.39	37.73	36.87	38.04	37.01	37.29	37.49	38.76	37.65	37.70
35	Bison	35.93	36.60	35.82	35.87	35.86	35.37	35.55	35.84	36.39	35.48	35.87
36	Punjab	41.13	42.36	40.51	40.28	42.06	41.17	41.24	41.93	40.49	41.75	41.29
37	Bolley Golden	38.38	38.35	37.82	37.73	38.50	37.97	37.93	38.43	39.50	38.06	38.27
38	Rio	38.20	38.88	37.66	36.86	38.45	37.34	38.28	38.03	39.31	38.15	38.12
39	Redwing	35.60	35.78	35.20	32.73	35.41	34.61	35.01	34.93	36.41	34.76	35.05
40	Linota	33.58	34.03	33.41	33.22	34.06	33.32	33.53	34.41	34.83	33.61	33.80

Standard deviation between laboratories: 1.55 percent of oil.
Experimental error: ±0.39 percent of oil.

these operations the ground samples should be kept in tightly stoppered glass bottles in a cool place and out of direct sunlight. After thoroughly mixing the ground sample weigh out accurately duplicate 2- to 5-g portions and transfer quantitatively to the extraction thim-

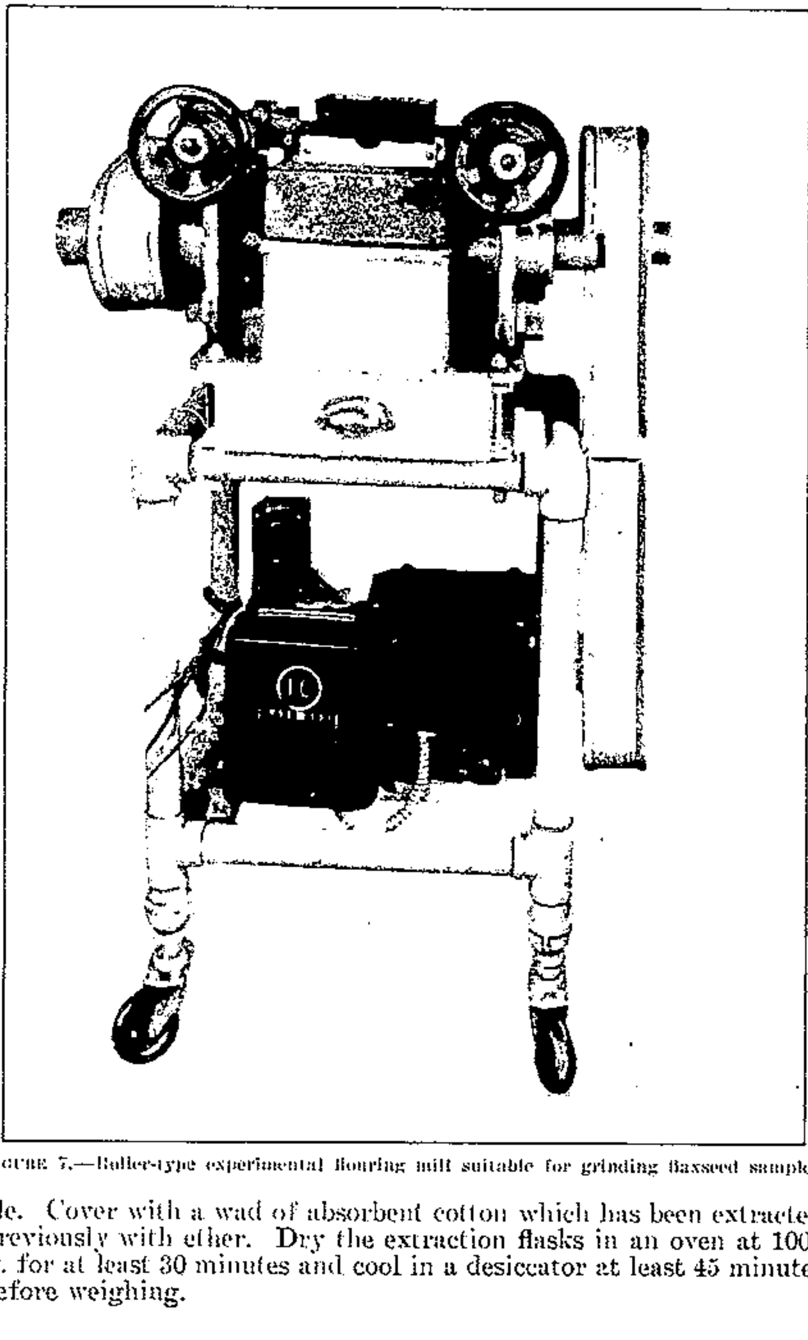

FIGURE 7.—Roller-type experimental flouring mill suitable for grinding flaxseed samples.

ble. Cover with a wad of absorbent cotton which has been extracted previously with ether. Dry the extraction flasks in an oven at 100° C. for at least 30 minutes and cool in a desiccator at least 45 minutes before weighing.

(4) Any standard type of ether-extraction apparatus should give satisfactory results when properly operated. Extract with a petroleum ether conforming to the official specifications for petroleum ether for cottonseed extraction (*23*). Adjust heat so that the solvent condenses at a rate of at least 100 drops per minute. After a 3- to 4-hour extraction remove the thimbles and place them in a well-ventilated oven at 100° C. for a time just long enough to drive off the solvent and any condensed moisture. Transfer the sample to a small mortar and grind thoroughly with a quantity of carborundum or fine washed and ignited sand equal to about one-half the weight of the sample. Replace in thimbles and continue the extraction for 16 to 18 hours.

(5) After the extraction is complete, disconnect the flasks and evaporate the bulk of the solvent on the steam bath, carefully tipping the flasks from time to time to remove the solvent vapors. When no more vapor appears to be generated remove the flasks from the steam bath and drive off the last traces of solvent by one of the following methods: (*a*) Place flasks in a vacuum oven at 95° to 100° C. for 30 minutes. (*b*) Place flasks in a well-ventilated air oven at 95° to 100° for 1½ hours. (*c*) Return flasks to steam bath for 1½ hours.

(6) Remove flasks from oven or steam bath and cool in a desiccator for at least 45 minutes. Weigh immediately on removal from desiccator. If duplicates do not agree within 0.2 percent of oil, the determination should be repeated.

Although the extraction method when properly used may be considered the most fundamentally accurate method for determining the oil content of flaxseed, it leaves much to be desired for routine commercial analysis. For certain commercial requirements a method is needed by means of which the oil content can be determined in a relatively short time. This is particularly true in the commercial inspection of carlots of flaxseed, when it is impracticable to hold the cars on track for the length of time required to run oil analyses by the conventional extraction procedure.

Using this standard petroleum-ether extraction method as a "reference" method, these studies were continued into the field of the development and perfection of a rapid method of analysis that would be sufficiently accurate, in comparison with the reference method, for commercial purposes.

THE CENTRIFUGAL METHOD

Lewis (*18*), in determining the oil content of pecans, has used a modification of the Babcock test for determining the butterfat content of dairy products. The method consists in digesting the ground nut meats with sulphuric acid, centrifuging the mixture in Babcock cream-test bottles, and measuring the volume of oil liberated. Traub [5] has used a similar method for determining the oil content of avacados.

Using a technique essentially the same as that of Lewis, it was found impossible quantitatively to separate the oil from the flaxseed meal. Increasing the concentration of sulphuric acid or the temperature of digestion resulted in a charring of the oil. Increasing the speed or time of centrifuging did not materially increase the quantity of oil liberated. It appears that the relatively large quantity of cel-

[5] Personal communication.

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lulose and related substances in the seed coat, which are indigestible in the sulphuric acid, mechanically prevent the quantitative liberation of the oil.

Further study of the method, however, indicated that the ratio of the quantity of oil liberated to the total oil content of the meal as determined by petroleum ether extraction was in all cases relatively constant. Of 10 samples tested, ranging in oil content from 33 to 43 percent, the percentages of the total oil liberated ranged from 88.55 to 92.09 and averaged 90.17. By dividing the weight of oil liberated by the factor 0.9017 it should therefore be possible to determine the approximate oil content by the following procedure:

PROCEDURE FOR THE CENTRIFUGAL METHOD

(1) Sample and grind the seed in the same way as for petroleum ether extraction.

(2) Weigh out accurately duplicate 4-g samples of the ground seed, introducing them into standard 6-inch Babcock milk-test bottles by means of a suitable glass funnel.

(3) Add 35 ml of 73.4-percent sulphuric acid, specific gravity 1.655, to each bottle and digest for 15 minutes in a water bath at 65° C., shaking the bottles occasionally.

(4) Place the bottles in 6-inch centrifuge cups, nearly fill the cups with water, and centrifuge at approximately 2,000 revolutions per minute for 5 minutes.

(5) Fill the bottles to the bottoms of the necks with the same acid and continue centrifuging for 3 minutes.

(6) Fill the bottles to near the tops of the graduated portions with acid and centrifuge for 1 minute.

(7) Add a drop or two more of the acid to facilitate reading, and measure the volume of oil with calipers. Each major division on the bottle neck is equivalent to 0.20 ml.

(8) Calculate the percentage of oil by the following formula:

$$\frac{100\ V\ (0.943-0.00075\ T)}{0.9017\ W}$$

where V=volume of liberated oil, in milliliters.
T=temperature at which reading is made, in degrees centigrade.
W=weight of meal used, in grams.

