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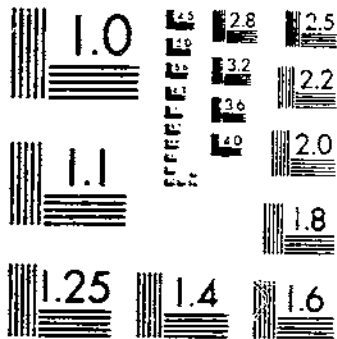
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RAPID TESTING OF OILSEEDS FOR OIL QUANTITY AND IODINE NUMBER OF OIL

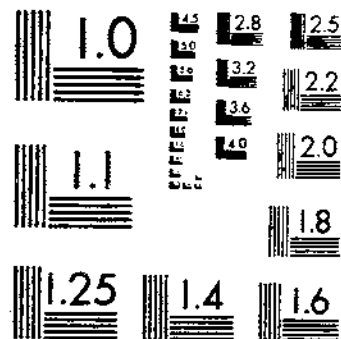
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Rapid Testing of

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Technical Bulletin No. 1171

UNITED STATES DEPARTMENT OF AGRICULTURE

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SUMMARY

The production of vegetable oils from oil-bearing seeds is a large industry in the United States and has brought about a growing market for oilseed crops. These vegetable oils are used in the manufacture of both edible and industrial products, such as oleomargarine and paint.

Since most oilseed crops are grown primarily for their intrinsic oil value, it is highly desirable that such seeds be marketed on the basis of the quantity and quality of their oil content. In the past this has not been practical because rapid analytical methods have not been available. This report reviews the older analytical methods and proposes new methods.

It was found that one of the most practical means of determining oil content is the dielectrometric method. This method and conversion tables for translating meter readings into percentage of oil are presented for flaxseed, soybeans, sunflower seed, and safflower seed. The method should be equally applicable to other oil-bearing seeds.

The quality of oil used for paint can be largely determined by its drying properties

which, in turn, are estimated by the degree of unsaturation of the glycerides which constitute the oil. This property of unsaturation is generally evaluated chemically by the determination of the iodine number of the oil.

Iodine number is defined as the number of grams of iodine which will be absorbed by 100 grams of oil.

This report shows the practicability of the refractometric method of determining iodine numbers. With the introduction of a simple, inexpensive, iodine-number refractometer, a rapid determination of iodine number of the oil in oilseeds becomes feasible. The relationship between iodine number and refractive index of freshly pressed or extracted oils from sound oilseeds is established. It would also be a simple matter to establish this relationship for other oils containing glyceride structures similar to those studied.

It has been reported that the iodine-number refractometers described here is being used at present in plant control of the hydrogenation process in the manufacture of shortenings and oleomargarine.

Issued October 1957

Rapid Testing of Oilseeds for Oil Quantity and Iodine Number of Oil¹

By M. H. Neustadt, Grain Technologist
Agricultural Marketing Service

NEED FOR RAPID OIL-TESTING METHODS

The production of vegetable oil from oil-bearing seeds is a large industry in the United States. Soybeans, flaxseed, sunflower seed, and safflower seed as well as most other oilseeds are grown primarily for the intrinsic value of their oil. For the year 1955 285 million bushels of soybeans were crushed from the total production of over 373 million bushels. Flaxseed production, although not so large, in 1955 totaled over 41 million bushels, of which more than 35 million bushels were crushed. Sunflower seed and safflower seed are relatively new crops, still unreported in USDA agricultural statistics; however, some estimates of production are available. In 1951 in the States of North Dakota and California 6,800,000 pounds of sunflower seed were produced. Safflower production for California alone in 1956 was 140 million pounds of seed.

Commercial lots of soybeans ordinarily contain from 17 to 22 percent of oil on a dry-matter basis. New varieties of sunflower and safflower are being bred for higher oil yield so that the varieties to be grown in the future will undoubtedly produce seed containing more oil. At present the range for oil content in sunflower seed is 22 to 36 percent and for safflower seed it is from 26 to 47 percent on a dry-matter basis. These values present enough of a range to be materially significant to the buyer who will process the oil-bearing seeds primarily for their oil content. A difference of 1 percent in oil content in 100 tons of seed means a difference of 1 ton of oil. When safflower oil is selling at about 16 cents per pound, as it is now, this would mean a difference in value of \$320. It is evident then that rapid, practical methods of determining oil content are desirable for trading in oilseeds.

The quality of an oil used in the paint industry—and the oils from all the seeds discussed in this publication can be used in the paint industry—is primarily determined by the drying properties of the oil. In turn, the drying properties of the oil are estimated by the degree of unsaturation of the glycerides which constitute the oil. This property of unsaturation is generally evaluated chemically by the determination of the iodine

¹ Some of the results reported in this paper were derived from studies made under authority of the Agricultural Marketing Act of 1946 (RMA Title II).

number of the oil. Iodine number is defined as the number of grams of iodine which will be absorbed by 100 grams of oil. In general, the higher the iodine number of an oil, the more suitable it is for paint and varnish making, and, conversely, the lower the iodine number of an oil, the more desirable in certain respects it is for edible use because low-iodine-number oils resist rancidification and are easier to convert by hydrogenation to a solidified fat. The oils offer enough of a range in iodine number (table 1) to make it worthwhile to have rapid, practical methods of determining this factor.

TABLE 1.—*Iodine Number of Oils in Oilseeds*

Oilseed	Iodine No. (Wijé)
Soybeans.....	(Range) 128-138
Flaxseed.....	155-200
Sunflower seed.....	125-136
Safflower seed.....	130-152

DETERMINATION OF OIL CONTENT

Extraction Method

Except those for soybeans and flaxseed, there are no nationally accepted official methods for determining oil content of the oil-bearing seeds covered in this study. The American Oil Chemists' Society (20) ² prescribes a method for soybeans which involves grinding the sample in an attrition mill, placing it in a Butt tube extraction apparatus with petroleum ether for two hours, regrinding in a mortar and again extracting for three hours. Zeleny and Neustadt (30) found that equivalent results could be obtained by grinding the sample in an experimental-type roller flour mill and extracting 16-18 hours in a Soxhlet apparatus. This latter method appears to be a simpler way of accomplishing the same results.

Zeleny and Coleman (29) developed a standard petroleum-ether extraction method, for determining oil content of flaxseed, which has been in use for several years and has proved satisfactory. The method is quite similar to one just described for soybeans. The tentative method prescribed by the American Oil Chemists' Society (20), for flaxseed oil content is also essentially similar to this method.

An entirely new approach toward grinding oil-bearing seeds was developed by Johnson, et al. (12). The mill in this case, described subsequently as the high-speed grinder-extractor (fig. 1), consists of a motor-driven, high-speed vertical shaft and propeller blade, rotating

² Numbers in parentheses refer to literature citations page 25.

within a cup in which the sample to be ground is confined. With this mill as a basis, standard methods for determining the oil content of safflower seed and sunflower seed were developed. An outline of the method for safflower seed follows:

1. Reduce the size of a dockage-free sample to approximately 50-75 g. by use of a mechanical device, such as the Boerner Divider, or by hand quartering.

2. Place the sample in the cup of the high-speed mill, and grind for 15 seconds.

3. Scrape the ground material from the sides and bottom of the cup with a spatula, and grind again for 15 seconds.

4. Transfer the ground sample to a capped sample jar, and mix thoroughly.

5. Samples should be weighed and extraction begun immediately after grinding to insure reproducible results. Weigh duplicate 10.0 gm. portions, transfer quantitatively to the extraction thimbles, and cover with wads of fat-free cotton.

6. Extract with Skellysolve F in a Soxhlet extractor for at least 16 hours.

7. Completely evaporate the solvent on a steam bath by agitating the oil-solvent mixture with a stream of air while under reduced pressure. Cool to room temperature and weigh.

Sunflower seed, which is larger than safflower seed, cannot be ground fine enough in the high-speed mill; therefore, a burr mill was used. Otherwise the procedure for analyzing sunflower seed for oil content after grinding is the same as for safflower seed (step 4).

The official and standard methods for determining oil content of oil-bearing seeds described are very satisfactory methods. However, they are time consuming, requiring from 6 to 18 hours. Because they are lengthy, these methods would be unacceptable for routine inspections, and for that purpose there has been a continuous effort to devise simple, rapid, and yet accurate methods for determining oil content. The standard methods have served as a basis and as a norm with which to compare rapid methods.

