EFFECT OF ULTRASONIC INSONATION ON SOLVENT EXTRACTION OF OIL FROM PEANUTS

by

Donald Gene Sutherland

Thesis submitted to the Graduate Faculty of the
Virginia Polytechnic Institute
in candidacy for the degree of

MASTER OF SCIENCE
in
Chemical Engineering

APPROVED:

Louis A. Pardue
Director of Graduate Studies

J. W. Whittemore
Dean of Engineering

APPROVED:

Frank C. Vilbrandt
Head of Department

Dudley Thompson
Supervisor or Major Professor

November, 1953

Blacksburg, Virginia

TABLE OF CONTENTS

LIST OF TABLES

Page

LIST OF FIGURES

Page

I. INTRODUCTION

The movement of one or more components of a system between phases occurs in many unit operations and is known as mass transfer. The extraction of solute from an inert solid by use of a solvent is one aspect of mass transfer and is known as liquid-solid extraction. A few samples of liquid-solid extraction in industry are the recovery of copper from oxidized copper ores; sugar from sugar beets; tannin from sumac berries; and oil from peanuts, soybeans, tobacco seeds, and fish livers.

Three factors that govern the rate of extraction of solute from a mixture of solute and inert solid by using an extracting solvent are: (1) concentration gradient between solute and extracting solvent, (2) area of contact between solute and extracting solvent, and (3) mass transfer coefficients of solute and solvent films.

Ultrasonic waves produce microagitation and thermal effects in systems containing liquids, particularly at phase boundaries where cavitation is most likely to occur. Austerweil has reported that liquid-solid extraction is facilitated by ultrasonic insonation; however, valid conclusions cannot be made until further investigations are conducted.

The purpose of this investigation was to determine the effects of intensity and time of 400-kilocycle insonation on solvent extraction of oil from peanuts.

II. LITERATURE REVIEW

Literature pertinent to the effects of ultrasonic insonation on extraction of oil from peanuts was divided into the following spheres of interest: (1) mass transfer, (2) liquid-solid extraction, (3) the system peanut meat-peanut oil-hexane, and (4) ultrasonics.

Mass Transfer

Mass transfer is defined(24) as movement of one or more components of a system from one phase to another. Basic aspects of the phenomenon of mass transfer are reviewed in this section.

Mass Transfer Operations. Examples of separation by mass transfer encountered in various chemical engineering operations are absorption of a component from the vapor phase, extraction of a component from a liquid or solid by solution in a solvent, and separation of a volatile component of a liquid from the mother liquid. Absorption, crystallization, extraction, distillation, stripping, humidification, and drying are all examples of unit operations which exemplify mass transfer.

Equipment used for mass transfer operations frequently incorporates a series of stages in which solvent and feed are brought into contact to allow transfer between phases to occur. A solvent recovery system usually supplements mass transfer equipment although not a part of the equipment proper(34,52,56,71,98). Effectiveness of the stage in accomplishing transfer of material depends upon many factors such as design of equipment, physical properties of the phases, and equilibrium relationships.

Molecular Diffusion. Molecular diffusion has been defined(107) as "... the spontaneous intermingling of miscible fluids placed in mutual contact, accomplished without the aid of mechanical stirring." If phases between which mass transfer occurs are stagnant or in laminar motion, the mechanism of transfer is molecular diffusion.

Diffusion in Gases. The relations which describe the process of molecular diffusion for gaseous systems are derived from the kinetic theory of gases. According to this theory(26,75) the resistance to diffusion of gas A through gas B, for ideal gases, is proportional to number of molecules of gas A per unit volume, number of molecules of gas B per unit volume, mean velocity of gas A relative to gas B, and length of the diffusion path. The rate of diffusion per unit area is:

$$N_A = (D_G A/RTx)(p_{A1} - p_{A2}) \qquad (1)$$

where:

N_A = mols of component A transferred per unit time, mol/hr

D_G = diffusivity or diffusion coefficient of gas A, mol/hr-sq ft-mol/cu ft

A = interfacial area, sq ft

R = universal gas constant, ft lb/lb mol-°K

T = absolute temperature, °K

x = length of diffusion path, ft

p_{A1}, p_{A2} = partial pressure of diffusion gas A in phase 1 and phase 2, respectively, lb/sq ft.

Diffusion in Liquids. Mass transfer by diffusion occurs frequently in liquid systems. Because of the lack of a more appropriate formula, diffusion equations similar to those derived for ideal gases are applied to diffusion in the liquid state by replacing the partial pressure potential with a concentration potential. The equation most often used is(26):

$$N_A = (D_L A/x)(C_{A1} - C_{A2}) \qquad (2)$$

where:

N_A = mols of component A transferred per unit time, mol/hr

D_L = liquid diffusivity, mol/hr-sq ft-mol/cu ft

A = interfacial area, sq ft

x = length of diffusion path, ft

C_{A1}, C_{A2} = concentration of diffusion liquid A in phase 1 and phase 2, respectively, mol/cu ft.

