Development of Buckeye Moisture Meter for Use on Oil-Bearing Materials

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ONE of the important variables in the storage and processing of oil seeds and related materials is moisture. A rapid, accurate moisture determination is required for the segregation of that portion of high moisture seed receipts which experience has shown cannot be stored without quality losses unless they be dried before storage. Oil extraction efficiency is directly related to moisture content of the prepared meats; and finally the control of moisture in meal or feed to an established uniform level is required for product quality.

A complete definition of the requirements for moisture determinations may be stated as follows:

1. *Rapid Method.* On-the-job analyses should require a total elapsed time of approximately 2-3 minutes.
2. *Accurate Within Practical Limits.* This will vary in different cases from ± 0.1% to ± 0.5%.
3. *Simple.* The method should be such that a non-technical operator can satisfactorily obtain reproducible results with the desired degree of accuracy.
4. *Inexpensive.* a) The method should be inexpensive to operate to the extent that the trend will be for more frequent moisture determinations instead of reduction in number to reduce costs. b) The initial cost and maintenance costs should be low to make practical the use of several meters throughout a plant at the sample station locations.

Early Work. The above listed requirements are not new. They have been recognized for many years. With the advance in instrumentation in industry it was only natural that the answer would be sought in the form of a meter or instrument into which a sample could be inserted; and instantaneously the moisture would be indicated. Twenty-three years have been spent within our company in research and in development of a meter which will meet these requirements.

The early tests were successful; and within a short period of time the meters were placed in general use. These meters have been used within the company for 17 years.

Reason for Publishing Work. The Buckeye Cotton Oil Company has, through work covering 23 years, developed a moisture meter which provides the means for a rapid and accurate moisture determination on oil-bearing materials. In addition, the meter is simple to operate and maintain. This, with a low initial cost, has led to its general "on-the-job" employment for process control, storage control, and product quality control.

This meter has proved to be very valuable in the processing of oil-bearing materials. Interest has been displayed by numerous other companies in the vegetable oil industry as well as in the milling industry. Because experience has shown this instrument to be adequate to meet the needs of our industry, it is our desire to make available the complete information on the development and final specifications for this moisture meter to all members of industry to whom such a meter might be of value.

In the remainder of this report references to samples and material upon which moisture determinations are made refer specifically to oil-bearing materials of a granular nature such as soybeans, cottonseed, cracked soybeans, soybean meal, and copra meal.

Theoretical Considerations

In order to design a moisture meter, it was necessary first to establish some characteristic of the materials to be analyzed which will vary with the moisture content and which can be measured with a consistent degree of accuracy. It is essential that the chosen characteristic be primarily or grossly dependent upon the moisture content and that other influencing factors can be compensated for, or that the other influencing factors are of such lesser degree of magnitude that their total effect on the variation due to the moisture content will be negligible.

In early work there were four possible measurements which appeared to satisfy these requirements:

1. Dielectric constant of the materials
2. D.C. resistance of the materials
3. Loss in weight of a sample after drying
4. Relative humidity of a gas surrounding a sample at the moisture equilibrium point.

Of these four methods the last two were not considered to be rapid enough for use with a relatively large sample, which was necessary in order to eliminate the sampling errors.

Some preliminary work done in 1929 and rechecked in 1933 indicated that the measurement of the dielectric constant was more reliable than the measurement of d.c. resistance. From then on the entire efforts were concentrated on the dielectric-type meter.

Recently there has been some consideration of a study of the method involving gamma ray or 1 cm. radio wave penetration of a sample. However, inasmuch as this is a relatively new field, it was not desired to undertake the extensive research required at this time.

As soon as the dielectric-type meter had been selected, the theoretical effect of temperature upon the dielectric constant became a matter for consideration. There appeared to be three methods of compensation for the temperature effects:

1. Fully automatic compensation within the circuit
2. Semi-automatic compensation with the correction set into the circuit manually after the temperature of the sample has been determined
3. Correction of meter reading by use of graphs or charts giving calibration over normal range of sample temperatures.