Rapid Extraction Methods

Werr (27) described a method for determining oil content by grinding seeds in a mortar with sand and anhydrous sodium sulfate. The ground sample is then shaken with benzene in a centrifuge tube and centrifuged. A 1-ml. aliquot of the supernatant liquid is slowly transferred to a weighed filter paper suspended above a hot plate and dried for 30 minutes, then weighed. The average specific gravity of such a solvent-oil mixture was used for calculations. It is evident that this procedure is laborious and requires about 1 hour to complete the determination.

Kobe (15) also utilized the centrifuge method; however, he used a mill-ground sample and the entire extract (3 or 4 extractions) was evaporated to dryness and weighed. Another aspect of the centrifuge method was

investigated by Lewis (17) in application to pecans and by Zeleny and Coleman (30) to flaxseed. Instead of using organic solvents for extraction, they utilized the Babcock method for butterfat content of dairy products by treating the ground oilseed with sulfuric acid and measuring volumetrically the amount of fat released. When an accuracy of ± 1 percent of oil is allowable as many as 8 determinations of the oil content of flaxseed can be made in 1 hour.

Micaeli and Desnuelle (21) devised a separatory funnel with a sintered glass disc above the stopcock. Samples are extracted in the funnel by shaking and then filtering with suction. They claimed good results with two extractions for low-oil-content samples but not for the higher ones.

When the blender-type mincer came into use as a homogenizer for animal and vegetable tissues, many scientific workers recognized the possibility of applying this device to the extraction of oil from oilseeds. Many fat extraction methods utilizing one of these devices were developed. Among these we find one by Kennedy and Unrau (11) who described a method for determining the oil content of safflower and sunflower seed. Pinto and Enas (24) applied the blender to copra and to oilseeds and Francois and Bleicher (3) worked with rapeseed, peanuts, soybeans, sunflower seed and palm kernels. Hamilton and Gilbert (4) used the blender for tung kernels.

All of these workers used the blenders to grind and extract, or simply to extract the oil from the ground sample with an organic solvent and then to determine gravimetrically the fat content of the extract. Some of the results reported did not appear to show much precision, for reasons which seem to be obvious. The Grain Division laboratories thoroughly investigated this method and found a lack of precision because leakage of the solvent-oil mixture occurred through the bearings of the cutting assembly and through the gaskets holding the cutting assembly in the container. The leakage does not occur with the first few samples but subsequent use leads to a gradual dissolution of the grease in the "oilite" bearings and a swelling of the rubber gaskets. The latter difficulty may be overcome by use of solvent-resistant gaskets.

Specific Gravity Methods

The methods of oil determination previously described are based on gravimetric or volumetric principles. However, it is possible to measure other physical properties of oil-solvent mixtures as a means of determining oil content and thus increase the speed and ease of the determination.

Harris (5) used the specific gravity of solvent-oil mixtures as his basis for determining oil content in the mixtures. He devised a specially calibrated hydrometer which gives oil content readings directly. The hydrometer became known as the "Lipometer" (6) and it was used on solvent extracts of various oilseeds and oilseed cakes.

McKinney and Rose (18) triturated ground tung fruit samples with

acetylene tetrabromide. Then they filtered the solution and determined the density of the filtrate. A reference curve was used to determine the percentage of oil in the solution. Subsequently McKinney and Holmes (19) combined a blender for extraction and specific gravity measurement of the solvent extract as a method for determining the oil content of tung products. To obtain greater precision than is possible with the "Lipemeter" they determined specific gravity with a pycnometer or alternatively with a specific gravity balance.

Refractometric Methods

Refractometric methods are another approach toward utilizing a physical property of the oil in oil-bearing seeds to determine their oil content. In essence the method consists of extracting the oil in a suitable solvent of fixed refractive index and then determining the change produced in the refractive index. Wesson (28) was probably responsible for the first application of this principle to the determination of the oil content of cottonseed. Much work followed along these lines in the past 30 years. Zeleny and Neustadt (30) supply a thorough review of the refinements in the method and its application to diverse products. The method yields precise and accurate results. In fact, Seharrer and Lamel (25) have presented evidence indicating that in cases where higher results are obtained by gravimetric extraction procedures, the higher values are often due to the presence of non-fatty substances in the extracts; and, therefore, the refractometric results are more likely to be correct.

There are several factors which impede the general adoption of this rapid method. Precision 5-place refractometers which are required for this method are rather expensive. Also, much effort is required in the hand-grinding of the sample-solvent mixture in a mortar and all measurements must be made precisely. Kaloyereas and Cruess (13), in determining the oil content of olives, have surmounted one of these difficulties by using a blender. Shannon (26), in the refractometric determination of oil content of avocado has eliminated the hand-grinding procedure by utilizing a power grinder consisting of a grinding shell and steel balls.

Dielectrometric Method

Basis for the Method.—The dielectrometric method is based on the use of a high-frequency oscillator. The theory and the application of high-frequency oscillators in chemical analysis have been briefly reviewed by Hunt et al (10) in their study of the use of a dielectric method to determine the oil content of soybeans. In essence, it was found that when a solution is placed within the coil or between condenser plates of a high-frequency oscillator, it affects the characteristics of that oscillator to an extent determined by the nature and the concentration of the sample. The addition of the sample to the coil or condenser produces changes in

frequency in accordance with the formula

$$F = \frac{1}{2\pi\sqrt{LC}}$$

where: F = frequency

L = inductance of the oscillator

C = capacitance of the parallel resonant circuit

The grid current or the beat frequency change can be measured and instruments based on these measurements have been designed.

Application of High-Frequency Oscillation to Analysis of Oil-bearing Seeds for Oil Content.—As is common in all other methods for determining oil content, this method requires that the oil be separated from the seed. To perform this rapidly in a simultaneous procedure of grinding and extracting, the high-speed grinder-extractor was developed. This mill is shown in Figure 1.

It is an impact, cutter-type, grinder-extractor consisting of a rapidly rotating shaft with a shaped, sharp blade at its extremity. The spindle speed is 15,000 r. p. m., and the blade tip speed is 135,000 inches per minute. It is powered through a V belt by a 3,450 r. p. m., $\frac{1}{2}$ hp. motor. The sample and solvent are contained in an aluminum cup. The grinder-extractor is capable of grinding 100 grams of soybeans in 100 ml. of solvent so that more than 98 percent of the residual meal will pass through a U. S. Standard No. 45 woven-wire cloth sieve.

Of the many good oil solvents available for extraction work orthodichlorobenzene proved to be the most satisfactory for this method. A technical grade of orthodichlorobenzene adjusted to a dielectric constant of 7.02 was used. Since most vegetable oils have a dielectric constant of less than 3.5, the solvent offered enough difference to make it possible to read changes in dielectric constant when small increments of oil were added to the solvent.

Although there are several dielectric meters on the market, they are generalized instruments covering a wide range in dielectric constant measurement. Such instruments were unsatisfactory for the measurement of oil-solvent mixtures. Hence an electronic tester, which is now on the market, was especially designed for this purpose.³

The principle of operation of this unit is shown in the block diagram in Figure 2. The complete circuit consists of an electronic oscillator and a frequency-indicating circuit. The oscillator generates a frequency of three megacycles and is coupled to the frequency-indicating circuit. Changes in frequency which occur in the oscillator are indicated on the meter in the circuit. Samples are placed in a test cell which is a physical capacitor or condenser of the coaxial-cylinder type. It is connected to that part of the oscillator circuit which determines the frequency of its oscillation.

³ Steinitz LOS Electronic Tester. The mention of products or firm names does not imply that they are either endorsed or recommended by the U. S. Department of Agriculture over those not mentioned.