Table 11 shows the analyses of 10 samples of flaxseed by the modified Babcock procedure. The results are compared with those obtained by the ether-extraction method.

Table 11.—*Comparison of the centrifugal method and the petroleum-ether extraction method for determining the oil content of 10 samples of flaxseed*

[All data are averages of duplicate determinations]

Sample no.	Oil by centrifugal method	Oil by petroleum-ether extraction method	Difference	Sample no.	Oil by centrifugal method	Oil by petroleum-ether extraction method	Difference
	Percent	*Percent*			*Percent*	*Percent*	
1	36.94	36.47	+0.47	37	38.01	37.83	+.18
2	37.44	37.74	−.30	47	34.74	35.37	−.63
3	37.32	37.48	−.16	60	42.20	41.40	+.80
4	39.68	40.05	−.37	71	33.38	33.08	+.30
11	42.71	43.01	−.30	72	34.50	34.49	+.01

From a consideration of the data and observations made regarding the carrying out of the method, it appears that the centrifugal method should be suitable for the determination of the oil content of flaxseed under conditions where a tolerance of ±1 percent of oil is allowable. As compared with the petroleum-ether extraction

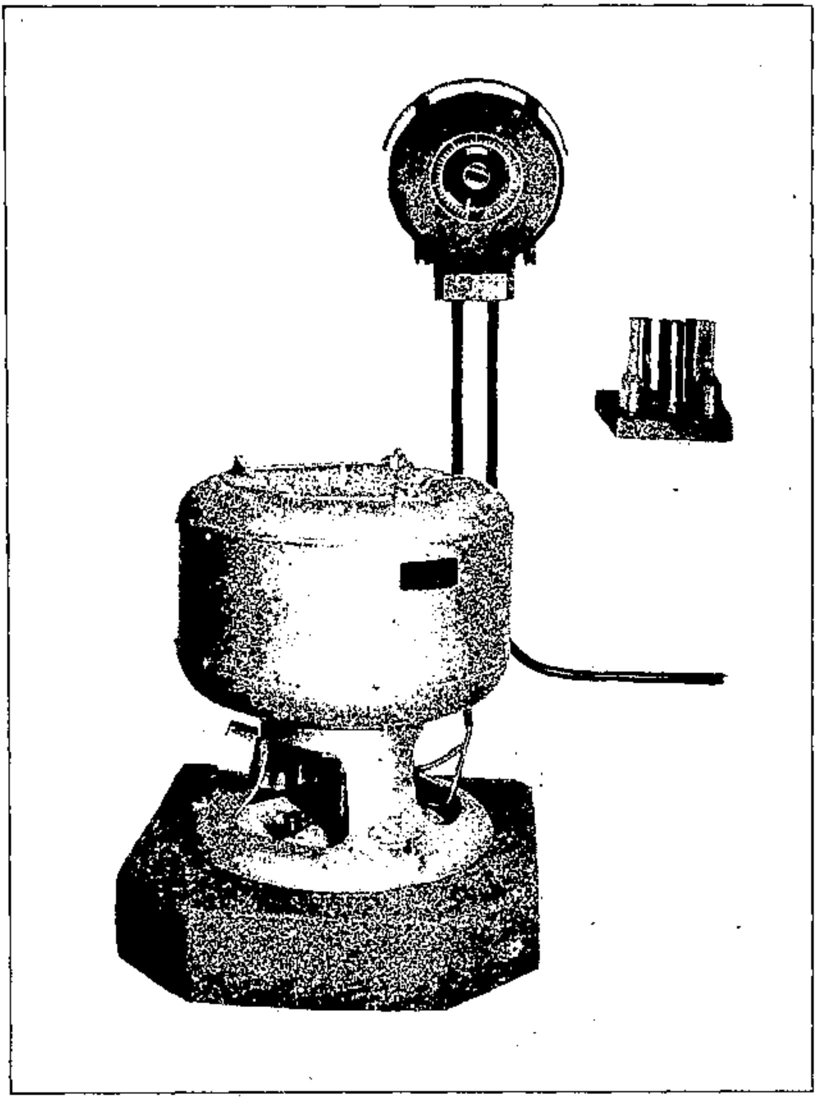

FIGURE 8.—Centrifuge for determining the oil content of flaxseed by the centrifugal method.

method, it has the advantages of speed and simplicity of operation. With a centrifuge accommodating eight Babcock bottles (fig. 8), the analyst can easily make eight analyses per hour. Further study may lead to refinements that will increase the accuracy of the method.

THE REFRACTOMETRIC METHOD

DEVELOPMENT OF THE METHOD

Wesson (*25*) was able to determine the oil content of cottonseed meal and meats by determining the refractive index of a halowax (α-chloronaphthalene) extract, the method being based on the fact that the refractive index of a mixture of cottonseed oil with halowax bears a linear relationship to the percentage of cottonseed oil in the mixture.

Coleman and Fellows (*4*) used this principle in the development of a method for the determination of the oil content of flaxseed. The method consists in mixing definite quantities of halowax and the finely ground seed in a mortar, thoroughly macerating the mixture to extract the oil, filtering the mixture, and determining the refractive index of a small quantity of the clear filtrate. From a previously prepared conversion table the percentage of oil in the seed corresponding to the refractive index of the mixture may be read. A large number of flaxseed samples, as well as samples of linseed cake, were analyzed by this procedure, the results showing good agreement with those obtained by ether extraction.

The method has been criticized adversely on the grounds that the variation in refractive index of the oils from different lots of flaxseed will be sufficient to render the results unreliable, since, to be exact, the method necessarily assumes a constant value for the refractive index of the oils from all samples of flaxseed. This objection has been emphasized during the recent drought years when oils of decidedly abnormal refractive indices have been produced because of unfavorable growing conditions. It also should be noted that during these same years new commercial varieties of flax have been introduced, the seeds of which yield oils having refractive indices quite different from those of oils from the older varieties. A careful study of the refractometric method, therefore, has been made to determine the probable magnitude of these errors and to develop, if necessary, adequate means of compensating for them.

Geddes and Lehberg (*7*) have modified the method by employing a mixture consisting of approximately equal parts by volume of halowax and α-bromonaphthalene as a solvent rather than halowax alone. This makes it possible to adjust the refractive index of the solvent accurately to a predetermined value, thus eliminating the necessity of preparing a new conversion table for each new batch of halowax. These investigators also recommend the removal of moisture from the flaxseed meal before maceration with the solvent. This was accomplished either by drying the ground samples overnight in a vacuum oven at 98° to 100° C. or, more simply, by adding a small quantity of anhydrous sodium sulphate to the sample before maceration.

Modifications of the refractometric method have also been developed by Rasteryaev (*22*) for the determination of oil in various oil-bearing seeds, using chloroform as a solvent; by Groenhof (*8*) for the estimation of oil in copra, using benzyl alcohol and tetrahydronaphthalene as solvents; and by Ilarionov and Demkovskiĭ (*9*) who found chlorobenzene a suitable solvent. Zander (*26*) in applying the method to oil-bearing seeds and their press cakes, used halowax to extract the oil, while Ermakov (*5*) used α-bromonaphthalene. Leithe (*10, 11, 12, 13, 14, 15*), and Leithe and Müller (*17*) have used the principle of the refractometric method in the determination of oil or fat in a variety of products including chocolate, dairy products, and soybeans. Leithe used benzine as a solvent in most of his work but later (*16*) showed bromonaphthalene to be superior for this purpose.

INFLUENCE OF VARIATION IN REFRACTIVE INDEX UPON THE ACCURACY OF THE REFRACTOMETRIC METHOD

Hopper[6] has determined the refractive indices of the oils from 3,080 samples taken from commercial carload lots of flaxseed. The refractive index values at 25° C. varied from 1.47535 to 1.47987. As the 5 years, 1930–34, during which time these samples were grown, were very unfavorable for the growing of flax, this range in refractive index is considerably greater than would be expected in normal crop years. From theoretical consideration, however, the error in the method of Coleman and Fellows due to this variation in refractive index should not be greater than ±0.5 percent of oil.

ADDITIONAL RESEARCH ON THE REFRACTOMETRIC METHOD

COLLECTION OF SAMPLES

For the further study of this method, 84 samples of flaxseed were obtained from various sources representing a great diversity of types and exhibiting a corresponding great diversity in physical and chemical characteristics. This assortment was represented by samples of the following types: (1) Domestic commercial; (2) Canadian commercial; (3) Indian commercial; (4) Argentine commercial; (5) 13 individual varieties; (6) samples grown experimentally in North Dakota, South Dakota, Minnesota, Kansas, California, Oregon, Wyoming, Arizona, Missouri, and New Jersey, and in the Province of Saskatchewan; (7) immature; (8) frost damaged; and (9) scabby.

The samples in this series showed the following ranges in physical and chemical characteristics: (1) Moisture content 4 to 16 percent, (2) oil content 32.57 to 45.66 percent (dry basis), (3) iodine number of oil (Wijs) 155.4 to 197.3, and (4) refractive index of oil at 25° C., 1.47589 to 1.48065. This range in refractive index is slightly greater than that noted for the 3,080 commercial samples previously analyzed by Hopper.