These methods will be described further in the discussion of the circuit. However the choice was made to eliminate automatic or semi-automatic temperature compensation in the interest of keeping the circuit as simple and trouble-free as possible. The advantage of the automatic compensation is that it eliminates human error. However this would be of value only if a completely reliable compensator could be built into the circuit which would accurately compensate for wide variations in temperature for a number of different materials. Automatic compensation is not of value if a meter is used on a material which does not vary in temperature more than about ± 5°F. In actual practice a large percentage of the applications were such that temperature compensation was not required because the samples were consistently uniform with respect to temperature.

Investigation of Various Circuits

When any insulating material, other than a vacuum, is placed between the plates of a condenser, the capacity of the condenser is changed with respect to the capacity in the absence of the material by a factor referred to as the dielectric constant. The dielectric constant of a material such as an oil seed meal was found to be constant, for practical consideration, at a given temperature when the material was bone-dry. Addition of moisture changed the dielectric constant by a measurable degree. Therefore it became evident that construction of a moisture meter required that an accurate method of measuring the dielectric constant of the material be developed.

The first meter built in the course of this development work which was successful in use to detect moisture changes in a sample was a simple regenerative circuit which was designed based upon a circuit published by Burton and Pitt in 1929 (1). The circuit adopted for the first meter was different from this circuit in that a grid leak, grid condenser, and a by-pass condenser were added, improved regeneration control was accomplished through a controlled cathode bias, and a power pack was added for A.C. operation in place of batteries.

Whereas this meter was termed a dielectric type, this is not strictly true in that the coil sample holder is affected not only by the dielectric loss but also by the change in the mutual inductance of the coils. The effect of inserting a dielectric in the field of the coil sample holder is to increase the distributed capacity of the coil. As the distributed capacity increases, the losses increase. These losses are the same as a resistance placed in series with the coil.

During the years following the construction of the first meter there were numerous changes made to improve the circuit. The first series of changes were incorporated in a meter circuit shown in Figure 1. In the original meter the bias resistor was critical, controlling the regeneration and the bias. This was changed (Figure 1) by placing the bias resistor at R_3 and bypassing it with condenser C_3. This gives a fixed bias control independent of regeneration. Regeneration control was obtained by using a variable resistor R_4 in series with condenser C_4. This arrangement also eliminated the problem of having the RF choke in a critical position. In this improved meter the sensitive range was varied by the regeneration control R_4.

In addition to these basic changes other improvements were: the conversion of the meter to battery operation, except for the filament transformer, to eliminate undesirable effects of large line voltage changes; and the inclusion in the circuit of built-in standards. The built-in standards consisted of a short circuited coil inductively coupled to the regenerative circuit. By controlling the resistance of the short circuited coil, it was possible to control the energy loss of the coil to values approximately those obtained when samples with various moisture contents

TABLE I

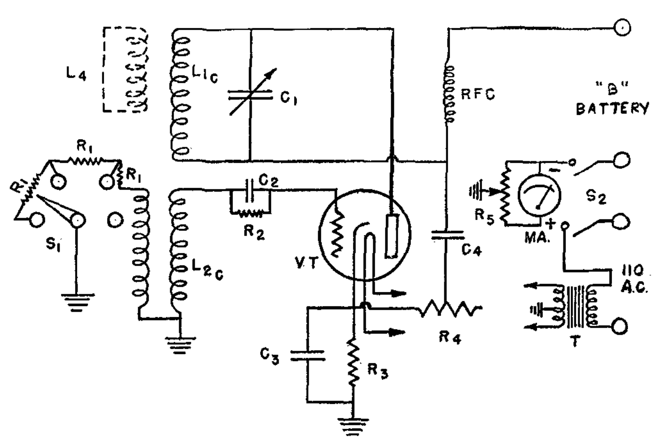
Comparison of Oven and Meter Moisture Determination Soybean Receipts

No. Freight-Cars	% H_2O Oven	% H_2O Meter
21	12.2	12.3
44	12.5	12.4
2	12.6	12.5
101	12.6	12.7
1,400	12.8	12.8
64	12.8	12.9
7	13.0	13.0
52	13.1	13.1
30	13.1	13.1
32	13.1	13.1
74	13.3	13.4
10	13.3	13.3
3	13.4	13.6
5	13.5	13.5
4	14.6	14.2
1,849*	13.1†	13.1†

* Total.
† Average.

were placed in the test cell. These standards are shown in Figure 1 by L_3, R, and S.