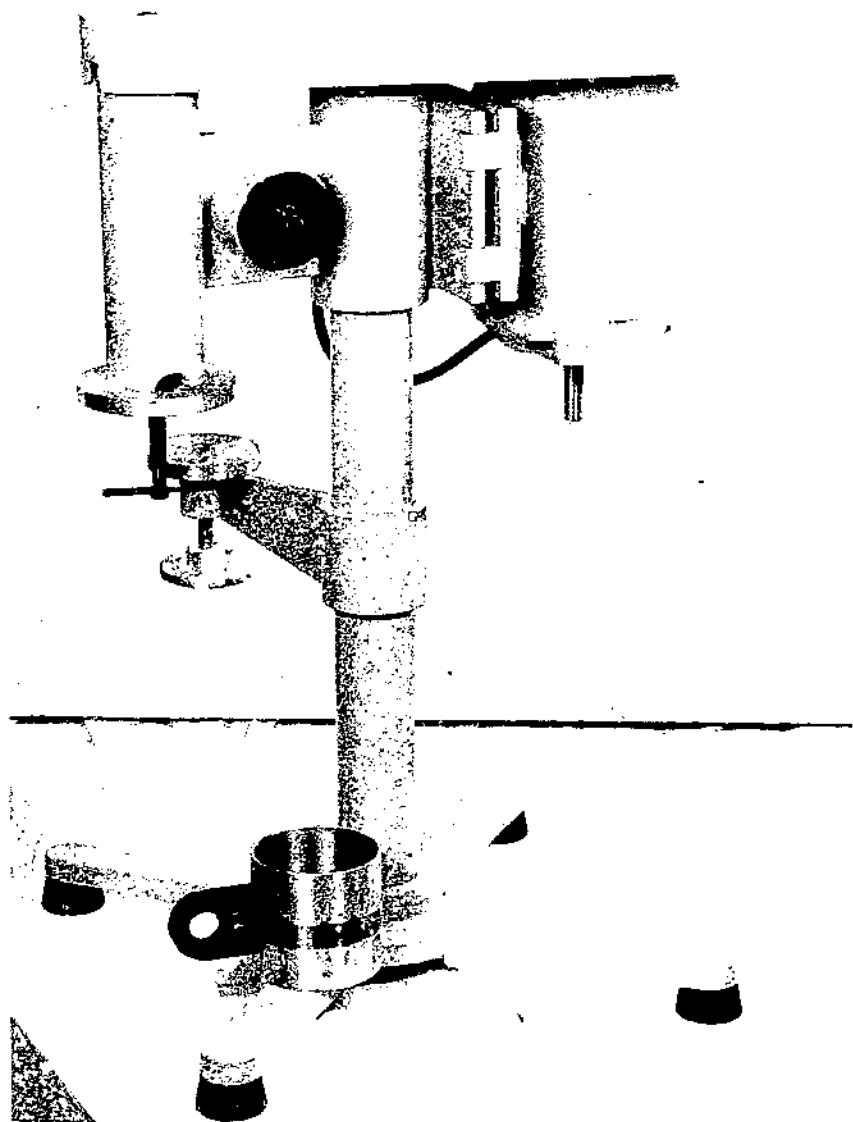


FIGURE 1. High-speed sander extractor.

EX 1233

The electronic tester functions on the principle that the radio frequency impedance of a condenser is changed when the properties of the dielectric between the plates are altered. The test cell condenser is designed to hold liquids. At the beginning of a test the cell is empty, and the dielectric between the cylinders of the cell is air. When an oil solvent is poured into the test cell, the impedance is lowered and the frequency of

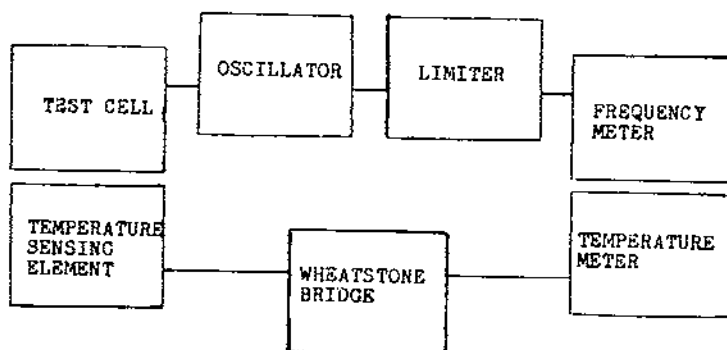


FIGURE 2.—Block diagram of the electronic tester.

AN-422B

the oscillator is changed. The change in frequency is indicated on the frequency meter. When a small quantity of oil is added to the solvent, the impedance is raised. If more oil is added to the solvent, there is a further change in the dielectric value and a corresponding increase in the impedance of the test cell condenser. Impedance changes shift the frequency of the oscillator circuit and are indicated on the frequency meter.

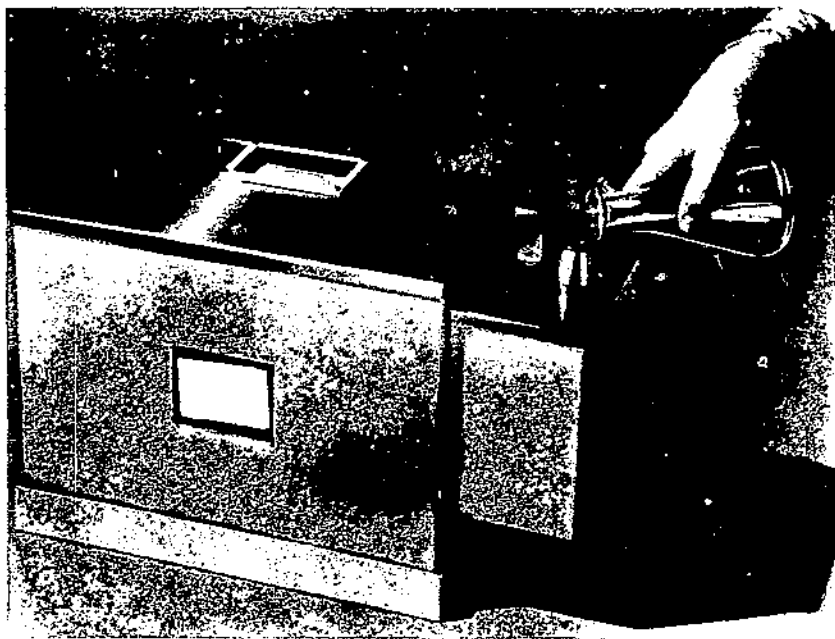
A front view of the instrument is shown in Figure 3. Figure 4 shows the condenser, a coaxial-cylinder cell.

Temperature is an important factor in dielectric measurements of liquids. The determination of the temperature for each sample in the cell is simplified by running the sample over a thermistor unit after the frequency change reading has been taken. When the inner electrode of the cell is disconnected from the connector housing, a temperature-indicating circuit is automatically connected with the meter. Removal of the center electrode allows the liquid to drain over the thermistor bulb, and the temperature can then be read on the meter.

The Dielectrometric Method for Soybeans

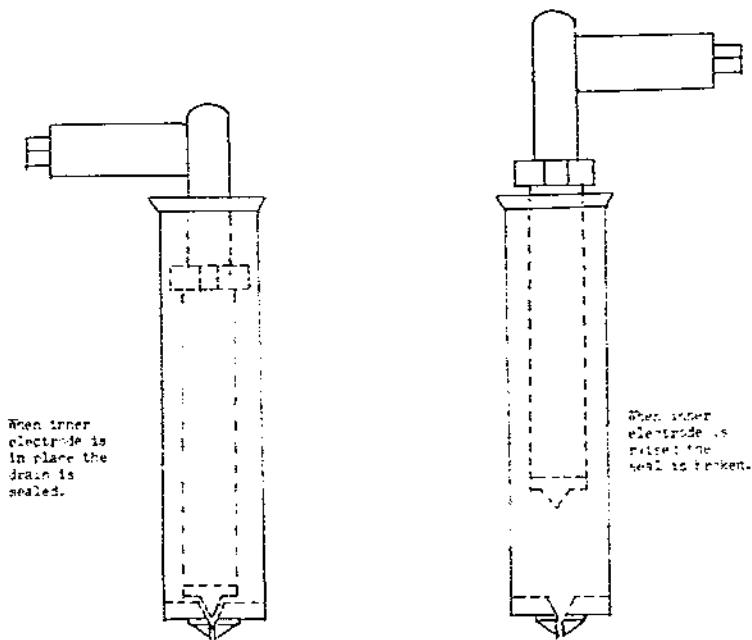
The equipment described was initially applied to the determination of the oil content of soybeans by Hunt et al (10), and then by suitable modification of the technique of analysis it was also adapted to the analysis of flaxseed by Hunt et al (11) and to safflower and sunflower seed by Johnson et al (12). The procedure for soybeans follows:

1. Turn on the tester and allow it to warm up for at least 15 minutes before taking any readings.
2. A solvent-oil mixture should be kept in the liquid cell at all times when the instrument is not in use. Drain this solution out and wipe both electrodes clean and dry with a soft cloth (preferably cheesecloth) and fill the cell with solvent. Allow to stand for a few minutes and then



NS 1229

FIGURE 3.—The electronic tester.



