dium-carbon catalysts, thereby speeding up the determinations, improving their precision, and extending their utility.

**Acknowledgment**

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# A Rapid Dielectric Method for Determining the Oil Content of Flaxseed[1]

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THE theory and application of high-frequency oscillators to chemical analysis was covered in a previous paper (1). Briefly summarized, the basic principle in the application of high-frequency oscillators to chemical analysis depends upon the fact that when a solution or solid 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 concentration of the sample. Any one or more of the characteristics or their changes, such as plate or grid current, plate or grid voltage, or frequency changes may be measured. 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 coil
$C$ = capacitance of the parallel resonant circuit

The electronic tester described in the previous paper 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 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.

The previous paper covered in detail the equipment and procedure for determining the oil content of soybeans by a dielectric method. The purpose of the present paper is to describe the modifications in the procedure that are necessary to adapt the dielectric method for determining oil content to flaxseed analysis and to present conversion data for computing percentage of oil from dielectric readings.

## The Application of High-Frequency Oscillators to Analysis of Flaxseed for Oil Content

Briefly, the dielectric method for determining the oil content of flaxseed and soybeans consists of simultaneously grinding and extracting the sample in a Stein Laboratory Mill (Figure 1)[2] in the presence of orthodichlorobenzene, filtering, and then measuring in the Steinlite LOS Unit (Figure 2)[2] the dielectric value of the filtrate and converting this value to percentage of oil.

The major difficulty faced in adapting the method to flaxseed was in the filtering. The flaxseed-orthodichlorobenzene mixture, after simultaneously grinding and extracting with the mill, was a gelatinous mixture that 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 five minutes with infrared radiant heat (1), 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 (Figure 3). This unit consists of two Chromalox,[2] type RAD, 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 soy-

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[1] The study on which these findings are based was made under authority of the Agricultural Marketing Act of 1946 (RMA, Title II).

[2] The mention of firm names or trade products does not imply that they are endorsed or recommended by the Department of Agriculture over other firms or similar products not mentioned.

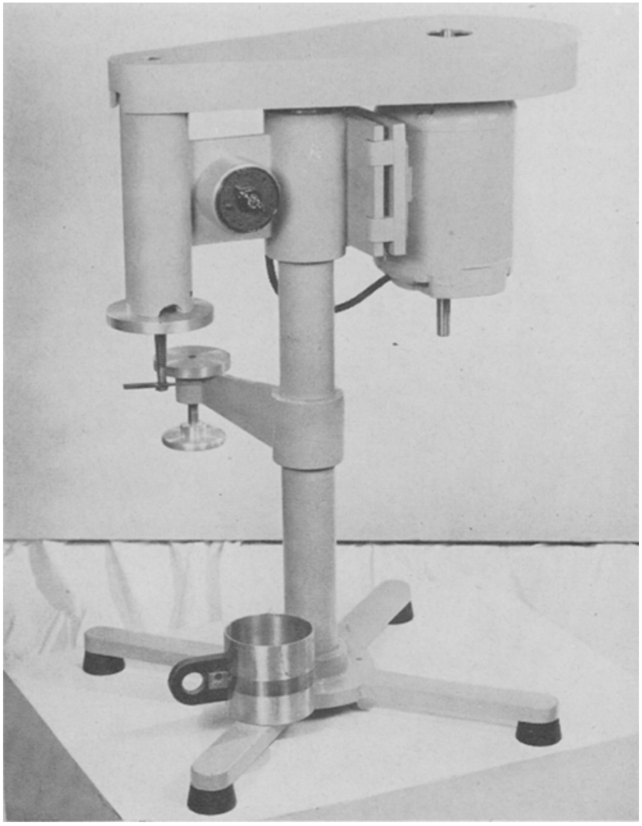

FIG. 1. High-speed grinder-extractor.

beans 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 separately by covering one compartment. There is an opening 1⁵⁄₁₆ x 18¼ 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.

## Analytical Procedure

1. Turn on the LOS unit 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 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 flaxseed if it is desired to express oil content at some fixed moisture basis.

4. Weigh to the nearest 0.1 g. 80 g. of flaxseed 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 Stein Laboratory Mill and add 120 ml. of orthodichlorobenzene, which has been previously adjusted to the proper dielectric value,[3] 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.