Validity of Equation 2 for liquid systems is doubtful in view of the assumptions that the kinetic theory described the liquid state, and that the total number of mols per unit volume of liquid is independent of composition. Because of inadequacy of the kinetic theory when applied to liquids, no generalized correlation of diffusivity for liquid phases has been found; however, empirical relationships are available(26).

Diffusion in Solids. Diffusion equations have been applied with dubious success to the transfer of mass within solid systems. When the transfer involves a purely homogeneous phase, such as solid diffusion that occurs in metals at high temperatures, reasonable results may be obtained by using Equation 2. When the transfer involves a fluid phase passing through pores of a solid phase, such as encountered in liquid-solid extraction and drying of solids, the diffusion equations do not give reliable results because of the influence of other forces such as capillarity(57).

The Rate Equation. Whenever there is a concentration gradient of a component between phases, there is a potential available tending to transfer the component in the direction of decreasing concentration. The rate of transfer equals the driving force divided by the resistance, or the product of conductance and driving force(6,25):

$$\text{Rate} = (\text{Conductance})(\text{Drop in potential}) \qquad (3)$$

and

$$\text{Conductance} = (\text{Transfer coefficient})(\text{Area}). \qquad (4)$$

Under steady state conditions, the rate of transfer is the same in each phase and may be written$^{(25,126)}$

$$N = k_1 A(p_1 - p_i) = k_2 A(p_i - p_2) \qquad (5)$$

where:

N = mole transferred between phases per unit time, mol/hr

k_1 = transfer coefficient in phase 1, mol/hr-sq ft-unit potential

k_2 = transfer coefficient in phase 2, mol/hr-sq ft-unit potential

A = interfacial area, sq ft

p = potential

subletters 1, 2, and i refer to phase 1, phase 2, and interface, respectively.

Equation 5 assumes that equilibrium conditions exist at the interface between the two phases; therefore, potential of the transferring component, $\underline{p_i}$, is the same in both phases at the interface. This is equivalent to postulating that the interface itself offers no resistance to the transfer process. Verification of this assumption by direct experimental measurements is difficult and probably impossible with present equipment$^{(25)}$.

Mass Transfer Coefficient. The over-all mass transfer coefficient may be considered(124) as the reciprocal of the resistance to mass transfer from one phase to another. When two films exist at the interface and the resistances of both are appreciable, the over-all resistance is actually composed of two resistances in series, and is equal to the sum of the resistances of the individual films. In terms of the individual film transfer coefficients, the over-all coefficient has been shown(27,33,124) to be

$$1/K_1 = 1/k_1 + H/k_2$$

where:

K_1 = over-all coefficient based on phase 1, mol/hr-sq ft-mol/cu ft when liquid, mol/hr-sq ft-atm when gaseous

k_1 = individual coefficient of phase 1, mol/hr-sq ft-mol/cu ft when liquid, mol/hr-sq ft-atm when gaseous

k_2 = individual coefficient of phase 2, mol/hr-sq ft-mol/cu ft when liquid, mol/hr-sq ft-atm when gaseous

H = distribution coefficient, no units when both phases are liquid or both gaseous, atm/mol/cu ft when phase 1 gaseous and phase 2 liquid.

The constant, H, is necessary because of the units in which the individual coefficients k_1 and k_2 are expressed. The difference is in the method of expressing concentration in the two films. Actually, H represents the ratio between equilibrium concentrations in the two phases, and unless the law of linear distribution applies, H may not be a constant. The relationship between the two over-all coefficients, expressed in terms of concentration in the two phases, is

$$H K_1 = K_2 \tag{7}$$

where:

H = distribution coefficient, no units when both p phases are liquid or both gaseous, atm/mol/cu ft when phase 1 gaseous and phase 2 liquid

K_1 = over-all coefficient based on phase 1, mol/hr-sq ft-mol/cu ft when liquid, mol/hr-sq ft-atm when gaseous

K_2 = over-all coefficient based on phase 2, mol/hr-sq ft-mol/cu ft when liquid, mol/hr-sq ft-atm when gaseous.

Liquid-Solid Extraction

Extraction is a specific case of mass transfer that designates (22,28) any operation in which one or more soluble constituents of a liquid or solid, consisting of a mixture of soluble and insoluble constituents, is transferred to a liquid called the solvent. The term liquid-solid extraction is restricted to those operations in which a solid phase is present, and includes operations frequently referred to as leaching, lixivation, and washing. Further mention of the term "extraction" in this thesis refers to the more specific unit operation of liquid-solid extraction.

Systems of Operation. Any of the methods by which interphase mass transfer processes can be conducted may theoretically be used in execution of solvent extraction. Continuous countercurrent multistage contact with and without reflux are common in industry, while single contact and simple multistage contact are generally employed in laboratory-scale investigations. The four basic systems of operation are briefly discussed in this section.

Single Contact. The simplest method of extraction, and the type most commonly employed in laboratory-scale investigations, is demonstrated by a single contact of all the solvent and feed to form a liquid and a solid phase(29,39). Solute is transferred from solid to liquid phase, the amount of transfer being controlled by equilibrium of the system under the conditions employed. The quantity of extract is small, and is limited by the amount of solvent employed.