METHOD OF STUDY

These 84 samples were analyzed for oil content by the standard petroleum-ether extraction method and by the refractometric method of Coleman and Fellows. The latter method was slightly modified in that a mixture of about 74 percent of halowax and about 26 percent of α-bromonaphthalene by weight, having a refractive index of 1.63940 at 25° C., was used instead of halowax alone as a solvent. Since a mixture of the two solvents with this refractive index could readily be accurately duplicated, the necessity of preparing a new conversion table for each new batch of halowax was eliminated.

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[6] Personal communication.

The 1:1 mixture of halowax and α-bromonaphthalene as used by Geddes and Lehberg (7) appears to have no important advantages over the present mixture and is considerably more costly because of its higher content of α-bromonaphthalene. Using mixtures of this standard solvent with a standard composite sample of flaxseed oil, a conversion table was prepared as follows.

PREPARATION OF CONVERSION TABLE

A mixture of flaxseed from a variety of sources was ground and completely extracted with petroleum ether. The ether was completely removed in a vacuum oven and portions of the resulting sample were mixed with the standard halowax, α-bromonaphthalene solvent in carefully determined proportions. The refractive indices of these mixtures at 25° C. are shown in the following tabulation:

Percent oil in mixture:	n_D^{25}
0.000	1.63940
4.877	1.62861
9.713	1.61853
11.537	1.61485
13.360	1.61114
15.610	1.60656
23.972	1.59062
100.000	1.47780

The percentage of oil in the mixture obtained in the actual analysis of the flaxseed may be calculated by the formula:

$$\frac{100Wx}{W'+Wx} = \text{percentage of oil in the mixture}$$

Where W = weight of ground flaxseed in grams
W' = weight of solvent in grams
x = weight of oil in grams in 1 g of flaxseed

Using this formula it may be shown that a range in the oil content of flaxseed of 30 to 45 percent will correspond to a range in the oil content of the solvent-oil mixture of approximately 10 to 15 percent, when the recommended ratio of ground seed to solvent is used (2 g of ground seed to 4 ml of solvent). Over this range the oil content of the solvent-oil mixture bears an essentially linear relationship to the refractive index, each increment of 1 percent in oil content corresponding to an increment of 0.002030 in refractive index at 25° C.

Thus the refractive index at 25° C. of the solvent-oil mixture may be calculated for any value of the oil content of the seed by the formula:

$$n_D^{25} = 1.61853 - 0.002030\left(\frac{100Wx}{W'+Wx} - 9.713\right)$$

Table 16 shows the refractive indices of the mixtures corresponding to flaxseed oil contents ranging from 28 to 46 percent.

CORRECTING THE REFRACTOMETRIC METHOD FOR DIFFERENCES IN REFRACTIVE INDEX OF THE OILS

A comparison of the analytical results obtained by the refractometric and petroleum ether methods on the 84 samples of flaxseed show variations between the two methods of from −0.66 to +0.53

percent of oil. To determine to what extent these variations are due to differences in the refractive indices of the oils themselves, refractive index readings were taken on the petroleum ether extracts. In nearly every case low results in the oil-content analyses were associated with high refractive indices and vice versa, indicating that the variability in refractive index of the oils constitutes an important source of error.

To compensate for these errors, a table of corrections has been computed (table 17) indicating the values to be added or subtracted from the percentages obtained from the standard conversion table (table 16) for oils of different refractive indices. These correction values should be added when the refractive index of the oil at 25° C. is greater than 1.4778 (the value for the oil used in preparing the conversion table) and subtracted when the refractive index of the oil is lower than that value.

APPLICATION OF CORRECTION

In order to apply these correction factors, samples of pure oils must be prepared for refractive index determinations. Studies were made, therefore, of several rapid methods for obtaining a satisfactory sample of pure oil. Oils were prepared from samples representing six varieties of flaxseed by each of the following methods: (1) Standard 20–24 hour petroleum ether extraction, (2) cold pressing with laboratory hydraulic press, and (3) rapid partial extraction accomplished by pouring 20 ml of petroleum ether through a paper filter in which have been placed approximately 2 g of the ground seed, driving off the solvent on a steam bath, and drying the extract for 20 minutes in an air oven at 105° C.

Refractive indices of the oils prepared by these methods are given in table 12. No significant difference between the three methods is observed. For practical reasons the rapid partial extraction method (3) appears to be most suitable where large numbers of samples are being analyzed.

TABLE 12.—*Refractive indices at 25° C. of oils prepared from 6 varieties of flaxseed as determined by 3 different methods*

Sample no.	Variety	Standard extraction	Cold press	Partial extraction	Sample no.	Variety	Standard extraction	Cold press	Partial extraction
		n_D^{25}	n_D^{25}	n_D^{25}			n_D^{25}	n_D^{25}	n_D^{25}
31	New Golden	1.47971	1.47971	1.47970	34	Walsh	1.47653	1.47657	1.47654
32	N. D. R. 114	1.47888	1.47889	1.47894	35	Bison	1.47618	1.47617	1.47621
33	Buda	1.47815	1.47816	1.47814	36	Punjab	1.47906	1.47907	1.47905

It should be noted that the difficulty experienced by Geddes and Lehberg (7) with cloudy filtrates when samples of high moisture are being analyzed has not been encountered with the present method on samples having moisture contents as high as 16.4 percent. It therefore does not appear to be necessary with the present technique to dry the ground flaxseed before or during the analysis.

Corrections for differences in the refractive index of the oils have been applied to the oil-content data as determined by the refractometric method on the 84 samples under investigation. These corrected data are tabulated in column (C), table 13. The coefficient of correlation between these data and the petroleum-ether extraction data taken as a standard is +0.993, with a standard error of prediction of ±0.26 percent of oil. The errors range from −0.30 to +0.30 percent of oil, with an average error of ±0.13 percent. Comparing these errors with the range of from −0.66 to +0.53 percent of oil, and an average error of ±0.27 percent for the uncorrected data, it may logically be concluded that in correcting for the differences in refractive index of the extracted oils the principal source of error in the refractometric method has been eliminated. The remaining experimental error is not due solely to inaccuracies in the modified refractometric method, but to a combination of these inaccuracies with those of the standard-extraction method.

TABLE 13.—*Comparison of the oil content of 84 samples of flaxseed as determined by the modified refractometric method and by the petroleum-ether extraction method*

[All data are averages of duplicate determinations]

Sample No.	Description of sample	Oil by petroleum ether extraction, dry basis (A)	Oil by petroleum ether extraction, 8 percent moisture basis (B)	Oil by modified refractometric method, 8 percent moisture basis (C)	Difference C−B
		Percent	*Percent*	*Percent*	*Percent*
108	Linota, 1935, Sheridan, Wyo.	32.57	29.96	30.03	+0.07
107	Redwing, 1935; Sheridan, Wyo.	33.72	31.02	31.04	+.02
111	Bison, 1935; Newell, S. Dak	34.65	32.15	32.07	−.08
71	Linota, 1934; Fargo, N. Dak	35.38	32.55	32.46	−.09
40	Linota	35.51	32.67	32.83	+.16
79	Linota, 1935; Moran, Kans	35.87	33.00	32.79	−.21
82	Linota, 1935; New Brunswick, N. J	36.27	33.37	33.32	−.05
32	N. D. R. 114	36.78	33.84	33.98	+.14
72	Redwing, 1934; Fargo, N. Dak	36.89	33.94	33.85	−.09
73	Buda, 1934; Fargo, N. Dak	37.04	34.08	34.21	+.13
64	Commercial, 11.3 percent H_2O, musty	37.35	34.36	34.55	+.19
39	Redwing	37.39	34.40	34.59	+.19
80	Redwing, 1935; Moran, Kans	37.79	34.77	34.87	+.10
47	Abyssinian, 1935; Davis, Calif	37.83	34.80	34.60	−.20
33	Buda	37.89	34.86	35.13	+.27
10	Domestic commercial	37.93	34.90	34.86	−.04
81	Bison, 1935; Moran, Kans	38.08	35.03	35.19	+.16
74	Rio, 1935; Fargo, N. Dak	38.20	35.14	35.12	−.02
5	Bison; Missouri	38.21	35.15	35.32	+.17
89	Domestic commercial, 1934	38.28	35.22	35.02	−.20
35	Bison	38.28	35.22	35.26	+.04
7	do	38.48	35.40	35.37	−.03
88	Domestic commercial, 1934	38.55	35.47	35.50	+.03
1	Bison; Arthur, N. Dak	38.88	35.77	35.85	+.08
66	No. 1 Canadian Western	38.90	35.79	35.57	−.22
70	Bison, 1934; Fargo, N. Dak	38.92	35.81	35.56	−.25
90	Domestic commercial, 1934	38.99	35.87	35.73	−.14
6	do	39.20	36.06	36.25	+.19
31	New Golden	39.33	36.18	35.90	−.28
84	Bison, 1935; Dickinson, N. Dak	39.44	36.28	36.35	+.07
8	Domestic commercial	39.53	36.37	36.66	+.29
87	Domestic commercial, 1934	39.56	36.40	36.57	+.17
68	No. 2 Canadian Western	39.68	36.51	36.53	+.02
93	Domestic commercial, 1934	39.73	36.55	36.76	+.21
62	Abyssinian, 1935; El Centro, Calif	39.81	36.63	36.82	+.19
48	Punjab, 1935; Shafter, Calif	39.84	36.65	36.86	+.21
51	Heavy frost damage, scabby	40.02	36.82	37.12	+.30
67	No. 1 Canadian Western, 1934	40.07	36.86	36.88	+.02
34	Walsh	40.12	36.91	36.88	−.03
2	Bison	40.15	36.94	36.78	−.16
3	Abyssinian; California	40.21	36.99	36.94	−.05
49	Punjab, 1935; Madera, Calif	40.24	37.02	36.82	−.20