It was evident that a more sensitive circuit was required to respond to small changes in moisture and to samples with low moisture content. A portion of the work dealt with the further improvement of the meter shown in Figure 1. It was possible, with the meters available, to determine with a relative degree of accuracy the moisture content of the various oil seed fractions. In the course of the work the Crosley Radio Corporation was contacted to build meters in larger quantities to supply the company's needs. Working from the meter design shown in Figure 1, the decision was made to substitute a voltage regulated power pack for the "B" batteries. When this



R1 - 50 OHMS (IRC - TYPE F-1)
R2 - 10 MEGOHM (IRC - TYPE F-1)
R3 - 200 OHM (IRC - TYPE F-1)
R4 - 50 OHM POTENTIOMETER (ELECTRODE RT 271)
R5 - 50 OHM POTENTIOMETER
C1 - 200 MMFD CONDENSER
C2 - 250 MMFD CONDENSER
C3 - 0.5 MFD CONDENSER
C4 - 2000 MFD CONDENSER
RFC - RADIO FREQUENCY CHOKE (CH-8)
T - FIL. TRANS. 2.5 VOLTS
S1 - SINGLE GANG 5 PT. SWITCH
S2 - TOGGLE SWITCH
VT - C-56 VACUUM TUBE
L1C - TICKLER COIL, 3/4" DIA. 65 TURNS #22 DCC
L2C - GRID COIL 3/4" DIA. 52 TURNS #22 DCC 3/8
L3 - 13 TURNS 1 1/2" DIA. SPACED 3 1/2 TURNS PER INCH
L4 - 38 TURNS WOUND AT TOP END OF L1C
MA - MILLIAMETER

FIG. 1

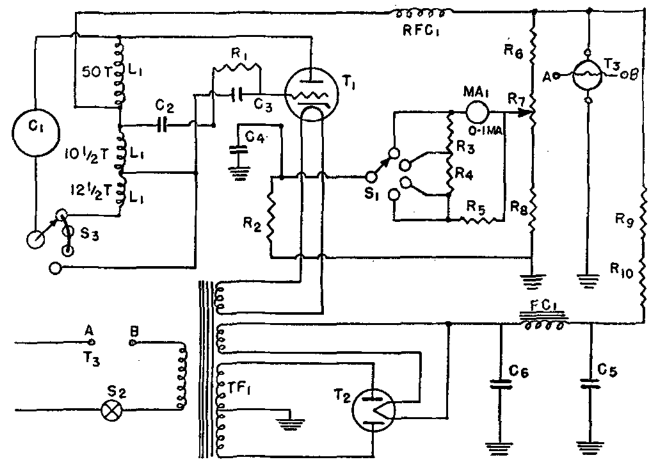
was done, together with accompanying required circuit changes, the meter would not oscillate. Crosley Radio Corporation engineers thought the circuit stability could be improved, and work was undertaken to design a new circuit.

The final design of the meter developed with Crosley Radio Corporation engineers had several major changes. The circuit is shown in Figure 2. The principal changes were:

	Early Meter Figure 1	Redesigned Meter Figure 2
*1. Power supply	"B" batteries	Power Pack
*2. Oscillating circuit	Modified tuned plate	Modified Hartley
*3. Sample holder	Conductance coils	Condenser plates
*4. Method of changing sensitivity	Change in frequency	System of shunts around milliammeter
5. Stability	Variable circuit	Constant circuit

The most important changes are indicated by asterisks. The advantages of these changes are:

1. *Power Supply.* The power pack will give a cheaper source of current than the "B" batteries and eliminate the problem of battery replacement.
2. It was decided that the modified Hartley oscillating circuit was more stable than the modified tuned plate circuit.
3. *Sample Holder.* The use of condenser plates for a sample holder is superior to the use of the two coils because a sample within the plates of a condenser affects the circuit by its dielectric loss. The coil sample is affected by dielectric loss and the change in mutual inductance of the coils. Further, changing the dielectric between the condenser plates will not greatly affect the frequency of oscillation, but a similar change between the coils makes a great change in frequency. For the most stable operation the frequency of oscillation should be constant.
4. *Changing Sensitivity.* The redesigned meter employed a system of shunting various amounts of the total current around the milliammeter in order to change the range of sensitivity of the meter. This was possible because, as described in "3" above, the frequency of oscillation was constant. In the earlier meters the range of sensitivity was changed by changing the frequency and violence of oscillations. This change served to eliminate three adjustments which simplified the operation of the meter.
5. *Stability.* The redesigned meter was superior to the early model because of its greater stability.