NS 1230

FIGURE 4.—Two views of the condenser, coaxial cylinder cell.

drain and reclean the electrodes. Balance the instrument by turning the selector switch to the red button and rotate the compensator control until the meter needle is at 45 (red line) on the "chart scale" of the meter. Refill the cell with solvent and read the meter with the selector switch at the "A" position. Lift the electrode to allow the solvent to drain out over the thermistor and take the temperature reading of the solvent on the "temperature scale" of the meter. Adjust the solvent reading to 85° F. by adding 4.5 scale divisions per degree below 85° F. or by subtracting the same value for each degree above 85° F. If the solvent reading, when adjusted to 85° F., is more than ± 5 scale divisions from a reading of 45 "A", determine the solvent correction to be added or subtracted to subsequent readings.

3. Determine the moisture content of the sample by the use of any electrical moisture tester which gives results equivalent to those obtained by the official method of the A. O. C. S.

4. Weigh to the nearest 0.1 gm. the equivalent of 100 gm. of soybeans on a moisture-free basis. The weight of sample may be calculated from the formula:

$$\text{Weight of sample in grams} = \frac{100}{100 - \%H_2O} \times 100$$

5. Place the weighed sample in the extraction cup of the grinder-extractor and grind for $\frac{1}{2}$ minute. Loosen the ground material from the bottom and sides of the cup with a spatula and add 100 ml. of orthodichlorobenzene, which has been previously adjusted to the proper dielectric value, from an all-glass automatic pipette or dispenser. Grind and extract for 4 more minutes. Timing of the operation is controlled by the time switch on the grinder-extractor.

6. Filter with vacuum through a 15-cm. Buchner funnel fitted with Whatman No. 1 filter paper or equivalent.

7. Fill the test cell nearly full with the filtrate. Stir the contents vigorously by rapidly rotating the inner electrode back and forth through an arc of approximately 270° for about 5 seconds. Snap the switch arm into the connector housing and turn the selector switch until a reading can be made on the scale. Disconnect the switch arm, re-agitate, and take a second reading. Repeat this procedure until three consecutive readings are in exact agreement. This is necessary to insure that the liquid and the cell are at equilibrium temperature. Disconnect the switch arm and lift the center electrode from the cell. Read the temperature scale after the solution has drained over the thermistor for 5 seconds.

8. Convert the meter reading to the "as is" oil content from the conversion table and then correct this value to oil content at 85° F. Since the sample was weighed on a moisture-free basis, this oil content is already expressed on a moisture-free basis.

Notes on Procedural Steps

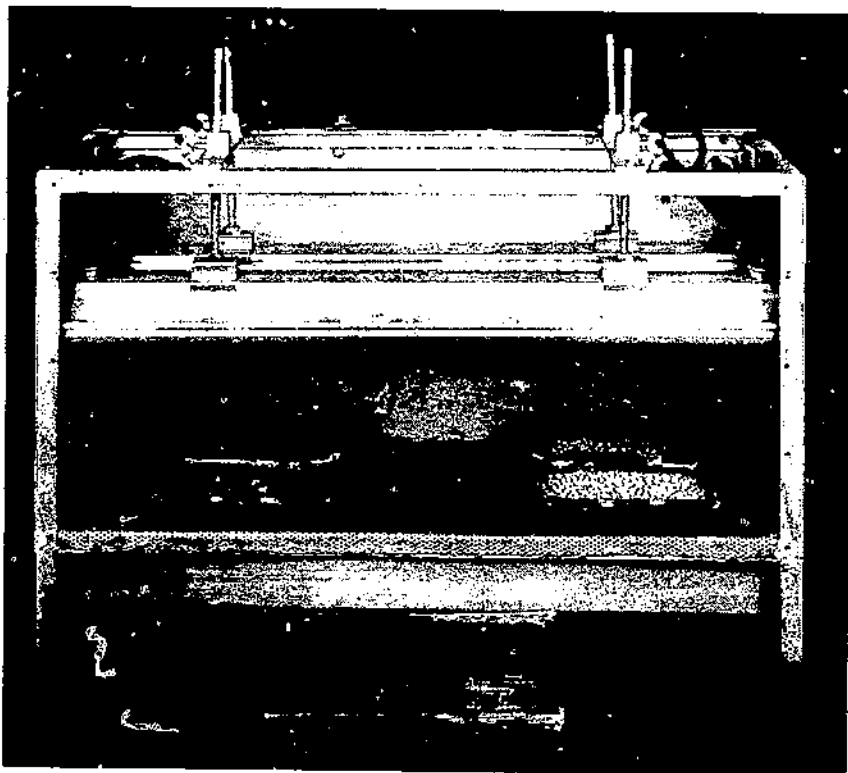
- a. Before making any tests on the solvent and before each sample is placed in the cell, the instrument must be balanced with the selector switch on the red button. Before balancing, the cell must be empty and wiped dry.
- b. As a rule, the solvent needs to be checked only once a day.
- c. Soybeans having a moisture content above 16 percent should be partially predried. The best moisture level is around 8 to 12 percent. Avoid drying the samples below 7 percent moisture. An infra-red radiant heater, designed especially for use in this procedure, will drive off about 5 percent of moisture in 5 minutes and will not harm the soybeans for use in the test (fig. 5). If time is not a critical factor, placing the samples in an air oven at 130° C. for 1 hour will dry them sufficiently. Weigh the sample before drying.
- d. Soybeans having a moisture content below 7 percent pose a special problem. Their moisture content cannot be accurately determined by electrical means and a shorter grinding-extraction time is necessary (3 minutes).

The Dielectric Method for Flaxseed

The major difficulty faced in adapting the method to flaxseed was in the filtering. The gelatinous flaxseed-orthodichlorobenzene mixture resulting from the grinding and extracting process required from 30 minutes to several hours to filter. This occurred regardless of the moisture content of the flaxseed. Vacuum filtering and pressure filtering, using many different types of filter paper, were tried without appreciably reducing the filtering time. As a similar filtering difficulty with wet soybeans was resolved by heating the samples for 5 minutes with infrared radiant heat, tests were made to determine the effect of infrared radiant heat on flaxseed. It was found that such heating of flaxseed for 4 minutes—regardless of original moisture—would prevent the formation of a gelatinous mixture during the grinding operation and the filtering time was reduced to 6 to 8 minutes per sample.

Since a current of air blowing over the sample was also necessary to carry off the released moisture and to help keep the surface of the samples cool and thereby lessen the danger of scorching, a special infrared, forced-draft, drying unit was designed and built (fig. 5). This unit consists of two 800-watt infrared radiant heaters mounted parallel on adjustable rods in a cabinet that has a perforated metal floor and a blower unit designed to blow air up through the floor. The heaters can be adjusted to any height up to 10 inches above the samples. The height used for flaxseed and soybeans is 7 inches above the samples. The three trays for holding the samples are each 8 x 6 x 1¼ inches and are divided by a partition to make two compartments, each 6 x 4 inches. Samples are placed in each compartment and, after heating, are poured out sepa-

rately by covering one compartment. There is an opening $1\frac{5}{16} \times 18\frac{1}{4}$ inches in the front part of the cabinet at floor level for inserting the trays into the unit. The trays are made of finely perforated metal or of heavy woven-wire screen so that air blowing up through the floor of the cabinet will pass through the samples.



BN-1231

FIGURE 5. Infrared, forced-draft, drying unit (with front panel removed).

Certain changes in the procedure for soybeans are necessary to make it applicable to flaxseed. In particular, steps 3, 4, 5, and 8 require the following substitutions:

3. Determine the moisture content of the flaxseed if it is desired to express oil content at some fixed moisture basis.

4. Weigh 80 gm. of flaxseed to the nearest 0.1 gm. and place in the tray of the drying unit. Preheat the drying unit for approximately 1 minute before putting the sample into it. Heat the sample for 4 minutes, stirring occasionally with a spatula to prevent surface overheating.

5. Place the dried sample in the extraction cup of the high-speed mill and add 120 ml. of orthodichlorobenzene, which has been previously adjusted to the proper dielectric value, from an all-glass automatic pipette or dispenser. Grind and extract for 4 minutes. Timing of the operation may be controlled by the time switch on the mill.

8. Convert the meter reading to the "as is" oil content from the conversion table and then correct this value to oil content at 85° F. To convert the oil content results from an "as is" basis to any moisture basis desired the following formula is used:

$$\frac{\% \text{ oil} \times (100 - \text{desired moisture basis})}{100 - \text{actual moisture}} \times 100$$

Flaxseed having a moisture content below 5 percent poses a special problem. In such cases the moisture content cannot be accurately determined by electrical means and a shorter grinding-extraction time is necessary (3 minutes).