TABLE 13.—*Comparison of the oil content of 84 samples of flaxseed as determined by the modified refractometric method and by the petroleum-ether extraction method*—Continued

[All data are averages of duplicate determinations]

Sample No.	Description of sample	Oil by petroleum ether extraction, dry basis (A)	Oil by petroleum ether extraction, 8 percent moisture basis (B)	Oil by modified refractometric method, 8 percent moisture basis (C)	Difference (C−B)
		Percent	*Percent*	*Percent*	*Percent*
75	No. 3 Canadian Western	40.28	37.06	36.85	−.21
37	Bolley Golden	40.33	37.10	37.18	+.08
50	Immature	40.38	37.15	37.09	−.06
38	Rio	40.56	37.32	37.20	−.12
83	Bison, 1935; New Brunswick, N. J.	40.57	37.32	37.39	+.07
85	Bison, 1935; Morris, Minn	40.59	37.34	37.45	+.11
78	Rio, 1935; Moran, Kans	40.61	37.36	37.33	−.03
76	Argentine, imported commercial	40.66	37.41	37.25	−.16
12	do	40.66	37.46	37.51	+.05
54	Punjab, 1935; Imperial, Calif	40.72	37.46	37.53	+.07
63	Abyssinian, 1935; Heber, Calif	40.77	37.51	37.56	+.05
96	Unknown	41.07	37.78	37.67	−.11
40	Punjab, 1935; Davis, Calif	41.15	37.86	38.12	+.26
98	Unknown origin, 12.4 percent moisture	41.20	37.90	37.84	−.06
99	Unknown origin	41.26	37.96	38.05	+.09
86	Domestic commercial, 1934	41.27	37.97	37.91	−.06
97	Unknown origin, 11.6 percent moisture	41.73	38.39	38.25	−.14
69	No. 4 Canadian Western	41.81	38.47	38.40	−.07
92	Domestic commercial, 1934	42.09	38.72	38.89	+.17
56	Punjab, 1935; Holtville, Calif	42.33	38.91	39.01	+.10
91	Domestic commercial, 1934	42.49	39.09	39.04	−.05
44	Punjab, 1935; Concord, Calif	42.53	39.13	39.20	+.07
45	Punjab, 1935; Rio Vista, Calif	42.63	39.22	39.42	+.20
57	Punjab, 1935; El Centro, Calif	42.65	39.24	39.10	−.14
64	Indian, 1934; Calexico, Calif	42.69	39.27	39.39	+.12
4	Punjab; California	42.70	39.37	39.19	−.18
53	Punjab, 1935; Holtville, Calif	42.80	39.43	39.25	−.18
42	Punjab, 1935; Davis, Calif	42.95	39.51	39.39	−.12
43	Punjab, 1935; Willows, Calif	42.99	39.55	39.49	−.06
77	Punjab, 1935; Yuma, Ariz	43.12	39.67	39.50	−.17
61	Punjab, 1935; Holtville, Calif	43.15	39.70	39.85	+.15
95	Unknown origin, 15.6 percent moisture	43.17	39.72	39.56	−.16
55	Punjab, 1935; Brawley, Calif	43.45	39.97	40.02	+.05
58	Punjab, 1935; Calipatria, Calif	43.51	40.06	40.13	+.07
59	Indian, 1934; Brawley, Calif	43.55	40.07	40.21	+.14
41	Punjab, 1935; Davis, Calif	43.80	40.30	40.00	−.30
52	Punjab, 1935; Calexico, Calif	43.88	40.37	40.18	−.19
36	Punjab	43.98	40.46	40.61	+.15
100	Unknown origin, 10.4 percent moisture	44.00	40.48	40.38	−.10
65	Indian, 1934; Brawley, Calif	44.22	40.68	40.83	+.15
60	Indian, 1934; Holtville, Calif	44.37	40.82	40.93	+.11
11	Indian, imported commercial	45.66	42.01	41.84	−.17

COLLABORATIVE STUDY OF THE REFRACTOMETRIC METHOD

As a further means of checking the accuracy of the modified refractometric method, 10 samples of flaxseed, each representing a single variety, were analyzed by five laboratories. The results obtained, together with the results on the same series of samples by the standard petroleum-ether extraction method as obtained by the same laboratories, are shown in table 14. In each case the results are compared with those obtained by the standard extraction method in the laboratory of the Grain Division.

TABLE 14.—*Comparison of the oil content of 10 samples of flaxseed, representing 10 individual varieties, as determined by 5 different laboratories, by the modified refractometric method and by the standard petroleum-ether extraction method*

[All data are averages of duplicate determinations]

MODIFIED REFRACTOMETRIC METHOD

Sample no.	Variety	Laboratory A	Laboratory B	Laboratory C	Laboratory D	Laboratory E	Average	Check[1]	Average − check
		Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*
31	New Golden	36.65	37.07	36.89	36.85	36.66	36.80	36.85	−0.05
32	N. D. R. 114	34.74	34.64	34.42	34.81	34.72	34.67	34.57	+.10
33	Buda	35.33	35.25	35.01	35.55	35.70	35.37	35.43	−.06
34	Walsh	37.73	37.62	37.63	37.95	37.56	37.70	37.59	+.11
35	Bison	36.63	36.00	36.05	35.90	35.72	35.94	35.68	+.26
36	Punjab	40.77	41.20	40.45	40.54	41.23	40.85	41.08	−.23
37	Bolley Golden	37.60	37.85	37.74	37.16	37.90	37.67	37.83	−.16
38	Rio	37.75	38.78	38.16	37.69	37.85	38.05	37.96	+.09
39	Redwing	35.13	34.40	34.92	34.36	35.23	34.83	35.03	−.20
40	Linota	33.34	32.77	34.16	33.70	33.51	33.50	33.34	+.16

STANDARD PETROLEUM—ETHER EXTRACTION METHOD

Sample no.	Variety	Laboratory A	Laboratory B	Laboratory C	Laboratory D	Laboratory E	Average	Check[1]	Average − check
31	New Golden	37.09	36.78	37.93	36.40	36.85	37.01	36.85	+0.16
32	N. D. R. 114	34.08	34.41	35.77	34.18	34.57	34.78	34.57	+.21
33	Buda	35.71	35.26	36.78	35.21	35.43	35.68	35.43	+.25
34	Walsh	37.73	37.29	38.78	37.01	37.59	37.68	37.59	+.09
35	Bison	35.82	35.55	36.39	35.37	35.68	35.76	35.68	+.08
36	Punjab	40.51	41.24	40.49	41.17	41.08	40.90	41.08	−.18
37	Bolley Golden	37.82	37.63	39.50	37.07	37.83	38.21	37.83	+.38
38	Rio	37.66	38.28	39.31	37.31	37.96	38.11	37.96	+.15
39	Redwing	35.20	35.01	36.41	34.61	35.03	35.25	35.03	+.22
40	Linota	33.41	33.63	34.83	33.32	33.34	33.69	33.34	+.35

[1] Determined by the standard petroleum-ether extraction method in the Grain Division Laboratory.

A statistical study of these data yields the results shown in table 15.

TABLE 15.—*Statistical analysis of data in table 14*

	Standard petroleum-ether extraction method	Modified refractometric method
	Percent	*Percent*
Standard deviation between laboratories	1.00	0.19
Experimental error	.35	.33
Average−check (10-sample average)	.21	.14

DESCRIPTION OF MODIFIED REFRACTOMETRIC METHOD

EQUIPMENT

The equipment includes one motor-driven experimental roller flouring mill with 6- by 6-inch steel rolls, corrugated 40 to the inch. The rolls should have a speed differential of about 9:7, and a speed of about 900 revolutions per minute for the faster roll (fig. 7).

One analytical balance.

One electric hot plate.

One refractometer with water-jacketed prisms having an accuracy of $n = \pm 0.00002$ within the ranges of 1.475 to 1.482 and 1.606 to 1.640. A suitable type of refractometer with interchangeable prism heads is shown in figure 9.

One temperature-regulating device for controlling the temperature of the water flowing through the refractometer jackets (optional).

One electric oven.
One accurately calibrated 5-ml pipette.
Halowax.
α-bromonaphthalene.
Ethyl alcohol for cleaning prisms.
Three-inch porcelain mortars with pestles.
Reagent-quality sea sand, or equivalent.
Supply of test tubes, 1½-inch glass funnels, folded filter papers, and absorbent cotton.