C1 – MOISTURE IND. CAPACITOR, .005" BRASS SHIM STOCK 5/8" WIDE, 4 7/8" LONG.
L1 – OSCILLATING COIL.
C2 – .02 MFD, 200 V. CONDENSER.
C3 – .0001 MFD CONDENSER.
C4 – 0.1 MFD CONDENSER.
C5 – 8.8.6 MFD CONDENSER.
C6 – 8.8.6 MFD CONDENSER.
R1 – 500 000 Ω RESISTOR, 2 W.
R2 – 500 Ω 1/2 W, RESISTOR.
R3 – 500 Ω 1/2 W, RESISTOR.
R4 – 275 Ω 1/2 W, RESISTOR.
R5 – 275 Ω 1/2 W, RESISTOR.
R6 – 30 000 2 W RESISTOR.
R7 – POTENTIOMETER 200 Ω.
R8 – 220 Ω 1/2 W, RESISTOR.
R9 – 4400 Ω RESISTOR.
R10 – 2600 Ω RESISTOR.
RFC1 – RF CHOKE 2.5 MH.
FC1 – FILTER CHOKE.
T1 – NO. 76 TUBE.
T2 – NO. 80 TUBE.
T3 – NO. 874 TUBE & SOCKET
TF1 – POWER TRANSFORMER, 6.3 VOLTS
S1 – A 81 SWITCH
S2 – LINE SWITCH
MA1 – PLATE METER, 0-1 MA

FIG. 2

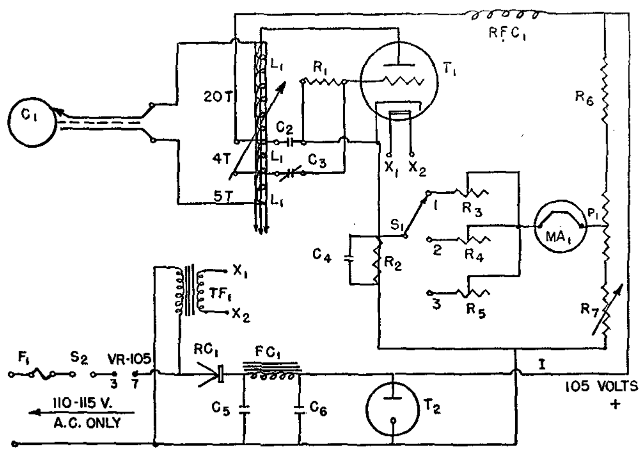
The redesigned meter was satisfactory for use on all of the various oil seed fractions, with the exception of those with very high moisture content (17% and above). The development work continued on this circuit however; and over a period from 1935 to the present time several changes were incorporated. These generally were made as small improvements; and therefore to trace each one would not be practical. Resulting from this work, which involved cooperative work with several companies engaged in the construction of electronic equipment, a circuit has been developed which was adopted as a standard meter for the Buckeye Cotton Oil Company. As would be expected, the suggestions for changes in the circuit came from many sources within and outside of the company. Several of these were made by companies which constructed meters for the Buckeye Cotton Oil Company.

The final circuit which has been adopted as a basic standard for the Buckeye Meter is shown in Figure 3, together with a complete parts list.

Analysis of Circuit. The basic circuit used is that of a modified Hartley oscillator in which the test cell serves as the capacitive branch of the parallel resonant network. Because the materials to be tested (oil seed fractions) are in the nature of poor dielectrics, the cell behaves as an imperfect condenser having an equivalent circuit consisting of a series or parallel combination of pure capacity and pure resistance, in which both quantities are non-linear functions of the moisture content and temperature of the material to be tested. The resistance represents the magnitude of the dielectric losses.

