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

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## Theory and Application of High-Frequency Oscillators to Chemical Analysis

ACCORDING to West, Burkhalter, and Broussard (12), who have surveyed the field, high-frequency oscillators have been in use for almost 40 years. However application of high-frequency oscillators to chemical analysis has occurred only within recent years. Among the first on record in this field are Jensen and Parrack (10), who in 1946 used a high-frequency oscillator to follow changes in ionic or dipole content in ionized or un-ionized solutions and thereby performed analytical titrations. Somewhat later (11) they made a more thorough study of the characteristics of various types of oscillators and concluded that the tuned-plate, tuned-grid type of circuit was most adaptable to analytical work since it could be made highly sensitive and still maintain reasonable stability and be readily brought back into oscillation when necessary.

Since 1946 there has been much further advancement in this field, both in the development of instruments and in their application to analyses of different materials. Fischer (7) described a dielectric meter for liquids, using a conventional capacitance substitution method. He was able to analyze ethanol-water mixtures with this device. Blake (5, 6) described a rectified radio-frequency method for conductimetric titrations and a differential radio-frequency comparator for analysis or preparation of standard solutions. Gent (8) described the measurement of the dielectric constants of liquids by a frequency deviation method, utilizing a crystal oscillator as a standard for continuously checking the frequency of the variable frequency oscillator when air is the dielectric.

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

Arditti and Heitzmann (2) and Anderson, Bettis, and Revinson (1) measured grid current. The instrument of West, Burkhalter, and Broussard (12) is based on the beat frequency changes induced when the composition of the core material of the tank circuit coil is altered. By use of their scheme, changes as small as 10 cycles in 4 megacycles can be measured. Using this instrument, West, Robichaux, and Burkhalter (13) were able to analyze the ternary mixture, water-benzene-methyl ethyl ketone. Hall and Gibson (9) performed titrations, using a bridge method for their high-frequency work. By 1951 the method of chemical analysis by high-frequency instruments had gained sufficient importance to warrant a round table discussion (4) at a national American Chemical Society Meeting. There is now at least one commercially available instrument (Sargent Oscillometer[2]) which measures capacitance and is available with a variety of cells for experimental use.

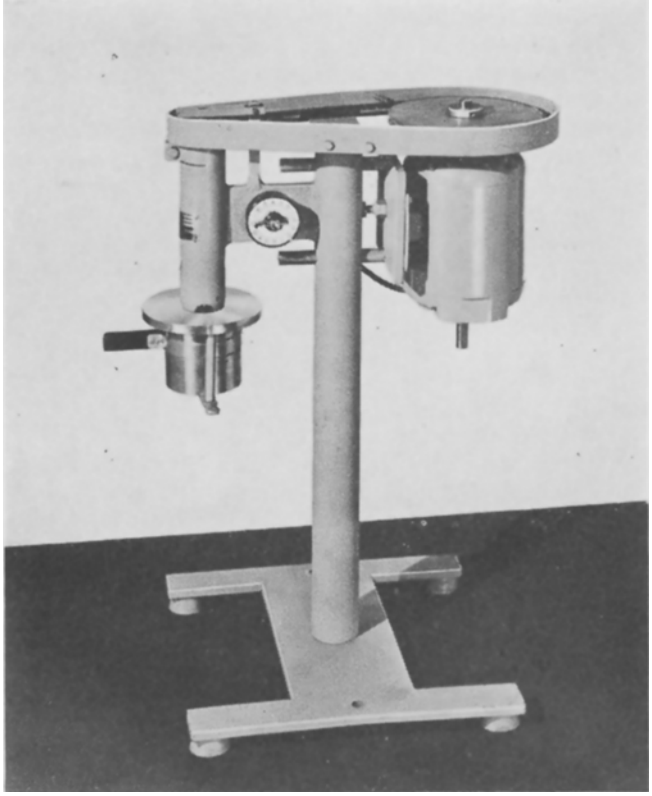
FIG. 1. High-speed grinder-extractor.

## The Application of High-Frequency Oscillation to Analysis of Soybeans for Oil Content

There were three phases to the problem of determining rapidly the oil content of soybeans by high frequency means. It was first necessary to find a good oil-solvent with such properties that it would show a sufficient change in dielectric constant when small increments of oil were added to it, i.e., the finding of a solvent differing greatly in dielectric properties from the soybean oil. The second phase was the development of a sensitive, accurate instrument for measur-

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[1] The study on which these findings are based was made under authority of the Research and Marketing Act of 1946.

[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.

ing dielectric changes. The third phase of the problem of determining the oil content was to develop a high-speed grinder-extractor for preparing the solvent-oil solution. This paper describes the successful development of these instruments and the method of analysis.