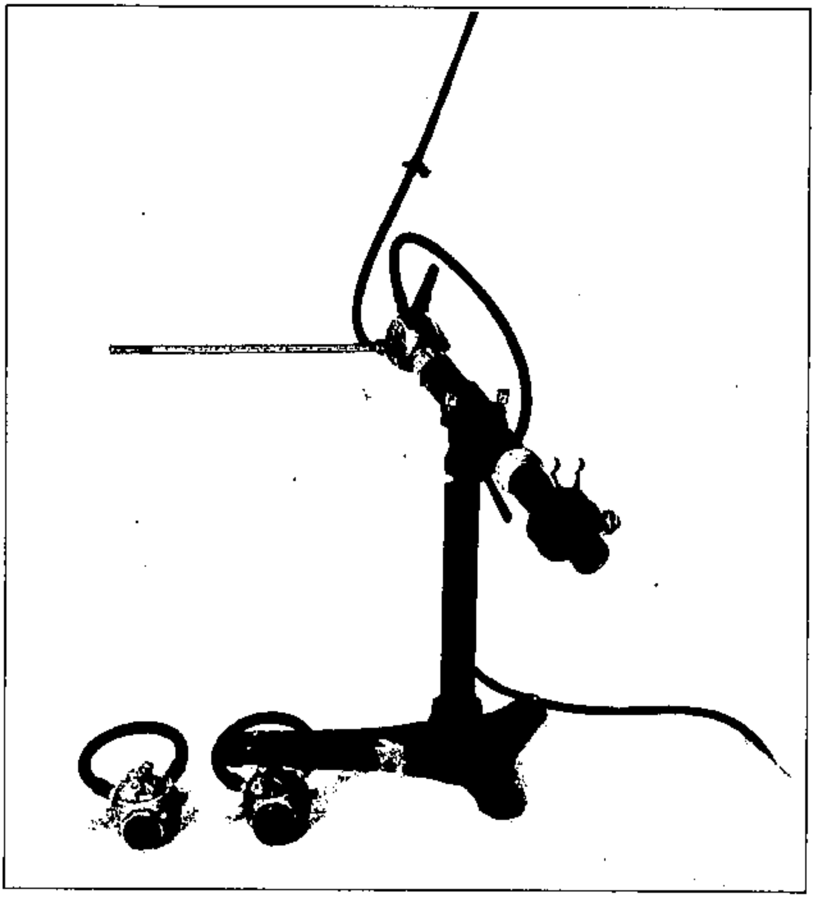

FIGURE 9.—Dipping-type refractometer with interchangeable double-prism heads suitable for the refractometric determination of the oil content of flaxseed and the iodine number of flaxseed oils.

Although the experimental error between the two methods did not differ significantly, the much lower standard deviation between laboratories in the case of the modified refractometric method indicates that this method will lead to more concordant results between different laboratories than will the extraction method when the analyses are carried out without central supervision. The results obtained by the refractometric method agree with the accepted check values somewhat better, on the average, than do the results obtained by the extraction method. The data obtained by these five laboratories, then, show a greater degree of accuracy and reliability for the modified refractometric method than for the standard petroleum-ether extraction method.

With a little experience, the analyst should be able to make a single determination by the modified refractometric method in 25 to 30 minutes as compared with the 16 to 24 hours generally required by the ether-extraction method. With the aid of two nontechnical assistants, it should be possible for the analyst to make 100 determinations in an 8-hour day, using a single set of equipment. The method is well adapted for use in commercial inspection work where an accurate method is required and where the time element is of major importance.

PREPARATION OF THE STANDARD SOLVENT

Prepare a mixture of halowax and α-bromonaphthalene having a refractive index $n_D^{25} = 1.63940 \pm 2$. Such a mixture contains approximately 74 percent of halowax and 26 percent of α-bromonaphthalene by weight but must be carefully adjusted so that the desired refractive index is attained. If a temperature-regulating device is available the determination of refractive index is simplified by passing water at exactly 25.0° C. through the water jacket of the refractometer. Equally satisfactory results may be obtained, however, by using water at room temperature and making the necessary temperature correction. For the above mixture this correction in refractive index is 0.00045 per 1°, to be added to the reading if the temperature is above 25.0° and subtracted if the temperature is below that point. It is important that all water-jacket temperature readings be made to the nearest 0.1°.

This solution should keep for a long period of time without perceptible change in refractive index, but it is advisable to check the solution from time to time. The solution should be kept in a glass- or lead-stoppered dark bottle and kept away from direct sunlight. Until the operator has had considerable experience in the use of the refractometer it is advisable to make several readings for each refractive index determination, taking an average value for the final result.

ANALYTICAL PROCEDURE

The analytical procedure follows.

(1) Obtain a representative sample of about 25 g of the clean seed either by hand quartering or by use of a mechanical sampling device.

(2) Pass the sample through the roller mill using the same precautions as noted in the instructions for the petroleum-ether extraction method.

(3) Weigh out accurately 2.5 g of the finely ground, well-mixed sample and transfer the weighed sample into a clean 3-inch porcelain mortar which has been previously heated to approximately 70° C. in an oven or on an electric hot plate at low heat.

(4) Add approximately 1 g of reagent-quality sea sand or similar abrasive and exactly 5 ml of the standard halowax, α-bromonaphthalene mixture. Since this mixture has a very high specific gravity it is highly important to measure its volume very accurately. This is best accomplished with an accurately calibrated 5-ml pipette having a delivery time of not less than 15 seconds.

(5) Grind the mixture in the mortar vigorously for 3 minutes, constantly scraping into the bottom the particles of meal that are thrown against the sides of the mortar.

(6) Filter the mixture through a Schleicher & Schull no. 588 folded filter paper, or other fat-free filter paper which will yield a clear filtrate, into a test tube.

(7) When the filtrate has cooled to room temperature, determine its refractive index at 25.0° C. to an accuracy of ±0.00002. If the reading is made at any temperature other than 25.0° make a temperature correction as described in the instructions for the preparation of the standard solvent, using a temperature coefficient of 0.00042 per 1°.

(8) Using table 16, note the percentage of oil corresponding to the refractive index reading obtained in (7). This is the uncorrected value for oil content.

(9) Place about 2 g of the ground sample in a fine paper filter in a glass funnel and pour over it about 15 ml of petroleum ether, collecting the clear filtrate in a small shallow evaporating dish. Carefully evaporate off the ether on a steam bath or hot plate at low heat, and place the dish in an oven at 105° C. for 20 minutes. Cool the oil thus prepared to room temperature and determine its refractive index at 25.0°. The temperature coefficient for the pure oil is 0.000357 per 1.0°, to be added if the temperature at which the reading is taken is above 25.0°, and subtracted if below that temperature. If preferred, this sample of oil may be prepared by pressing a small sample of the ground seed in a laboratory hydraulic press and filtering the oil so obtained if it is not entirely clear.

(10) From the refractive index of the oil as determined in (9) subtract the value 1.47780 (the refractive index at 25.0° C. of the composite sample of oil used in obtaining the data for table 16). Using this difference, determine from table 17 the correction to be applied to the uncorrected value for oil content as determined in (8). If the difference is positive add the correction; if negative, subtract.

SAMPLE DETERMINATION

Suppose the refractive index as determined in (7) is 1.61149 at 27.3° C.

$$n_D^{27.3}=1.61149$$
$$n_D^{25}=1.61149+[(27.3-25.0)\times 0.00042]$$
$$=1.61246$$

Referring to table 16:

$n_D^{25}=$ 1.61246 corresponds to an oil content of 37.55 percent. This is the uncorrected value.

Then suppose the refractive index of the oil as determined in (9) is 1.47960 at 23.3° C.

$$n_D^{23.3}=1.47960$$
$$n_D^{25}=1.47960-[(25.0-23.3)\times 0.000357]$$
$$=1.47899$$

Then:

$$1.47899-1.47780=+0.00119$$

Referring to table 17:

A difference of 0.00119 between the refractive index of the oil in the sample under investigation and the sample used in preparing the conversion table indicates a correction of 0.26 percent of oil for a sample containing approximately 38 percent of oil. Since the difference is positive the correction is to be added to the uncorrected value.

37.55 percent + 0.26 percent = 37.81 percent oil

TABLE 16.—*Conversion table for determining the percentage of oil in flaxseed from the refractive index of the halowax, α-bromonaphthalene extract at 25° C.*