Circuit analyses may be simplified by consideration of only the major effects of variation in the magnitudes of the parellel resistance, inductance, and capacity network. In addition, it may be assumed that the dielectric constant and the dielectric losses are essentially independent of the frequency within reasonable limits. On this basis it may be stated that the relative magnitudes of the reactive elements (capacity and inductance) serve primarily to determine the resonant frequency of the network whereas the magnitude of the losses (resistance component) governs the relative response in the vicinity of resonance. When such a network is incorporated in a vacuum tube circuit, it is the reactive components that dictate the frequency of oscillation. The resistance component exercises an effect on the amplitude of oscillation. The ultimate effect of unduly large losses in the resonant network is the cessation of oscillation.

In the circuit a d.c. milliammeter is used as a detector and indicator. This milliammeter detects variations in the amplitude of the oscillations by measuring the average d.c. current flowing in the cathode circuit of the vacuum tube. This method of detection places no additional loading on the resonant network and therefore does not act to impair performance. It is possible to null the meter for such d.c. current as flows under those conditions chosen as reference



C_1 – MOISTURE CAPACITOR, .005" BRASS SHIM STOCK 5/8" WIDTH, 4 7/8" LENGTH.
L_1 – OSCILLATING COIL BUILT ON 3/4" FORM WITH VARIABLE SLUG.
C_2 – .01 MFD, 200 VOLT CONDENSER.
C_3 – 100 UU FD, CONDENSER.
C_4 – 0.1 MFD CONDENSER.
C_5 – 10 MFD CONDENSER.
C_6 – 10 MFD CONDENSER.
R_1 – 470 M Ω RESISTOR, 1/2 W
R_2 – 500 Ω, 1/2 W RESISTOR.
R_3 – 6000 Ω, 1 W RESISTOR.
R_4 – 6000 Ω, 1 W RESISTOR.
R_5 – 6000 Ω, 1 W RESISTOR.
R_6 – 30000 Ω, 2 W RESISTOR.
R_7 – 1000 Ω, 2 W VARIABLE RESISTOR.
P_1 – 200 Ω, 2 W POTENTIOMETER.
MA_1 – 0-1 M AMPERES, PLATE METER.
T_1 – 6J5 TUBE.
T_2 – VR-105 TUBE.
FC_1 – 8 MH
RFC_1 – RF CHOKE, 2.5 MH
RC_1 – HALF WAVE RECTIFIER
TF_1 – FILAMENT TRANSFORMER 6.3 VOLTS.
S_1 – A-81 SWITCH.
S_2 – LINE SWITCH.
F_1 – AG 3 BUSS FUSE
COAXIAL CABLE – TYPE RG 59 U

FIG. 3

through the potentiometric arrangement between the d.c. voltage supply and the cathode circuit of the tube. Action of the circuit is such that any increase in losses in the resonant network produces a corresponding increase in the average d.c. plate or cathode current. A gaseous voltage regulating tube is used to maintain the reference voltage for the detector circuit and the plate voltage for the vacuum tube oscillator reasonably stable with normal line fluctuations.

As is customary in power oscillators, the necessary biasing is obtained by the grid-leak method. This method is advantageous for use in a moisture meter circuit as there is no shift in the operating point of the vacuum tube which changes the average plate or cathode d.c. current. The grid return is made directly to the cathode rather than through the resistance network in the cathode circuit.

Effect of Variables

In operation of the meter there are certain variables other than the moisture content of the sample which will affect the circuit. These are temperature of the sample, density of the sample, and particle size, and, of course, the cleanliness of the meter and sample tubes.

The temperature effect is non-linear. The effect is as follows. For any sample of material at a constant moisture content, the losses in the resonant circuit will increase with increasing temperatures. Further these losses due to temperature increase are not constant but vary in magnitude with variations in the moisture content of the material. Therefore any method of compensating for the temperature effect would have to accomplish appropriate modulation of the signal amplification or detector sensitivity or introduce some compensating resistive element in the resonant circuit. The primary difficulty is the devising of a circuit to provide a modulating signal that is a non-linear function of both percentage moisture and temperature. Possible circuits or components which might affect the desired modulation are:

1. Non-linear resistors of negative temperature co-efficient
2. Operation of vacuum tubes on the parabolic portions of their characteristics with special consideration given to variable mμ tubes.
3. Special vacuum tube circuits exhibiting negative resistance characteristics
4. Reactance tube circuits. Actually no work has been done on these circuits. An attempt was made to employ a variable condenser in the circuit to compensate for temperature effect. For one type of material a condenser can be hand-trimmed and calibrated so that the major portion of the temperature effect can be compensated for by manual control. This proved to be satisfactory for normal ranges of moisture and temperature. However the use of manual compensation does not eliminate personnel errors and could introduce added errors by improper use or by introducing additional components which are subject to functional irregularities. For the standard Buckeye Meter the temperature compensation is accomplished through the use of graphs with the moisture *vs.* meter reading curves plotted for each 10°F. over the normal range of temperature for the materials tested. Figure 4 shows this temperature effect on the curves for soybeans.

Density of the material to be tested is another variable which affects the meter readings. For a given material the losses in the resonant circuit increase as the density increases. This means that the loading of the test cell is an important operation and should be carried out in such a way as to insure uniform density for each sample. In actual practice the loading of the test cell is not complicated. A procedure such as tapping the cell gently three times on a rubber mat is generally adequate to give uniform density to each sample. The density is also affected by the particle size of the material to be tested. It is necessary to have calibration curves for each form of the materials tested. For example, whole soybeans and ground raw soybeans will have slightly different curves. Experience has shown that, in general practice in process control, the particle size of a fraction does not vary enough to require more than one calibration curve.

Cleanliness of the meter and sample tubes is important. In any electrical circuit the values of certain of the component parts may be affected by the accumulation of dirt, moisture, or dust. The test cells also will change in dielectric value if they become dirty or pick up moisture.

Operation of Meter

Following the initial warm-up period required for all vacuum tube circuits, the meter in plant use is normally left turned on continuously throughout periods when samples are to be run. This may be a matter of hours, days, or weeks. When a sample is to be run, the following procedure is followed:

1. The meter is zeroed with a clean, dry, empty sample tube in place in the test cell.
2. The sample tube is removed from the test cell, the sample is poured into the tube, and the tube is tapped gently three times on a rubber mat or stopper to compact uniformly the material in the tube. The tube is then reinserted into the test cell.

CALIBRATION CURVES FOR SOYBEANS

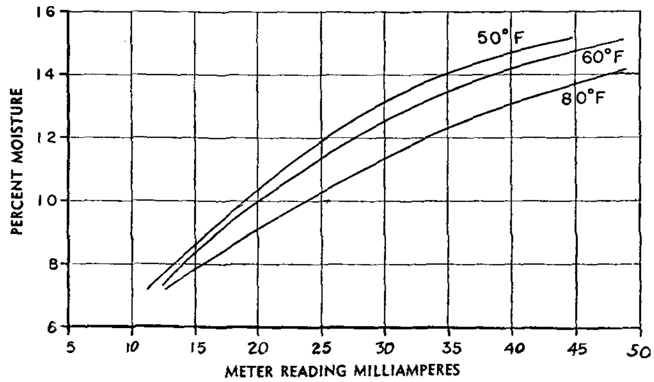


FIG. 4

CALIBRATION CURVES FOR COTTONSEED

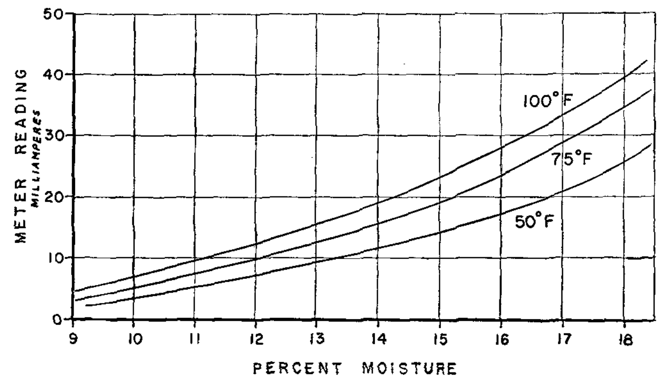


FIG. 5

3. The meter deflection is noted and recorded.
4. Temperature of the sample is noted and recorded. This may be obtained by one of several methods. One of the most common is the insertion of a quick response thermometer into the test cell immediately after filling, noting the maximum temperature, and removing thermometer before the tube is placed in the test cell.
5. Moisture content of the sample is read from the graph or tables, using the proper graph or table for the temperature of the sample.