The Dielectrometric Method for Safflower and Sunflower Seed

Since there are no well-established methods for determining either the oil content or the moisture content of safflower and sunflower seed, standard procedures for determining these factors were developed to provide a basis of reference. The experimental work involved in the development of the reference method is described in the paper by Johnson et al. (12). The adopted standard procedure for determining the oil content of safflower seed and sunflower seed is described earlier in the section under "Determination of Oil Content—Extraction Method."

With the Karl Fischer method for determining moisture content as a reference, practical routine methods for determining moisture content of safflower seed and sunflower seed were adopted. Moisture content of safflower seed is determined by heating 10-gm. samples of whole seed for 1 hour at 130° C. in an air oven and that of sunflower seed by heating 10-gm. samples of whole seed for 3 hours at 130° C. in an air oven. In each case the loss in weight is calculated as the moisture content.

The dielectrometric method for determining the oil content of safflower seed and sunflower seed is essentially the same as that described for soybeans except for the changes in steps 3, 4, 5, and 8 as follows:

3. Determine the moisture content of the safflower seed or sunflower seed sample since it is usually desirable to express oil content on some definite moisture basis.

4. Weigh 80 ± 0.1 gm. of safflower seed or sunflower seed into the extraction cup.

5. Grind extract in the high-speed mill with 120 ml. of orthodichlorobenzene for 4 minutes.

8. Convert the meter readings to the "as is" oil content using the proper conversion table and then apply the temperature correction. To convert the oil content results from an "as is" basis to any moisture basis desired, the following formula may be used:

$$\frac{\% \text{ oil} \times (100 - \text{desired moisture basis})}{100 - \% \text{ moisture of sample}} \times 100$$

Experimental Data and Results With the Dielectrometric Method

Soybeans.—Eighty-six samples of soybeans ranging in oil content from 18.2 percent to 23.3 percent were analyzed under varying conditions of time and temperature of extraction. Two electronic testers for making readings under varying and at constant room temperatures were used. From 2 to 10 tests were made on each sample, and the meter readings were graphically plotted against the oil content as determined by the Soxhlet extraction procedure. Figure 6 shows the distribution of these points along the regression line. The regression line is plotted from the regression equation:

$$y = 14.77 + .09409x$$

where y = oil content (percentage)
 x = meter readings

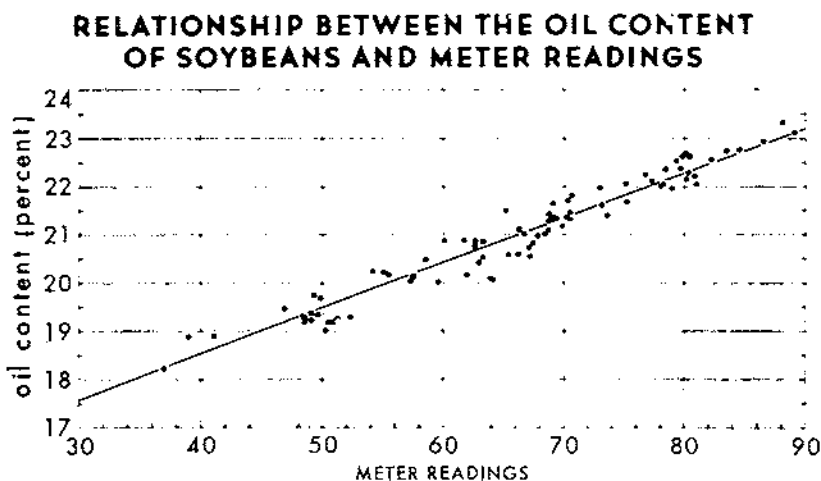


FIGURE 6.—Relationship between oil content of soybeans and meter readings.

A conversion table (table 2) to translate meter readings into oil content was drawn up from the regression equation. The standard error of estimate in determining oil content by the dielectric method was found to be 0.27 in terms of percentage of oil. The coefficient of determination ($r^2 \times 100$) was found to be 96.0, that is, 96 percent of the variance in oil content is concomitant with variation in meter readings.

Flaxseed.—Seventy-four samples of flaxseed ranging in oil content from 37.04 percent to 44.14 percent were analyzed under varying conditions of time and temperature of extraction, using two electronic testers for reading each sample. Replicate determinations were made on each sample, and their meter readings were graphically plotted against the oil content as determined by the regular extraction procedure. Figure 7

TABLE 2.—*Electronic Oil Tester Conversion Chart for Soybeans*

Meter reading	Selector button D—Oil content (percent)	Meter reading	Selector button D—Oil content (percent)	Temperature correction	F. Percent oil
10	15.76	56	20.02	70	-5.25
11	15.95	57	20.11	71	-4.90
12	15.94	58	20.20	72	+4.55
13	16.03	59	20.30	73	+4.20
14	16.12	60	20.40	74	+3.85
15	16.21	61	20.50	75	-3.50
16	16.30	62	20.60	76	+3.15
17	16.39	63	20.70	77	+2.80
18	16.48	64	20.80	78	+2.45
19	16.57	65	20.90	79	+2.10
20	16.68	66	21.00	80	+1.75
21	16.77	67	21.10	81	+1.40
22	16.86	68	21.20	82	+1.05
23	16.95	69	21.30	83	+0.70
24	17.04	70	21.40	84	+0.35
25	17.13	71	21.50		
26	17.22	72	21.60	85	0
27	17.31	73	21.68		
28	17.40	74	21.78	86	-0.35
29	17.50	75	21.87	87	-0.70
30	17.60	76	21.96	88	-1.05
31	17.70	77	22.05	89	-1.40
32	17.80	78	22.14	90	-1.75
33	17.90	79	22.23	91	-2.10
34	18.00	80	22.32	92	-2.45
35	18.09	81	22.41	93	-2.80
36	18.18	82	22.50	94	-3.15
37	18.27	83	22.60	95	-3.50
38	18.36	84	22.70	96	-3.85
39	18.45	85	22.80	97	-4.20
40	18.54	86	22.90	98	-4.55
41	18.63	87	23.00	99	-4.90
42	18.72	88	23.09	100	-5.25
43	18.81	89	23.18		
44	18.90	90	23.27		
45	19.00	91	23.36		
46	19.10	92	23.45		
47	19.20	93	23.54		
48	19.30	94	23.63		
49	19.39	95	23.72		
50	19.48	96	23.81		
51	19.57	97	23.90		
52	19.66	98	23.98		
53	19.75	99	24.07		
54	19.84	100	24.16		
55	19.93				

shows the distribution of these points along the regression line. The regression line is plotted from the regression equation:

$$y = 34.92 + .14567x$$

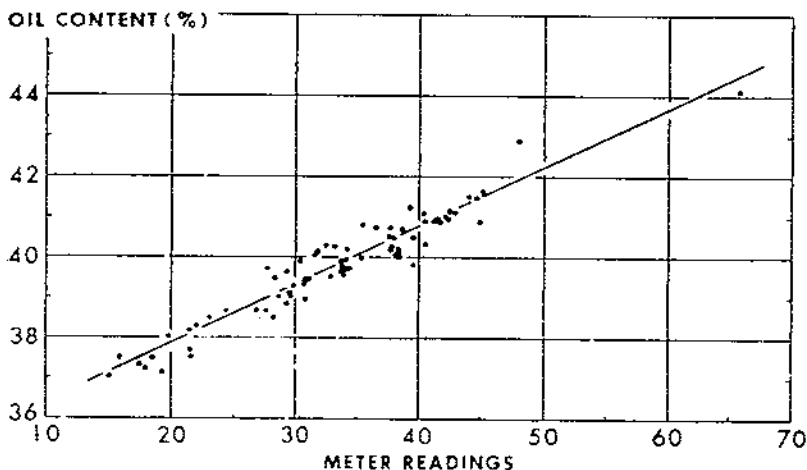
where y = oil content (percentage)

x = meter readings

A conversion table (table 3) to translate meter readings into oil content was prepared from the regression equation. The standard error of estimate in determining oil content by the dielectric method was found to be

0.35 in terms of percentage of oil. The coefficient of determination ($r^2 \times 100$) was found to be 94.1, that is, 94.1 percent of the variance in oil content is concomitant with variation in meter reading.