n_D^{25}	Oil	n_D^{25}	Oil	n_D^{25}	Oil	n_D^{25}	Oil
	Percent		*Percent*		*Percent*		*Percent*
1.61837	28.0	1.61554	32.5	1.61279	37.0	1.61012	41.5
1.61831	28.1	1.61548	32.6	1.61273	37.1	1.61006	41.6
1.61824	28.2	1.61542	32.7	1.61267	37.2	1.61000	41.7
1.61818	28.3	1.61535	32.8	1.61261	37.3	1.60995	41.8
1.61811	28.4	1.61529	32.9	1.61255	37.4	1.60989	41.9
1.61805	28.5	1.61523	33.0	1.61249	37.5	1.60983	42.0
1.61799	28.6	1.61517	33.1	1.61243	37.6	1.60977	42.1
1.61792	28.7	1.61511	33.2	1.61237	37.7	1.60971	42.2
1.61786	28.8	1.61504	33.3	1.61231	37.8	1.60966	42.3
1.61779	28.9	1.61498	33.4	1.61225	37.9	1.60960	42.4
1.61773	29.0	1.61492	33.5	1.61219	38.0	1.60954	42.5
1.61767	29.1	1.61486	33.6	1.61213	38.1	1.60948	42.6
1.61760	29.2	1.61480	33.7	1.61207	38.2	1.60942	42.7
1.61754	29.3	1.61473	33.8	1.61201	38.3	1.60937	42.8
1.61748	29.4	1.61467	33.9	1.61195	38.4	1.60931	42.9
1.61742	29.5	1.61461	34.0	1.61189	38.5	1.60925	43.0
1.61735	29.6	1.61455	34.1	1.61183	38.6	1.60919	43.1
1.61729	29.7	1.61449	34.2	1.61171	38.7	1.60913	43.2
1.61723	29.8	1.61443	34.3	1.61177	38.8	1.60908	43.3
1.61716	29.9	1.61437	34.4	1.61165	38.9	1.60902	43.4
1.61710	30.0	1.61431	34.5	1.61159	39.0	1.60896	43.5
1.61704	30.1	1.61424	34.6	1.61153	39.1	1.60890	43.6
1.61697	30.2	1.61418	34.7	1.61147	39.2	1.60884	43.7
1.61691	30.3	1.61412	34.8	1.61141	39.3	1.60879	43.8
1.61685	30.4	1.61406	34.9	1.61135	39.4	1.60873	43.9
1.61679	30.5	1.61400	35.0	1.61130	39.5	1.60867	44.0
1.61672	30.6	1.61394	35.1	1.61124	39.6	1.60861	44.1
1.61666	30.7	1.61388	35.2	1.61118	39.7	1.60856	44.2
1.61660	30.8	1.61382	35.3	1.61112	39.8	1.60850	44.3
1.61653	30.9	1.61376	35.4	1.61106	39.9	1.60844	44.4
1.61647	31.0	1.61370	35.5	1.61100	40.0	1.60839	44.5
1.61641	31.1	1.61363	35.6	1.61094	40.1	1.60833	44.6
1.61635	31.2	1.61357	35.7	1.61088	40.2	1.60827	44.7
1.61628	31.3	1.61351	35.8	1.61082	40.3	1.60821	44.8
1.61622	31.4	1.61345	35.9	1.61076	40.4	1.60816	44.9
1.61616	31.5	1.61339	36.0	1.61071	40.5	1.60810	45.0
1.61610	31.6	1.61333	36.1	1.61065	40.6	1.60804	45.1
1.61604	31.7	1.61327	36.2	1.61059	40.7	1.60799	45.2
1.61597	31.8	1.61321	36.3	1.61053	40.8	1.60793	45.3
1.61591	31.9	1.61315	36.4	1.61047	40.9	1.60787	45.4
1.61585	32.0	1.61309	36.5	1.61041	41.0	1.60782	45.5
1.61579	32.1	1.61303	36.6	1.61035	41.1	1.60776	45.6
1.61573	32.2	1.61297	36.7	1.61029	41.2	1.60770	45.7
1.61566	32.3	1.61291	36.8	1.61024	41.3	1.60764	45.8
1.61560	32.4	1.61285	36.9	1.61018	41.4		

TABLE 17.—*Corrections to be applied to results obtained in the analysis of flaxseed for oil content by the refractometric method*

[Values to be added when ($n_D^{25}-1.4778$) is positive, subtracted when ($n_D^{25}-1.4778$) is negative]

(Corrections in terms of percent of oil indicated)

$n_D^{25}-1.4778$	28[1]	29[1]	30[1]	31[1]	32[1]	33[1]	34[1]	35[1]	36[1]	37[1]	38[1]	39[1]	40[1]	41[1]	42[1]	43[1]	44[1]	45[1]	46[1]	47[1]	48[1]
0.0001	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.03	0.03	0.03	0.03	0.03
.0002	.03	.03	.03	.03	.04	.04	.04	.04	.04	.04	.04	.04	.05	.05	.05	.05	.05	.05	.05	.05	.06
.0003	.05	.05	.05	.05	.05	.06	.06	.06	.06	.06	.06	.07	.07	.07	.07	.07	.08	.08	.08	.08	.08
.0004	.06	.06	.07	.07	.07	.07	.08	.08	.08	.08	.09	.09	.09	.09	.10	.10	.10	.10	.11	.11	.11
.0005	.08	.08	.08	.09	.09	.09	.10	.10	.10	.11	.11	.11	.11	.12	.12	.12	.13	.13	.13	.14	.14
.0006	.09	.10	.10	.10	.11	.11	.11	.12	.12	.13	.13	.13	.14	.14	.15	.15	.15	.16	.16	.16	.17
.0007	.11	.11	.12	.12	.12	.13	.13	.14	.14	.15	.15	.16	.16	.17	.17	.17	.18	.18	.19	.19	.20
.0008	.12	.13	.13	.14	.14	.15	.15	.16	.16	.17	.17	.18	.18	.19	.19	.20	.20	.21	.21	.22	.22
.0009	.14	.14	.15	.15	.16	.17	.17	.18	.18	.19	.19	.20	.21	.21	.22	.22	.23	.23	.24	.25	.25
.0010	.15	.16	.17	.17	.18	.19	.19	.20	.20	.21	.22	.22	.23	.24	.24	.25	.26	.26	.27	.27	.28
.0011	.17	.17	.18	.19	.20	.20	.21	.22	.22	.23	.24	.25	.25	.26	.27	.27	.28	.29	.29	.30	.31
.0012	.18	.19	.20	.21	.21	.22	.23	.24	.24	.25	.26	.27	.27	.28	.29	.30	.31	.31	.32	.33	.34
.0013	.20	.21	.22	.22	.23	.24	.25	.26	.27	.27	.28	.29	.30	.31	.31	.32	.33	.34	.35	.36	.36
.0014	.21	.22	.23	.24	.25	.26	.27	.28	.29	.29	.30	.31	.32	.33	.34	.35	.36	.37	.37	.38	.39
.0015	.23	.24	.25	.26	.27	.28	.29	.30	.31	.32	.32	.33	.34	.35	.36	.37	.38	.39	.40	.41	.42
.0016	.24	.25	.27	.28	.28	.30	.31	.32	.33	.34	.35	.36	.37	.38	.39	.40	.41	.42	.43	.44	.45
.0017	.26	.27	.28	.29	.30	.31	.32	.33	.35	.36	.37	.38	.39	.40	.41	.42	.43	.44	.45	.47	.48
.0018	.28	.29	.30	.31	.32	.33	.34	.35	.37	.38	.39	.40	.41	.42	.44	.45	.46	.47	.48	.49	.50
.0019	.29	.30	.32	.33	.34	.35	.36	.37	.39	.40	.41	.42	.44	.45	.46	.47	.48	.50	.51	.52	.53
.0020	.31	.32	.33	.34	.36	.37	.38	.39	.41	.42	.43	.45	.46	.47	.48	.50	.51	.52	.53	.55	.56
.0021	.32	.33	.35	.36	.37	.39	.40	.41	.43	.44	.45	.47	.48	.50	.51	.52	.54	.55	.56	.58	.59
.0022	.34	.35	.37	.38	.39	.41	.42	.43	.45	.46	.48	.49	.50	.52	.53	.55	.56	.57	.59	.60	.62
.0023	.35	.37	.38	.40	.41	.43	.44	.45	.47	.48	.50	.51	.53	.54	.56	.57	.59	.60	.61	.63	.64
.0024	.37	.38	.40	.41	.43	.44	.46	.47	.49	.50	.52	.54	.55	.57	.58	.60	.61	.63	.64	.66	.67
.0025	.38	.40	.42	.43	.45	.46	.48	.49	.51	.53	.54	.56	.57	.59	.61	.62	.64	.65	.67	.69	.70
.0026	.40	.41	.43	.45	.46	.48	.50	.51	.53	.55	.56	.58	.60	.61	.63	.64	.66	.68	.69	.71	.73
.0027	.41	.43	.45	.46	.48	.50	.52	.53	.55	.57	.58	.60	.62	.64	.65	.67	.69	.70	.72	.74	.76
.0028	.43	.45	.46	.48	.50	.52	.53	.55	.57	.59	.60	.62	.64	.66	.68	.69	.71	.73	.75	.77	.78
.0029	.44	.46	.48	.50	.52	.54	.55	.57	.59	.61	.63	.65	.66	.68	.70	.72	.74	.76	.77	.79	.81
.0030	.46	.48	.50	.52	.53	.55	.57	.59	.61	.63	.65	.67	.69	.71	.73	.74	.76	.78	.80	.82	.84

[1] Percent oil as determined from table 16.

DETERMINATION OF FLAXSEED OIL QUALITY

IODINE NUMBER AS A MEASURE OF OIL QUALITY

From a commercial standpoint, the principal criterion of the quality of the oil that may be pressed from a given lot of flaxseed is the rapidity with which a thin film of it will dry to a hard surface. This tendency of the oil to form a solid film on exposure to the air is due to the oxidation of the unsaturated fatty acids that are constituents of the oil molecule. The three principal unsaturated fatty acids of linseed oil are oleic, linoleic, and linolenic acids, the chemical constitution of which shows them to possess one, two, and three double bonds, respectively. Since in the oxidation or drying process each of these double bonds is capable of absorbing a given quantity of oxygen, the total number of double bonds or total degree of unsaturation is proportional to the total quantity of oxygen that the oil is potentially capable of absorbing. The rapidity with which this oxygen is absorbed, however, depends not only on the total degree of unsaturation but on the relative proportions of the three unsaturated fatty acids, since the double bonds of linolenic acid oxidize more rapidly than those of linoleic, and those of linoleic oxidize more rapidly than those of oleic.

For commercial purposes, however, iodine number, which is a measure of the total degree of unsaturation, has been used as an approximate measure of the relative drying times of linseed oils. Since oils from different samples of flaxseed vary to some extent in their proportions of the three unsaturated acids, this method is subject to some error. Except in unusual cases, however, the iodine number may be considered as a reasonably reliable measure of the relative drying time. The development of a rapid method for the determination of iodine number should, therefore, be of value in the routine analysis of flaxseed.