The technique of operation of the meter varies considerably with different operators. The primary concern is that the operator or operators using a single meter follow standard procedure which will permit reproduction of their results. In other words, the basic accuracy of the meter is fixed; but the operator technique can affect the overall accuracy of the meter tests just as poor technique can affect the moisture determination by the oven method.

Calibration of Meter. The normal procedure for calibration of the meter is to set up a series of samples of the desired material with moisture content varying in 2% steps over the range normally encountered. These samples are read on the meter at different temperatures in 10°F. intervals. Each time a sample is read on the meter, a sample is withdrawn for check oven moisture. A series of three or four readings for each moisture content at each temperature is sufficient to give data to prepare graphs for use with the meter.

A continuous calibration is run on all meters simply by having one sample checked per day by the oven moisture method. These check analyses serve to provide additional calibration data and also serve to detect when the meter is in need of repair.

Results

The Buckeye Meter has been successfully used over a period of several years on whole soybeans, cracked soybeans, soybean meal, cottonseed, cottonseed meats, cottonseed meal, sesame seed, sesame meal, and copra meal.

Typical response curves are shown in Figure 4 for soybeans and Figure 5 for cottonseed. These curves are generally the same for all meters of the same circuit design; however, in practice, each meter is checked for calibration prior to its use.

The basic accuracy of the circuit should be in the range of ± .1% moisture when the meter is properly constructed and operated. In actual use the accuracy may be in the range of ± 0.25%. This appears to be a rather large deviation, but it is actually very close to the standard deviation of a single moisture determination run in an oven (1 hr.). Reasons for the deviations are, of course, the technique of the operator, accuracy with which the temperature is read, uniformity of packing of sample, and cleanliness of the sample tube. With care in operation and by reading a sample three times and using the average of the three readings, the accuracy of the determinations can be improved. In practice, the required accuracy dictates the precautions to be taken in the operation of the meter.

Table I is a consolidation of the results of checking samples from 1,845 freight cars of soybeans by oven and by Buckeye Meter. The greatest difference recorded was 0.4% H_2O between the two methods. Reliability of the meter moisture as referred to the oven moisture is demonstrated by the weighted average of all results, there being .01% difference between the two, as well as the averages of the various groups representing shipments, which checked to within 0.1% in all but one case. These data are furnished in substantiation of the earlier statement that the Buckeye Meter has an accuracy roughly equivalent to the standard deviation of single determinations run by the one-hour oven method.

Summary

The development of the Buckeye Moisture Meter has covered a period of 23 years during which time considerable work has been done within the company on this meter. In addition, there have been numerous contacts outside of the company who have contributed to the development of these meters. These have been companies or individuals who were retained to construct or analyze the meter. Among these outside contacts are the Crosley Radio Corporation, which built some of the earlier models; A. D. Little Inc., who, in a consulting capacity, analyzed the circuits of some of the meters; and two companies who have in recent years constructed these meters for the Buckeye Cotton Oil Company. These companies are: Industrial Engineering Company, Louisville, Ky., and Moisture Register Company, Alhambra, Calif.

The meter, which has been adopted as a standard within the Buckeye Cotton Oil Company, has proved to be highly satisfactory for process control work. The accuracy of the meter in plant use has been

found to be roughly equivalent to the standard deviation of single oven determinations. Certain variables must be compensated for either by method or technique of operation; namely, temperature of sample and density of material in sample tube. The precautions required are determined by the accuracy desired.

The meter has been found to fill a very necessary function in the process control in the milling of oil-bearing materials. The initial cost has been kept at a minimum by designing the meter for specific application, and further the operating and maintenance costs are extremely small. Because of the success we have had with this meter and because experience has shown it to be a valuable process tool, it has been made available to industry for use in whatever application it may be found to serve a useful function.

[Received October 27, 1952]

Sesamolin Adsorption by Bleaching Agents

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THE Indian oil technologists engaged in the manufacture of Banaspati (edible hydrogenated oil) have to face the problem that certain bleaching earths, although effective in reducing the color of the oil, decompose sesamolin. Most of the bleaching earths produced by manufacturers in sterling areas are of this type, and this has presented a new problem to the industry. A bleaching earth which causes a loss of only a small amount of sesamolin is acceptable, but if it removes the sesamolin completely, either by adsorption or splitting, it should not be used.