*The Stein Laboratory Mill.*[2,3] For the grinding of soybeans and simultaneously extracting the oil from them with an oil solvent, many commercially available devices were tried that were not successful because of solvent leakage in the blender type of instruments or lack of power in the overhead type of drive. However the Stein Laboratory Mill, which was developed specifically for this purpose, has been found to be very satisfactory. This grinder-extractor 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., ½ H.P. motor. The sample and solvent are contained in an aluminum cup. This grinder-extractor is capable of grinding 100 grams of soybeans in 100 ml. of solvent so fine that more than 98% of the residual meal will pass through a U. S. Standard No. 45 woven-wire cloth sieve.

*The Steinlite LOS Unit.*[2,3] 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.

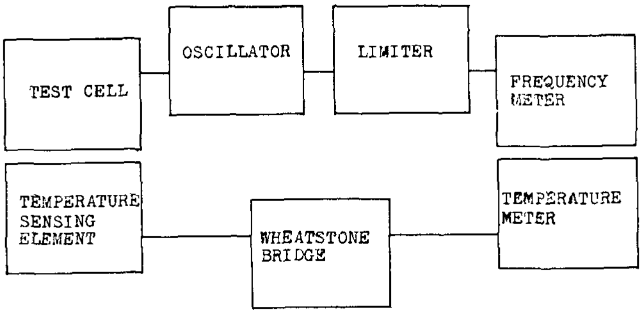

Fig. 2. *Block diagram of the electronic tester.*

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 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, coaxial-cylinder cell.

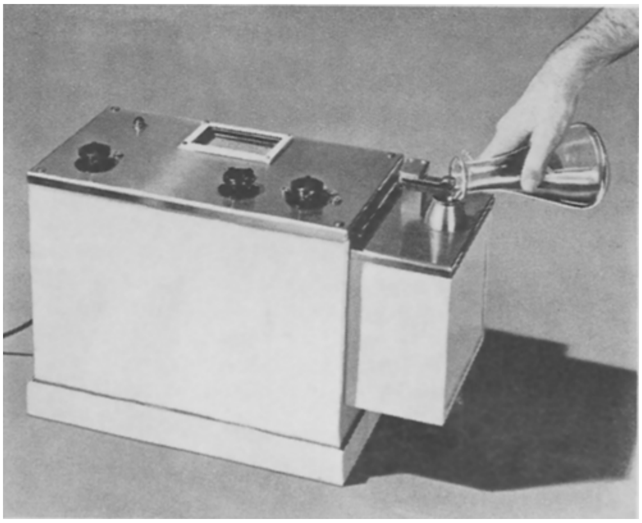

Fig. 3. The electronic tester.

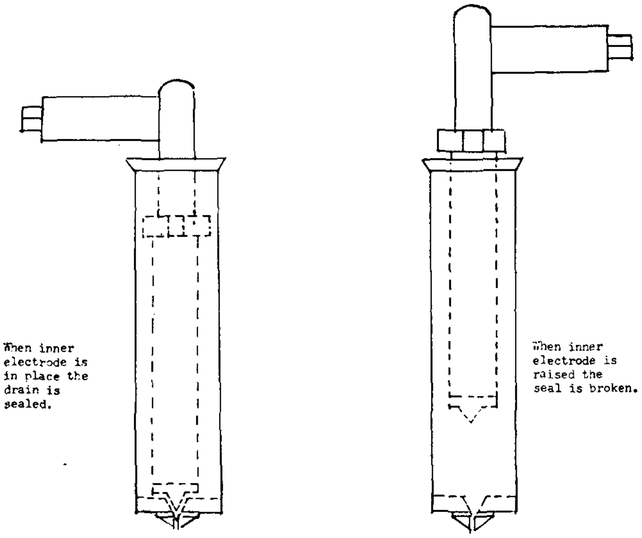

Fig. 4. Two views of the condensor, coaxial-cylinder cell.

Temperature is an important factor in dielectric measurements of liquids and 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.

*Analytical Procedure.*

1. Turn on the LOS unit and allow it to warm up for at least 15 minutes before taking any readings.

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[3] These instruments manufactured by the Fred Stein Laboratories, Atchison, Kans.

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 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 grams 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 ½ 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,[4] 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% should be partially predried. The best moisture level is around 8 to 12%. Care should be taken not to dry the samples below 7% moisture. When it is necessary to predry the soybean sample, the use of an infra-red radiant heater will drive off about 5% of moisture in 5 minutes and will not harm the soybeans for use in the test. If time is not a critical factor, placing the samples in an air oven at 130°C. for one hour will dry them sufficiently. An infra-red heater is being designed especially for use in this procedure.

d. Soybeans having a moisture content below 7% pose a special problem. Their moisture content cannot be accurately determined by electrical means and also a shorter grinding-extraction time is necessary (3 minutes).