RELATIONSHIP BETWEEN THE OIL CONTENT OF FLAXSEED AND METER READINGS



HN 1224

FIGURE 7.—Relationship between the oil content of flaxseed and meter readings.

Safflower Seed and Sunflower Seed.—Seventy-six samples of safflower seed having a range in oil content from 31.3 to 39.9 percent and 81 samples of sunflower seed having a range of oil content from 22.3 to 34.1 percent, representing two crop years, were analyzed for oil content by both the standard and dielectric methods. Duplicate determinations were made by each method. Two electronic testers were used for reading each filtrate under varying room temperatures. The meter readings corrected to 85° F. were graphically plotted against the oil content determined by the overnight extraction procedure.

Figure 8 shows the relationship between oil content and respective meter readings for both safflower seed and sunflower seed.

The regression lines are plotted from the following regression equations, derived statistically from the data:

$$\text{Safflower seed} \quad y = 32.5287 + 0.1616x$$

$$\text{Sunflower seed} \quad y = 21.5700 + 0.1430x$$

where y = oil content (%) ("as is" moisture basis)

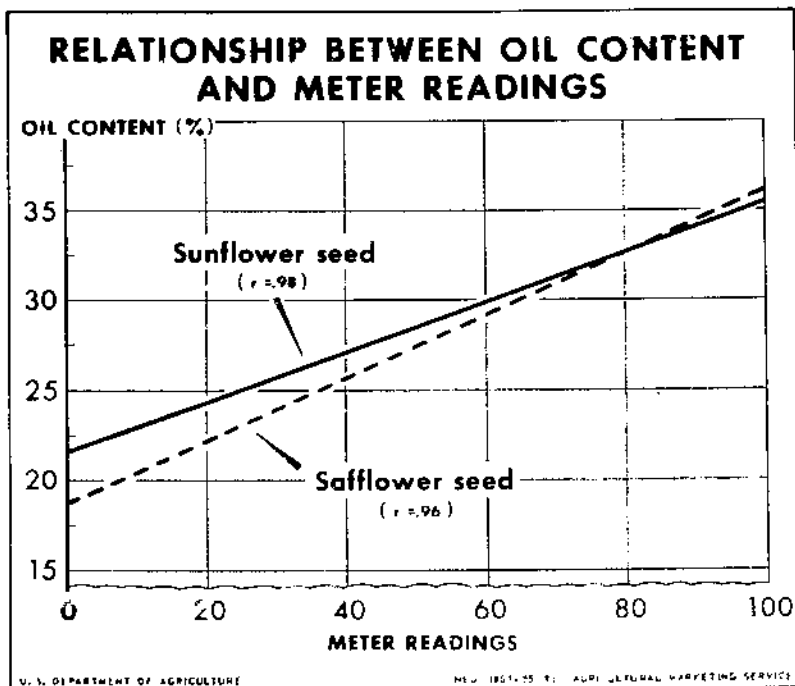
x = meter readings (corrected to 85° F.)

Conversion charts (tables 4 and 5) for converting meter readings to oil content percentages were prepared from the regression equations for each oilseed. The standard error of estimate in determining oil content by the rapid dielectric method was found to be 0.27 in terms of percentage

TABLE 3.—*Electronic Oil Tester Conversion Chart for Flaxseed*

Meter reading	Selector button		Meter reading	Selector button		Temperature correction	
	D—oil content, (percent)	E—oil content, (percent)		D—oil content, (percent)	E—oil content, (percent)	F.	Percent oil
10		36.04	56	31.29	12.91	70	-8.55
11		36.19	57	31.44	13.09	71	-7.08
12		36.34	58	31.59	13.24	72	-7.41
13		36.49	59	31.74	13.39	73	-6.84
14		36.64	60	31.89	13.51	74	-6.27
15		36.79	61	32.01	13.69	75	-5.70
16		36.91	62	32.19	13.84	76	-5.13
17		37.09	63	32.31	13.99	77	-4.56
18		37.24	64	32.49	14.14	78	-3.99
19		37.39	65	32.61	14.29	79	-3.42
20		37.54	66	32.79	14.44	80	-2.85
21		37.69	67	32.94	14.59	81	-2.28
22		37.84	68	33.09	14.74	82	-1.71
23		37.99	69	33.21	14.89	83	-1.14
24		38.14	70	33.39	15.01	84	0
25		38.29	71	33.54			
26		38.44	72	33.69		85	0
27		38.59	73	33.81			
28		38.74	74	33.99		86	-1.57
29		38.89	75	34.14		87	-1.14
30		39.04	76	34.29		88	-1.71
31		39.19	77	34.44		89	-2.28
32		39.34	78	34.59		90	-2.85
33		39.49	79	34.71		91	-3.42
34		39.64	80	34.89		92	-3.99
35		39.79	81	35.01		93	-4.56
36		39.94	82	35.19		94	-5.13
37		40.09	83	35.31		95	-5.70
38		40.24	84	35.49		96	-6.27
39		40.39	85	35.64		97	-6.84
40		40.54	86	35.79		98	-7.41
41		40.69	87	35.94		99	-7.98
42		40.84	88	36.09		100	-8.55
43		40.99	89	36.24			
44		41.14	90	36.39			
45		41.29	91	36.54			
46		41.44	92	36.69			
47		41.59	93	36.84			
48	30.09	41.74	94	36.99			
49	30.21	41.89	95	37.14			
50	30.39	42.04	96	37.29			
51	30.54	42.19	97	37.44			
52	30.69	42.31	98	37.59			
53	30.81	42.49	99	37.71			
54	30.99	42.64	100	37.89			
55	31.14	42.79					

of oil for safflower seed and 0.34 percent for sunflower seed. The coefficient of determination ($r^2 \times 100$) for safflower seed is 92.0 and for sunflower seed is 96.0, that is, 92 percent of the variance in oil content is concomitant with variation in meter readings in the case of safflower seed and 96 percent of the variance in oil content is concomitant with variation in meter readings in the case of sunflower seed.



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FIGURE 8.—The relationship between the oil content of safflower seed and sunflower seed and respective meter readings.

Value of the Dielectrometric Method

The dielectrometric method appears to be quite suitable for practical application to the rapid determination of the oil content of soybeans, flaxseed, sunflower seed, and safflower seed. The cost of the equipment, the grinder-extractor and the electronic tester, is between \$800 and \$900. Filtration equipment cost is very minor and the solvent costs 7 to 8 cents per determination. Results can be obtained in about 15 to 20 minutes, depending on the material tested. If a series of samples is analyzed, the time per sample is further reduced because the grinding-extracting of one or more samples can take place while previous extracts are filtering. It is estimated that two analysts working with two grinder-extractors and one electronic tester could analyze from 20 to 25 samples per hour.

The technique of analysis is simple enough so that untrained technicians can, with brief instructions, perform the analysis with the speed and accuracy indicated previously.

A rapid, practical test of this type should readily lend itself to application in the areas where these oilseeds are grown and marketed. It should be equally applicable to other oilseeds. The test could be used in the trading of oilseeds on the basis of the principal factor affecting their intrinsic value—the oil content.