RELATIONSHIP BETWEEN IODINE NUMBER AND REFRACTIVE INDEX

It has long been known that a positive correlation exists between refractive index and iodine number of animal and vegetable oils in general. Lewkowitsch (*19, v. 1, p. 338*), however, after accumulating data on a large number of different oils, concluded that no definite relationship existed between these two factors. Niegemann and Kayser (*20*), on the other hand, reported such a relationship in the case of oils from flaxseed samples grown in a given region. Arnold (*1*) and Backer (*3*) have demonstrated a relationship between iodine number, saponification number, and refractive index.

Pickering and Cowlishaw (*21*) have developed the following mathematical equation to show the relationship between refractive index, iodine number, saponification number, and acid number:

$$n_D^{40}=1.4643-0.000066S-\frac{0.0096A}{S}+0.000117I$$

where

S = saponification number
A = acid number
I = iodine number

This equation was shown to apply to freshly prepared oils from various kinds of oil-bearing seeds. The relationship does not hold, however, for oils that have been prepared for an appreciable length of time, since the oxidation and polymerization which take place when the oil stands cause a marked increase in the refractive index.

The saponification number of the flaxseed oil is relatively constant for all varieties and types of seed, and the acid value of the freshly prepared oil is uniformly low except in the case of badly damaged seed. It should, therefore, be possible to determine the iodine number directly from the refractive index of the freshly prepared oil provided the oil is prepared in such a way that no appreciable amount of oxidation, polymerization, or hydrolysis can take place.

Hopper[7] has determined the refractive indices and iodine numbers of expressed oils from 1,500 samples of flaxseed and noted a very significant relationship between the two values. Geddes and Lehberg (7) obtained a correlation of +0.647 between the refractive index and iodine number of oils extracted by diethyl ether, with a standard error of prediction for iodine number of ±2.75.

A study has been made of the relationship of refractive index to iodine number of the oils extracted with petroleum ether from a series of 96 samples of flaxseed including the 84 samples described on page 21. Wijs iodine numbers were determined by the method specified by the Federal Specifications Board (*6*). The data are tabulated in columns 3 and 4 of table 18 in the order of increasing iodine number. The coefficient of correlation between the refractive index and iodine number is +0.9965 with a standard error of prediction for the iodine number of ±0.82.

TABLE 18.—*Iodine number (Wijs) and refractive index of oils from 96 samples of flaxseed*

Laboratory no.	Description of sample	Iodine number (A)	n_D^{25}	Iodine number from refractive index (B)	Difference, B−A
81	Bison, 1935; Moran, Kans.	155.4	1.47580	156.6	+1.2
74	Rio, 1934; Fargo, N. Dak.	157.0	1.47582	156.1	−.9
78	Rio, 1935; Moran, Kans.	158.1	1.47627	159.9	+1.8
35	Bison	159.3	1.47617	159.0	−.3
5	Bison; Missouri	161.5	1.47653	162.1	+.6
106	Bison, 1935; Sheridan, Wyo.	161.9	1.47657	162.5	+.6
7	Bison, 1934	162.1	1.47644	161.4	−.7
34	Walsh	162.2	1.47657	162.5	+.3
70	Bison, 1934; Fargo, N. Dak.	162.5	1.47651	162.0	−.5
84	Bison, 1935; Dickinson, N. Dak.	164.4	1.47686	165.0	+.6
4	Bison; Arthur, N. Dak.	165.5	1.47679	164.4	−1.1
79	Linota, 1935; Moran, Kans.	166.0	1.47708	166.8	+.8
85	Bison, 1935; Morris, Minn.	166.4	1.47708	166.8	+.4
38	Rio	167.9	1.47713	167.3	−.6
105	Rio, 1935; Sheridan, Wyo.	168.0	1.47713	167.3	−.7
73	Buda, 1934	170.2	1.47747	170.2	.0
83	Bison, 1935; New Brunswick, N. J., scabby seed	171.0	1.47743	169.9	−1.1
89	Domestic commercial, 1934	171.2	1.47756	171.0	−.2
93	do.	172.5	1.47779	172.9	+.4
107	Redwing, 1935; Sheridan, Wyo.	172.6	1.47775	172.6	.0
80	Redwing, 1935; Moran, Kans.	172.6	1.47790	173.9	+1.3
111	Bison, 1935; Newell, S. Dak.	172.9	1.47773	172.4	−.5
108	Linota, 1935; Sheridan, Wyo.	173.7	1.47789	173.8	+.1
6	Domestic commercial	173.9	1.47790	173.9	.0
71	Linota, 1934; Fargo, N. Dak.	174.1	1.47809	175.5	+1.4

[7] Personal communication.

TABLE 18.—*Iodine number (Wijs) and refractive index of oils from 98 samples of flaxseed*—Continued

Laboratory no.	Description of sample	Iodine number (A)	n_D^{25}	Iodine number from refractive index (B)	Difference, B−A
2	Bison	174.4	1.47790	173.0	−1.4
110	Rio, 1935; Newell, S. Dak.	174.5	1.47796	174.4	−.1
10	Domestic commercial	174.6	1.47789	173.8	−.8
4	Punjab, California	175.2	1.47809	175.5	+.3
8	Domestic commercial	175.5	1.47814	176.0	+.5
33	Buda	176.1	1.47815	176.1	.0
46	Punjab, 1935; Davis, Calif	176.1	1.47819	176.4	+.3
82	Linota, 1935; New Brunswick, N. J., scabby seed	176.2	1.47814	176.0	−.2
88	Domestic commercial, 1934	177.8	1.47819	176.4	−1.4
87	do	179.1	1.47852	179.2	+.1
65	Indian, 1934; Brawley, Calif	179.2	1.47863	180.2	+1.0
72	Redwing, 1934; Fargo, N. Dak.	179.5	1.47853	179.3	−.2
64	Indian, 1934; Calexico, Calif	179.6	1.47845	178.6	−1.0
41	Punjab, 1935; Davis, Calif	179.8	1.47859	179.8	.0
112	Redwing, 1935; Newell, S. Dak	180.0	1.47860	179.9	−.1
42	Punjab, 1935; Davis, Calif	180.1	1.47851	179.1	−1.0
48	Punjab, 1935; Shafter, Calif	180.2	1.47869	180.7	+.5
43	Punjab, 1935; Willows, Calif	180.4	1.47869	180.7	+.3
69	Indian, 1935; Brawley, Calif	180.7	1.47871	180.9	+.2
54	Punjab, 1935; Imperial, Calif	180.9	1.47877	181.4	+.5
40	Linota	181.0	1.47869	180.7	−.3
44	Punjab, 1935; Concord, Calif	181.0	1.47866	180.4	−.6
92	Domestic commercial, 1934	181.3	1.47889	182.4	+1.1
39	Redwing	181.3	1.47872	181.0	−.3
90	Domestic commercial, 1934	181.4	1.47882	181.8	+.4
60	Indian, 1934; Holtville, Calif	181.5	1.47889	182.4	+.9
32	N. D. R. 114	181.7	1.47889	182.4	+.7
37	Bolley Golden	181.8	1.47865	180.4	−1.4
11	Imported Indian, commercial	182.0	1.47897	183.1	+1.1
69	No. 4 Canadian Western	182.0	1.47900	183.3	+1.3
47	Abyssinian, 1935; Davis, Calif	182.1	1.47891	182.6	+.5
57	Punjab, 1935; El Centro, Calif.	182.4	1.47893	182.7	+.3
113	Linota, 1935; Newell, S. Dak	182.6	1.47871	181.1	−1.5
68	No. 2 Canadian Western	182.8	1.47898	183.1	+.3
53	Punjab, 1935; Holtville, Calif	183.0	1.47905	183.8	+.8
101	Rio, 1935; Union, Oreg.	183.8	1.47915	184.9	+1.1
12	Imported Argentine, commercial	183.9	1.47908	183.6	−.3
78	do	184.0	1.47899	183.2	−.8
36	Punjab	184.0	1.47907	183.9	−.1
67	No. 1 Canadian Western, 1934	184.2	1.47902	183.5	−.7
61	Punjab, 1935; Holtville, Calif	184.7	1.47918	184.9	+.2
56	do	185.2	1.47912	184.4	−.8
86	Domestic commercial, 1934	185.2	1.47916	184.7	−.5
77	Punjab, 1935; Yuma, Ariz	185.5	1.47926	185.6	+.1
55	Punjab, 1935; Brawley, Calif.	185.8	1.47923	185.3	−.5
58	Punjab, 1935; Calipatria, Calif.	186.3	1.47933	186.2	−.1
52	Punjab, 1935; Calexico, Calif	186.3	1.47940	186.8	+.5
115	Bison, 1935; Saskatoon, Saskatchewan	188.3	1.47960	188.5	+.2
49	Punjab, 1935; Madera, Calif	188.5	1.47946	187.3	−1.2
66	No. 1 Canadian Western	188.5	1.47955	188.1	−.4
31	New Golden	188.6	1.47971	189.4	+.8
96	Unknown	188.6	1.47973	189.6	+1.0
114	Rio, 1935; Saskatoon, Saskatchewan	188.9	1.47949	187.5	−1.4
45	Punjab, 1935; Rio Vista, Calif	188.9	1.47957	188.2	−.7
98	Unknown	190.5	1.47971	189.4	−1.1
3	Abyssinian; California	190.5	1.47982	190.4	−.1
103	Redwing, 1935; Union, Oreg	190.7	1.47987	190.8	+.1
102	Bison, 1935; Union, Oreg.	190.9	1.47992	191.3	+.4
100	Unknown	190.9	1.47992	191.3	+.4
51	Heavy frost damage and scabby seed	191.0	1.47981	190.3	−.7
97	Unknown	191.8	1.48010	192.8	+1.0
99	do	191.9	1.48001	192.3	+.4
104	Linota, 1935; Union, Oreg.	192.7	1.48010	192.8	+.1
62	Abyssinian, 1935; Meloland, Calif.	193.2	1.48016	193.3	+.1
95	Unknown	193.4	1.48024	194.0	+.6
50	Immature	194.6	1.48017	193.4	−1.2
116	Redwing, 1935; Saskatoon, Saskatchewan	194.6	1.48030	194.5	−.1
117	Linota, 1935; Saskatoon, Saskatchewan	194.9	1.48047	196.0	+1.1
94	Domestic commercial, 1934	195.2	1.48021	193.7	−1.5
109	Redwing, 1935; Edmonton, Alberta, immature	196.0	1.48053	196.5	+.5
63	Abyssinian, 1935; Heber, Calif.	197.3	1.48065	197.5	+.2