*Experimental Data and Results.* Four criteria were established for the selection of a suitable solvent for this procedure, namely: a) it should be a good oil solvent; b) the specific gravity should be such as to hold the soybeans in uniform suspension during grinding-extraction; c) the dielectric constant should differ as much as possible from that of the oil; and d) the boiling point should be high so as to minimize evaporation loss during grinding-extraction. Many solvents were tested and of these orthodichlorobenzene (technical grade) proved to be the one most nearly fulfilling the criteria. Since the technical grades of orthodichlorobenzene tend to vary in their physical properties, especially in dielectric constant, it is necessary to blend different grades of orthodichlorobenzene to produce a solvent reading of 45 on the "A" button.[4]

The feasibility of using the electronic tester for determining oil content in a solvent-oil mixture was determined by preparing mixtures of orthodichlorobenzene and soybean oil corresponding to the quantity of oil likely to be encountered in normal soybeans. Then preliminary testing of methods for grinding and extracting soybeans was performed and the proper method was adopted.

In order to test the accuracy of the new method 86 samples of soybeans ranging in oil content from 18.2% to 23.3% were analyzed under varying conditions of time and temperature of extraction, using two electronic testers for making readings under varying and at constant room temperatures. From two 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.

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

Figure 5 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

A conversion table 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 correlation was found to be + 0.98.

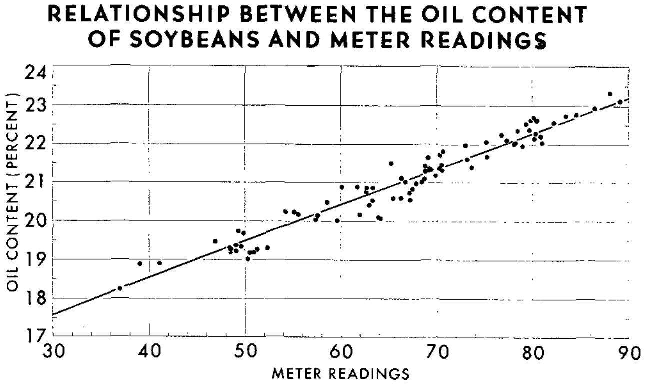

FIG. 5. Relationship between oil content of soybeans and meter readings.

*Discussion.* The method of analysis described above appears to be quite suitable for practical application to the rapid determination of the oil content of soybeans. Results on a single sample can be obtained in about 15 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 30 samples per hour.

The technique of analysis is simple enough so that nonchemists 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, Kans. They have worked closely with this laboratory in developing the method, and we are deeply grateful to them for their aid in designing and redesigning the equipment, when necessary, to meet the needs of the tests. These instruments are covered by patents or patents pending (14).

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# The Isolation of Monoglycerides from Lard and from Bread[1]

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## SECTION A

### General Procedures, Results, and Discussion

MANY experts in the field of fats have concluded that natural edible oils (at least before extensive refining) contain up to 1% of monoglycerides (1, 6-7, 9, 11-16). These conclusions were based upon the expected chemistry of fats and upon such properties of the fat as acetyl values, and periodic acid oxidation studies. Furthermore, because the main components of fats are triesters of glycerol, it seems likely that some hydrolysis will take place during cooking of a food that contains fat, and that cooked products will contain an appreciable amount of monoglycerides. Monoglycerides have been reported as being formed from a triglyceride shortening during the baking of bread (5).

This report describes a study of the monoglyceride content of lard and of baked bread by actually isolating the monoglycerides, using techniques similar to those of Jones and co-workers (10), who isolated monopalmitin from pancreas.

### General Procedure

*Extraction and Purification of Monoglycerides.* To isolate the monoglycerides, methods were chosen which involved only solvent extraction and crystallization. The following procedure combines the best features of the several modifications presented in the experimental section. The lipids are removed by stirring with large volumes of warm petroleum ether (Skellysolve F). The Skellysolve F is preferred as a solvent to an ethyl ether-ethanol mixture since, in the extraction of bread, ether-alcohol removes large quantities of sugar, flour, and starch products. The fat recovered from the Skellysolve F extract, after removal of the solvent, is further worked up by the following procedure which is suitable for most fats. The extracted fat is first treated with 10-20 volumes of acetone. The phospholipids which are present in the fat precipitate and are removed. After removal of the acetone the fat is dissolved in 20 parts of ethyl ether and washed with distilled water. Vigorous shaking

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[1] Communication No. 186 from the laboratories of Distillation Products Industries. Presented at the Houston Meeting of the American Oil Chemists' Society, April 29, 1952.