TABLE 1.—*Electronic Oil Tester Conversion Chart for Safflower Seed*

Meter reading	Selector button		Meter reading	Selector button		Temperature correction	
	D—oil content, (percent)	E—oil content, (percent)		D—oil content, (percent)	E—oil content, (percent)		
10.....	20.44	31.14	56.....	28.16	41.58	70	+ 7.12
11.....	20.61	34.31	57.....	28.63	41.74	71	+ 6.65
12.....	20.79	34.47	58.....	28.81	41.90	72	+ 6.17
13.....	20.96	34.63	59.....	28.98	42.06	73	+ 5.70
14.....	21.13	34.79	60.....	29.16	42.22	74	+ 5.22
15.....	21.31	34.95	61.....	29.33	42.38	75	+ 4.75
16.....	21.48	35.11	62.....	29.51	42.55	76	+ 4.28
17.....	21.66	35.28	63.....	29.68	42.71	77	+ 3.80
18.....	21.83	35.44	64.....	29.86	42.87	78	+ 3.33
19.....	22.01	35.60	65.....	30.03	43.03	79	+ 2.85
20.....	22.18	35.76	66.....	30.20	43.19	80	+ 2.38
21.....	22.36	35.92	67.....	30.38	43.35	81	+ 1.90
22.....	22.53	36.08	68.....	30.55	43.52	82	+ 1.43
23.....	22.70	36.24	69.....	30.73	43.68	83	+ .95
24.....	22.88	36.41	70.....	30.90	43.84	84	+ .48
25.....	23.05	36.57	71.....	31.08	44.00
26.....	23.23	36.73	72.....	31.25	44.16	85	0
27.....	23.40	36.89	73.....	31.43	44.32
28.....	23.58	37.05	74.....	31.60	44.48	86	+ .18
29.....	23.75	37.21	75.....	31.77	44.65	87	+ .95
30.....	23.93	37.38	76.....	31.95	44.81	88	+ 1.43
31.....	24.10	37.54	77.....	32.12	44.97	89	+ 1.90
32.....	24.27	37.70	78.....	32.30	45.13	90	+ 2.38
33.....	24.45	37.86	79.....	32.47	45.29	91	+ 2.85
34.....	24.62	38.02	80.....	32.65	45.45	92	+ 3.33
35.....	24.80	38.18	81.....	32.82	45.62	93	+ 3.80
36.....	24.97	38.35	82.....	33.00	45.78	94	+ 4.28
37.....	25.15	38.51	83.....	33.17	45.94	95	+ 4.75
38.....	25.32	38.67	84.....	33.34	46.10	96	+ 5.22
39.....	25.50	38.83	85.....	33.52	46.26	97	+ 5.70
40.....	25.67	38.99	86.....	33.69	46.42	98	+ 6.17
41.....	25.84	39.15	87.....	33.87	46.58	99	+ 6.65
42.....	26.02	39.31	88.....	34.04	46.75	100	+ 7.12
43.....	26.19	39.48	89.....	34.22	46.91
44.....	26.37	39.64	90.....	34.39	47.07
45.....	26.54	39.80	91.....	34.57	47.23
46.....	26.72	39.96	92.....	34.74	47.39
47.....	26.89	40.12	93.....	34.91	47.55
48.....	27.06	40.28	94.....	35.09	47.72
49.....	27.24	40.45	95.....	35.26	47.88
50.....	27.41	40.61	96.....	35.44	48.04
51.....	27.59	40.77	97.....	35.61	48.20
52.....	27.76	40.93	98.....	35.79	48.36
53.....	27.94	41.09	99.....	35.96	48.52
54.....	28.11	41.25	100.....	36.14	48.69
55.....	28.29	41.41

DETERMINATION OF OIL QUALITY

In addition to the quantity of oil in oilseeds the quality of that oil is an important consideration. Whether the oil is used industrially or for food purposes the quality in terms of unsaturation needs to be known. As has already been indicated, the degree of unsaturation is most commonly measured by the iodine number. The chemical determination of iodine number by the Wijs method is time-consuming and requires the

TABLE 5.—*Electronic Oil Tester Conversion Chart for Sunflower Seed*

Meter reading	Selector button D—Oil content (percent)	Meter reading	Selector button D—Oil content (percent)	Temperature correction	<i>F. Percent oil</i>
10.....	23.00	56.....	29.58	70	+8.11
11.....	23.14	57.....	29.72	71	+7.60
12.....	23.29	58.....	29.86	72	+7.05
13.....	23.43	59.....	30.01	73	+6.51
14.....	23.57	60.....	30.15	74	+5.97
15.....	23.72	61.....	30.29	75	+5.43
16.....	23.86	62.....	30.44	76	+4.88
17.....	24.00	63.....	30.58	77	+4.34
18.....	24.14	64.....	30.72	78	+3.80
19.....	24.29	65.....	30.87	79	+3.26
20.....	24.43	66.....	31.01	80	+2.71
21.....	24.57	67.....	31.15	81	+2.17
22.....	24.72	68.....	31.29	82	+1.63
23.....	24.86	69.....	31.44	83	+1.09
24.....	25.00	70.....	31.58	84	+0.54
25.....	25.15	71.....	31.72	85	0
26.....	25.29	72.....	31.87	86	-0.51
27.....	25.43	73.....	32.01	87	-1.09
28.....	25.57	74.....	32.15	88	-1.63
29.....	25.72	75.....	32.30	89	-2.17
30.....	25.86	76.....	32.44	90	-2.71
31.....	26.00	77.....	32.58	91	-3.26
32.....	26.15	78.....	32.72	92	-3.80
33.....	26.29	79.....	32.87	93	-4.34
34.....	26.43	80.....	33.01	94	-4.88
35.....	26.58	81.....	33.15	95	-5.43
36.....	26.72	82.....	33.30	96	-5.97
37.....	26.86	83.....	33.44	97	-6.51
38.....	27.00	84.....	33.58	98	-7.05
39.....	27.15	85.....	33.73	99	-7.60
40.....	27.29	86.....	33.87	100	-8.11
41.....	27.43	87.....	34.01
42.....	27.58	88.....	34.15
43.....	27.72	89.....	34.30
44.....	27.86	90.....	34.44
45.....	28.01	91.....	34.58
46.....	28.15	92.....	34.73
47.....	28.29	93.....	34.87
48.....	28.43	94.....	35.01
49.....	28.58	95.....	35.16
50.....	28.72	96.....	35.30
51.....	28.86	97.....	35.44
52.....	29.01	98.....	35.58
53.....	29.15	99.....	35.73
54.....	29.29	100.....	35.87
55.....	29.44

skill of a trained technician or a chemist. The same objections can be applied to other well-known methods of iodine number determination; namely, the Hanus, Hubl, Kaufmann, and Rosenmund-Kuhnemann methods.

Rapid Methods for Iodine Number

Hoffman and Green (7) used mercuric acetate as a catalyst in the Wijs method to obtain complete iodine absorption in 3 minutes. Benham

and Klee (2) modified the Rosenmund-Kuhnhenn method so that only a 1-minute reaction time was required. Mukherjee (22) tried an aqueous solution using hypochlorous acid as a reagent, and reduced reaction time to 5 minutes. The results obtained with these methods do not, for all types of oils, check with the results obtained with the generally accepted Wijs method. In addition, as much, or more, manipulative skill is required for these methods as for the Wijs method.

Iodine Number by Refractive Index Measurements

Niegemann and Kayser (23) appear to be the first investigators to show that a relationship exists between the refractive index and the iodine number of oils. Shortly thereafter Arnold (1) published his observations on the same subject. Since then there have been numerous papers describing this relationship for many different types of oils.

The practicability of using the refractive index of a freshly prepared vegetable oil from sound plant material as a relatively accurate measure of its iodine number was first demonstrated by Zeleny and Coleman (29) working with flaxseed. This work was very shortly confirmed by the work of Hopper and Nesbitt (8) and of Lehberg and Geddes (16). The refractometric method for determining oil iodine numbers was later applied to soybeans by Zeleny and Neustadt (30), who found that the relationship between refractive index and iodine number appeared to be identical for the freshly prepared oils of both flaxseed and soybeans. Some evidence exists to indicate that the relationship between the refractive index and the iodine number of freshly prepared vegetable oils is essentially identical for all or most oils that contain not more than traces of glycerides of unsaturated fatty acids other than oleic, linoleic, and linolenic acids.

The refractometric method for determining the iodine numbers of the oils in flaxseed and soybeans is now rather commonly used by processors because of its convenience and the rapidity with which tests can be made. Determinations may be made with freshly extracted oils from which all traces of solvent have been removed, or, more simply, with oils freshly expressed from the whole or freshly ground seed by means of a small laboratory-type hydraulic press. The method is not applicable to certain types of damaged seed or to old seeds. Only a few drops of oil are needed. For accurate work the refractive index of the oil should be determined to an accuracy of ± 0.00002 . The refractive index is commonly determined at 25.0° C., either by controlling the temperature of the refractometer prisms to that temperature or by determining accurately the actual temperature of the prisms and correcting the value obtained to a temperature of 25.0° C. The refractive index temperature correction factor for flaxseed oil is 0.000357 and for soybean oil 0.000364 per degree C. The corrections are added when refractive index readings are taken at temperatures above 25.0° C. and subtracted when readings

are taken below that temperature. The iodine number may then be calculated from the refractive index by means of the equation:

$$\text{Iodine number (Wijs)} = 8584.97 n_D^{25} - 12513.83$$

or by means of the published conversion tables for flaxseed oil (29) and soybean oil (30).

THE HAND REFRACTOMETER

Unless a precision refractometer is already on hand for other types of work, such as the determination of oil content, it is generally too expensive (about \$2,200 for the refractometer and constant temperature devices) to acquire one for use in determining iodine number. The dipping type of refractometer with interchangeable prisms, which can also be used for this type of work, is less expensive than the precision refractometer.