With the introduction of the Baudouin test in India as a measure of the sesame oil present in Banaspati, the effect of the bleaching earths and carbons used in processing on the values obtained in the test are of fundamental importance. The characteristic Baudouin test is caused by sesamol ($C_7H_6O_3$), a component of sesamolin ($C_{20}H_{18}O_7$), one of the unsaponifiable components present in sesame oil but not present in other natural oils (1).

Honig (3) showed that acid bleaching earths can lead to the formation of free sesamol. Budowski *et al.* (2) showed that neutral clay and activated carbon, as well as acid clay, can produce free sesamol from sesamolin and can reduce the total of the free and combined sesamol of sesame oil. They also found that nearly all the free sesamol is lost during deodorization of the oil.

This investigation was undertaken to provide additional information concerning the effect of various bleaching clays on the sesamol content of oils for the benefit of manufacturers of hydrogenated oil.

Experimental

1. *William Garrigue Plant.* Twenty-five tons of crude ground-nut oil mixed with 7-10% sesame oil were refined with caustic soda at 60°C. in a steam-jacketed neutralizer equipped with a mechanical agitator. After refining, the oil was settled for 8 hours and siphoned off to a wash tank where it was given several washes with hot water to remove soaps. The amount of sesame oil added to different batches was varied, and the Baudouin tests were conducted on both the crude and refined oils of each batch. The washed oil was bleached in a 12-ton soft bleacher for 1 hour at 90°C. under 27.5 in. vacuum. The effect of different earths in various proportions was noted. The bleached, filtered oil was hardened to a melting point of 37°C. in a 9-ton autoclave. One pound of activated carbon per ton of oil charged was added with the catalyst. The cooled, filtered, hardened oil was given a light alkali wash and several hot water washes. The oil was then treated with different quantities of bleaching earths, and the decrease in Baudouin test color was noted. The bleaching conditions were similar to those used in the soft oil bleacher. The oil was deodorized in a 9-ton deodorizer for 4 hours at 150 to 160°C. under 29.5 in. of vacuum.

TABLE I

Effect of Refining Sesame Oil and Bleaching With 0.5% Clay on Color and Baudouin Test Readings

	Bleaching earth, %	Color	Baudouin test color
1. Crude sesame oil (F.F.A. 2.0%)......	......	4.5Y-0.5R	12.5R-1.0Y
2. Oil after neutralizing......	......	....	11.6R-0.9Y
3. Bleached oil a) Fulmond-525 C......	0.5	2.0Y-0.2R	9.5R-0.9Y
4. Bleached oil b) B.C. clay......	0.5	2.0Y-0.2R	10.6R-0.9Y

2. *Laboratory Experiment.* Sesame oil and different sesame oil mixtures were bleached in the laboratory in 1-lb. batches at 90°C. for 20 minutes, using mechanical agitation and the Baudouin test color noted.

Following the procedure of the Baudouin test, sesame oil is mixed with liquid paraffin as a diluent in the ratio 1:80. Five ml. of this mixture is slightly warmed in a test tube. To this is added 5 ml. concentrated HCl (Sp. Gr. 1.19) and 8 drops of a 2% solution of freshly distilled furfural in neutral alcohol. The test tube is shaken for 2 minutes, and the pink acid layer is allowed to settle for 8 minutes. The acid layer is filtered through a Whatman filter paper, and the filtrate is collected in a 1-cm. cell of a Lovibond Tintometer (British Drughouses Pattern, made in England, Patent No. 299194).

The experimental results obtained are tabulated in Tables I to IV.

In the case of pure sesame oil the dilution in the Baudouin test was 20 times greater than that used for the processed oil.

Discussion of Results

Examination of the data for the plant scale experiment using Fulmond earth in Table III shows that at the end of the processing the sesamoline was virtually completely removed. The data indicate that the Fulmond earth used in the soft oil bleacher split some of the sesamoline into sesamol, which was partially destroyed in the hydrogenator, and the Fulmond earth used in the hard oil bleacher split still more of the sesamoline into sesamol, which was lost during the deodorization. Thus the double bleaching with