From the regression equation—

$$\text{Iodine number} = -12513.83 + 8584.97\ n_D^{25}$$

a table has been computed (table 19) for the estimation of iodine number from refractive index.

TABLE 19.—*Conversion table for determining Wijs iodine number of freshly prepared flaxseed oil from refractive index*

[Data calculated from regression equation: $I = -12513.827 + 8584.966\ n_D^{25}$]

n_D^{25}	Iodine number	n_D^{25}	Iodine number	n_D^{25}	Iodine number	n_D^{25}	Iodine number	n_D^{25}	Iodine number
1.4733	134.4	1.4750	149.0	1.4767	163.6	1.4784	178.2	1.4801	192.8
1.4734	135.3	1.4751	149.9	1.4768	164.5	1.4785	179.0	1.4802	193.6
1.4735	136.1	1.4752	150.7	1.4769	165.3	1.4786	179.9	1.4803	194.5
1.4736	137.0	1.4753	151.6	1.4770	166.2	1.4787	180.8	1.4804	195.4
1.4737	137.8	1.4754	152.4	1.4771	167.0	1.4788	181.6	1.4805	196.2
1.4738	138.7	1.4755	153.3	1.4772	167.9	1.4789	182.5	1.4806	197.1
1.4739	139.6	1.4756	154.1	1.4773	168.7	1.4790	183.3	1.4807	197.9
1.4740	140.4	1.4757	155.0	1.4774	169.6	1.4791	184.2	1.4808	198.8
1.4741	141.3	1.4758	155.9	1.4775	170.5	1.4792	185.1	1.4809	199.6
1.4742	142.1	1.4759	156.7	1.4776	171.3	1.4793	185.9	1.4810	200.5
1.4743	143.0	1.4760	157.6	1.4777	172.2	1.4794	186.8	1.4811	201.4
1.4744	143.8	1.4761	158.4	1.4778	173.0	1.4795	187.6	1.4812	202.2
1.4745	144.7	1.4762	159.3	1.4779	173.9	1.4796	188.5	1.4813	203.1
1.4746	145.6	1.4763	160.2	1.4780	174.8	1.4797	189.3	1.4814	203.9
1.4747	146.4	1.4764	161.0	1.4781	175.6	1.4798	190.2	1.4815	204.8
1.4748	147.3	1.4765	161.9	1.4782	176.5	1.4799	191.1	1.4816	205.7
1.4749	148.1	1.4766	162.7	1.4783	177.3	1.4800	191.9	1.4817	206.5

It should be distinctly understood that this method of determining iodine number is meant to apply only to samples of oil extracted with petroleum ether or cold-pressed from freshly ground flaxseed. Immaturity, frost damage, or scabbiness of the seed do not appear to affect the accuracy of the results. Samples of flaxseed, however, that have become distinctly musty because of prolonged storage at high moisture contents often cause difficulty in analyzing for iodine number by this method. The oils from such samples have generally undergone hydrolytic changes.

The method is not dependable for the determination of iodine number of commercial linseed oils since their processing tends to alter the refractive index, but it may be used to determine the iodine number of the commercially prepared raw linseed oil that a given lot of flaxseed will produce. When the oil content of a sample of flaxseed has been determined by the modified refractometric method, no additional labor is required to determine the iodine number of the oil, since the refractive index value of the oil itself is determined and may be converted into iodine value by using table 19.

In case the iodine number of the oil is to be determined independently the following procedure may be used.

REFRACTOMETRIC PROCEDURE FOR DETERMINING IODINE NUMBER

(1) Grind a representative sample of the clean flaxseed with a suitable type mill.

(2) Prepare a sample of oil from the freshly ground seed by one of the following methods:

(*a*) Press the oil from a small quantity of the ground seed with a small laboratory hydraulic press, filtering the pressed oil if it is not clear.

(*b*) Pour about 15 ml of petroleum ether over about 2 g of the freshly ground seed in a fine filter paper fitted to a glass funnel, collecting the filtrate in a small shallow evaporating dish. Evaporate the bulk of the solvent on a steam bath and place the dish in an oven at 105° C. for 20 minutes.

(3) Determine the refractive index of the oil at 25.0° C. If the reading is taken at any other temperature, add 0.000357 for each 1.0° above 25.0° and subtract that value for each 1.0° below that temperature.

(4) Convert refractive index value into iodine number (Wijs) by use of table 19.

SPECIAL PRECAUTIONS IN USE OF THE REFRACTOMETER

(1) The refractometer should be carefully checked for accuracy before being used. For this purpose auxiliary testing prisms of known refractive index are generally supplied with the instrument. Several such auxiliary prisms should be used with the instrument in order to insure accuracy in all parts of the range. In instruments having replaceable prism heads, each head should be equipped with at least two testing prisms. If errors are detected that cannot be eliminated by readjustment of the instrument, a correction table should be prepared. It is a good practice to have the instrument periodically inspected by someone who is thoroughly familiar with optical instruments.

(2) Before the instrument is read, sufficient time must be allowed for the liquid to acquire the temperature of the prisms. The reading will remain constant only when this equilibrium is established.

(3) Clean the faces of the prisms thoroughly between determinations. This is best accomplished by wiping with dry absorbent cotton, then with cotton dipped in ethyl alcohol, and finally again with the dry cotton. Solutions containing the halowax, α-bromonaphthalene solvent should not be left on the prisms longer than necessary to make the reading, since in some refractometers the cement holding the prisms in place is corroded by this solvent.

SUMMARY

The quantity and quality of linseed oil which may be produced from a given quantity of flaxseed are subject to wide variations depending on the source of the seed. These differences in the seed are due chiefly to varietal differences and to the climatic conditions under which the seed was grown.

Methods now in common use for the determination of oil quantity and quality are too time-consuming for commercial inspection procedures. Rapid analytical methods have therefore been developed for the routine determination of the oil content and oil quality of flaxseed.

The principal sources of error in the ether-extraction method for the determination of the oil content of flaxseed have been studied. As a result an extraction procedure has been developed which eliminates these errors to a large degree, and which serves as a standard check method for use in the development of more rapid methods.

A centrifugal method for the rapid determination of oil content has commercial possibilities under conditions where an accuracy of ±1-percent oil is sufficient. The method is very simple in operation and requires relatively simple equipment. Approximately eight determinations may be made in an hour, by one analyst. The method is not recommended when a high degree of accuracy is required.

The refractometric method originally proposed by Coleman and Fellows (*4*) has been perfected so as to eliminate its chief source of error. Certain features of the original method have also been simplified so that the average analyst may quickly master the technique. A high degree of accuracy may be attained by the modified method, as evidenced by the fact that in the analysis of 84 samples of flaxseed, covering a wide variety of types, by both the refractometric method and the petroleum-ether extraction method, a correlation coefficient of +0.993 was obtained with a standard error of prediction of ±0.26 percent of oil for the modified refractometric method. Results obtained by collaborative study show a greater degree of accuracy and reliability for the modified refractometric method than for the standard petroleum-ether extraction method.

A single analysis by the modified refractometric method can be made in about 30 minutes, as compared with the 16 to 24 hours generally required for the extraction method. Approximately 100 analyses may be made in an 8-hour day with a single set of equipment by one analyst with the aid of two nontechnical assistants.

The refractive index of flaxseed oil prepared by special methods may be used as a direct measure of iodine number, the latter value being now used commercially as an approximate measure of the drying quality of the oil. In the analysis of oils from 96 samples of flaxseed, with iodine numbers (Wijs) ranging from 155 to 197, a correlation coefficient of +0.9965 between iodine number and refractive index was found, with a standard error of prediction of iodine number of ±0.82. When the oil content has been determined by the proposed refractometric method no additional labor is involved in determining the iodine number refractometrically.

For the purpose of routine analysis of flaxseed the refractometric methods for the determination of oil content and iodine number (Wijs) should yield results essentially as accurate as those obtained by the conventional procedures, and at the same time effect a substantial saving in time.

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