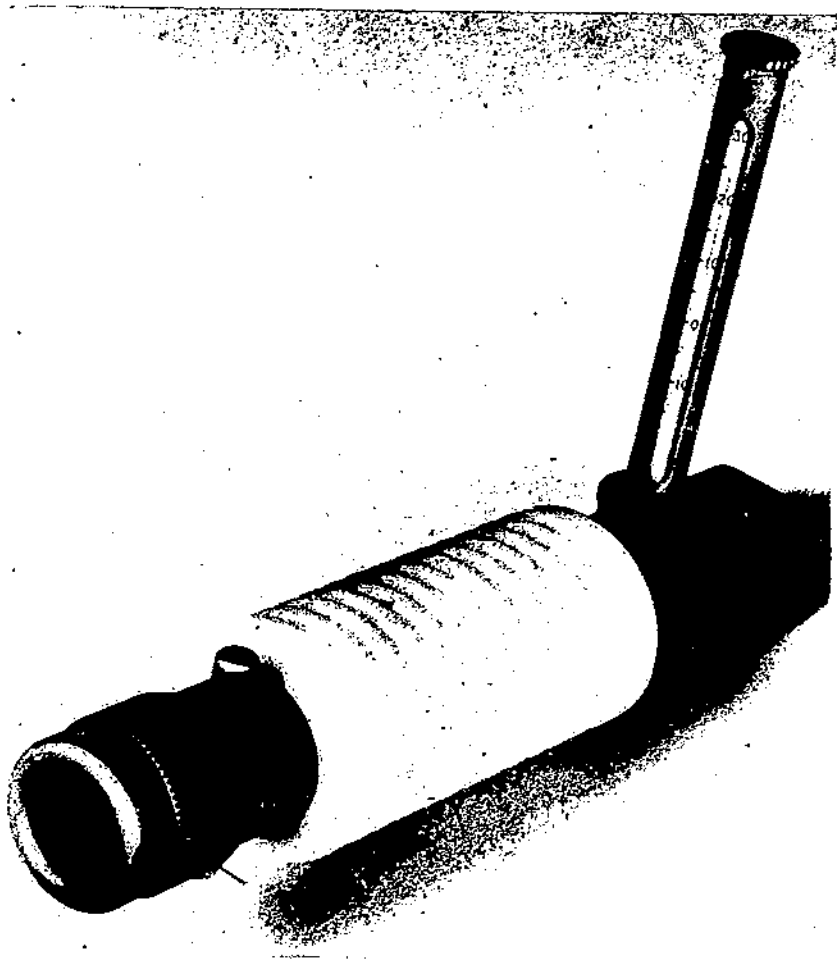
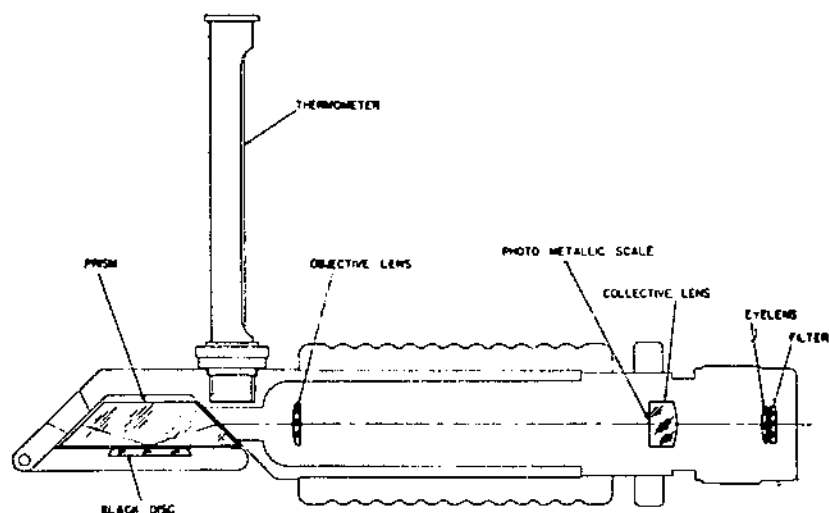


FIGURE 9.—Iodine-number refractometer.

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However, this equipment, like the precision refractometer, is large and would not readily lend itself to application in routine testing of oilseeds for the iodine number of their oils.

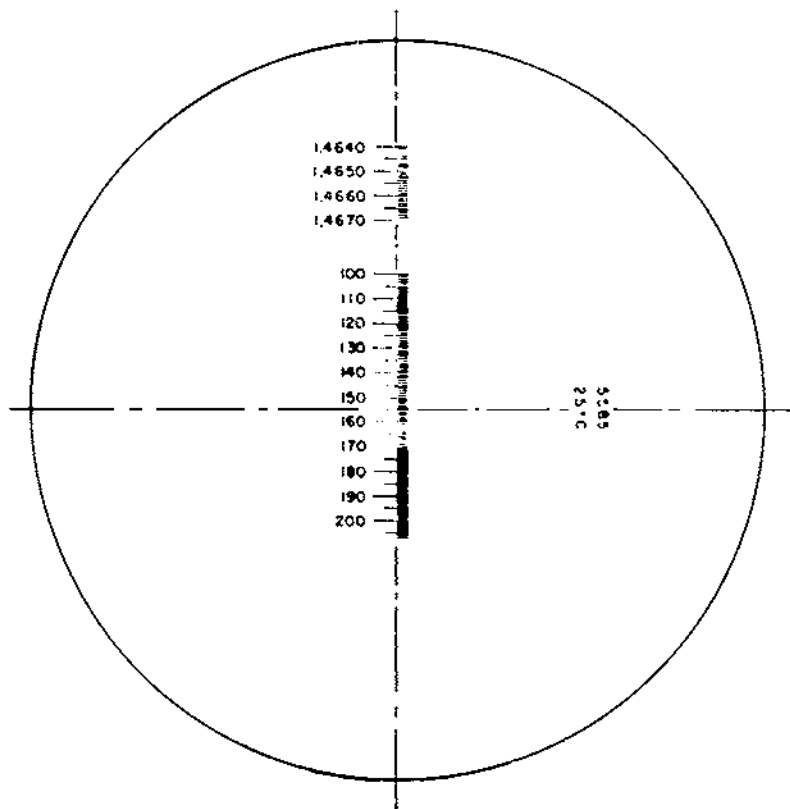
Hunt et al (9) working with this problem developed a simple, direct reading iodine number refractometer shown in Figure 9. Sketches showing the principal functional parts and the visual field of this refractometer are shown in Figures 10 and 11. This refractometer has a range in refractive index from 1.4693 to 1.4817 which is equivalent to iodine numbers of 100.1 to 206.5. The scale reads directly in iodine number and it is accurate within ± 1.0 iodine number. Another unique feature of it is that it has a thermometer imbedded close to the prism which is calibrated to read directly in iodine-number corrections.



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FIGURE 10.—Diagrammatic sketch showing principal functional parts of iodine-number refractometer.

The first experimental model of this instrument was tested with 89 samples of oil, consisting of flaxseed oil, soybean oil, and various mixtures of flaxseed and soybean oils, covering a relatively wide range in iodine numbers. The readings were compared with those obtained on the same samples with a precision refractometer that had been carefully checked for accuracy. The mean deviation between the results obtained with the two instruments was 0.28 iodine-number units and the maximum deviation was 0.7 units. Thus the experimental hand refractometer was shown to be in satisfactory agreement with the precision refractometer. Readings with the hand refractometer were made using different sources of light, including daylight, fluorescent light, and light from various types of incandescent bulbs. All types of light used were found to be equally effective, provided that proper light intensities were used.



HN-4225

FIGURE 11.—Visual field of iodine-number refractometer.

The iodine-number scale in the hand refractometer is graduated in whole units but can readily be read to the nearest one-half unit. With a little experience, an operator can estimate one-tenth iodine-number units.

A person with little technical training, using the new, iodine-number refractometer and a simple, hand-operated laboratory hydraulic press, can determine routinely, and with satisfactory accuracy, oil iodine numbers of flaxseed or soybeans at the rate of about one test every 5 minutes, starting with the original unground flaxseed or soybean samples. Such a procedure would appear to meet the requirements for speed and practicability needed in routine testing.

It has been reported that the hand refractometer is being used at present in plant control of the hydrogenation process in the manufacture of shortenings and oleomargarine.

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