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, reagitate, 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

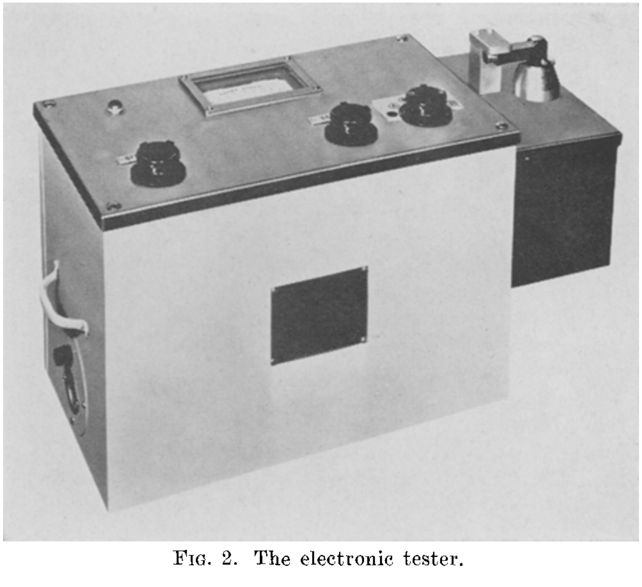

FIG. 2. The electronic tester.

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[3] The manufacturer of the apparatus has provided an adjusted and blended solvent of the appropriate properties under the name Steinlite Solvent No. 1.

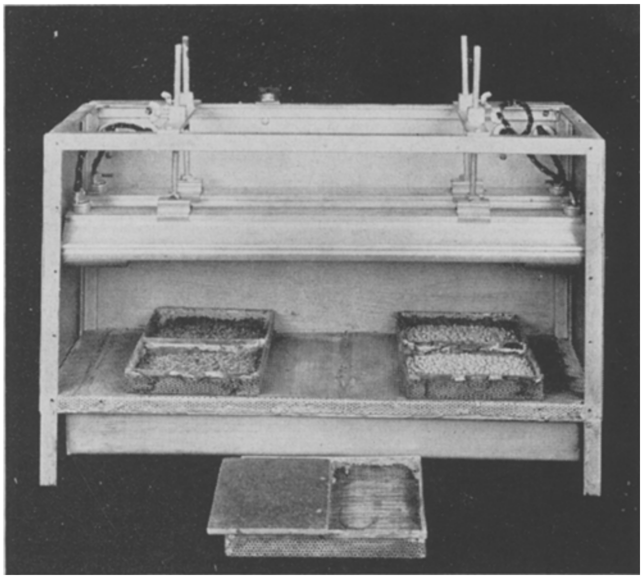

FIG. 3. Infrared, forced-draft, drying unit (with front panel removed).

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:

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

### 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) Flaxseed having a moisture content below 5% poses a special problem. Its moisture content cannot be accurately determined by electrical means and also a shorter grinding-extraction time is necessary (3 minutes).

### Experimental Data and Results

The successful application of the dielectric method to the analysis of soybeans for oil content, as described in a previous paper, gave indication that the method would be applicable to flaxseed and perhaps other oil-bearing seeds. After preliminary studies with flaxseed and modification of the method for soybeans to flaxseed, 74 samples of flaxseed ranging in oil content from 37.04% to 44.14% were analyzed under varying conditions of time and temperature of extraction, using two electronic testers for reading each sample under varying and at constant room temperatures. 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 4 shows the distribution of these points along the regression line.

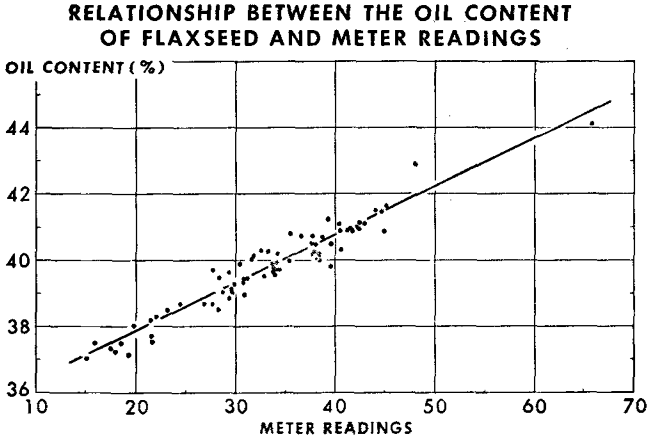

FIG. 4. Relationship between the oil content of flaxseed and meter readings.

The regression line is plotted from the regression equation:

$$y = 34.92 + .14567x$$

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

A conversion table 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 0.346 in terms of percentage of oil. The coefficient of correlation was found to be + 0.97.

### Discussion

The method of analysis described above appears to be quite suitable for practical application to the rapid determination of the oil content of flaxseed. Results on a single sample can be obtained in about 20 minutes, the variable being the rate of filtration. If a series of samples is analyzed, then the time per sample is further reduced because the grinding-extraction 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 non-chemists can, with brief instructions, perform the analysis with the speed and accuracy previously indicated.

### Acknowledgment

The mill and the electronic tester were built for this work by the Fred Stein Laboratories, Atchison, Kansas, and they have worked closely with the United States Department of Agriculture in developing the method.

The infrared forced-draft drying unit was built by the Mechanical Shops of the Agricultural Research Center, Beltsville, Maryland.

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