Simple Multiple Contact. Simple multiple contact(29,39) employs a number of single contact stages in series, with extract being removed from each stage. Raffinate from each stage is employed as feed for the next. Simple multiple contact systems are inefficient in their use of solvent, because fresh solvent must be added at each contact. However, this process may be used to accomplish any desired degree of extraction except complete separation of solute and carrier solid, if economy of solvent is ignored.

Countercurrent Multiple Contact. The countercurrent multiple contact process(29,39) is the most universally used of the extraction processes, because maximum concentration of solute is obtained with a given number of stages and a given solvent to feed ratio. Feed and solvent are fed into the system at opposite ends of the extraction unit, and the raffinate and extract flow countercurrently and continuously from stage to stage throughout the unit.

Countercurrent Extraction with Reflux. A finer degree of separation of feed components is often attained by employing reflux. Elgin and Wynkoop(39) have stated that the principle justifying employment of reflux in extraction "... is analogous to that in distillation, namely, that the outgoing extract layer is given an opportunity to be in equilibrium with a richer raffinate (i.e., the extract reduced in solvent content)." Use of reflux to achieve sharper separation of feed into extract and raffinate products requires employment of large quantities of solvent, and a proportionally larger heat consumption in effecting separation of the extract and solvent. The principal advantage of application of reflux to the extraction process is that it is possible to obtain the same degree of separation with moderately or poorly selective solvents, that can be obtained with highly selective solvents in a process without reflux.

Solvent and Feed Contact. Contact between solvent and feed may be provided by either, or a combination, of three principles(23): (1) dispersed contact, (2) moving solid bed, and (3) stationary solid bed. Mixtures of solid particles which form a bed of low porosity, either originally or during extraction, are usually treated in the dispersed state rather than in a solid bed. Stationary solid bed extraction involves a minimum amount of material handling and is often used where large quantities of solids are treated, or where the characteristics of the solid particles are such that continuous movement of solids is undesirable. In cases where the quantity of solids treated is small and the solids do not form a bed of low porosity, the moving solid bed principle is often employed.

Dispersed Contact. Where solids are fine enough to be kept in suspension by agitation, agitators and/or thickeners may be employed(23). Where both agitators and thickeners are employed, the agitators are used for contacting the solids with the liquid to dissolve the soluble material present in the solids, and the thickeners are used to separate solids from solution. In some cases the agitation necessary for adequate contact between solids and solvent is accomplished by countercurrent flow of feed and solvent through a pipe. For small scale batch operations, agitation and separation may be conducted in the same vessel.

Moving Solid Bed. Many commercial extraction units employ a solid bed moving countercurrently to the solvent(45,52,54,71,77,100). The Bollman or Hansa-Muhle soybean extractor(23,46) is an example of an extraction unit employing the moving solid bed principle. This extractor is similar to a bucket elevator, consisting of a series of perforated buckets suspended on a pair of circular chains. As the buckets move upward, solvent is sprayed on the bucket nearest the top, which contains bean flakes almost exhausted of oil. Extract flows through the bed, through the perforations in the bucket, and downward to the next bucket. A series of countercurrent multiple-stage contacts is thus achieved until the solution of solvent and oil reaches the bottom of the unit. The Kress extractor(63) has proved satisfactory as a moving solid bed extractor for pilot plant scale operation, but has not been employed commercially.

<u>Stationary Solid Bed.</u> Where a stationary bed of feed is employed(23), the most commonly used type of equipment consists of open tanks. If the solvent is corrosive, a protective lining is used. The open tanks are often provided with "false bottoms" which serve as supports for the bed of solids, but allow passage of extract. The solids are placed in the tank in such a way as to prevent size segregation of solid particles, and the solvent is then introduced at either the top or bottom of the tank. Equilibrium of the system employed, porosity of the solid bed, and rate of extraction desired are factors which determine whether the unit is operated purely as a batch operation or operated with solvent flowing continuously.

<u>Rate of Extraction.</u> The principles that determine the rate of extraction correspond closely to the principles that determine the rate of mass transfer in other diffusional processes(8). Consider a solid particle, containing an extractionable material, in contact with the extracting solvent. Let the concentration of solute in the extract at any chosen instant be represented by $\underline{c}$. At the same instant, the concentration of solute at the interface is

represented by $\underline{c'}$, which may or may not be equal to the concentration of solute in the solid itself, depending on the structure of the solid. In either case, the rate of diffusion from the interface into the extract is proportional to the concentration gradient, $\underline{c' - c}$. In many solids, such as peanuts, tanbark, sugarbeets, etc., the solute is contained in cells and the process involves the diffusion of this soluble material through the cell walls. The cell walls act as semipermeable membranes, and the process of diffusion involves the phenomenon of osmosis. The cells are finely divided through the mass of inert solid; therefore, the logical procedure would be to grind the solid to the fineness necessary to rupture individual cells. But, since material ground to pass a 200-mesh screen represents almost the limit of fine grinding, and material ground to this fineness will contain hundreds of cells, this suggestion is impracticable. Although individual cells are not broken by grinding, the number of cell membranes exposed to solvent is increased.

Diffusional processes are accelerated by performing the operation at as high a temperature as practicable. Also, any surface film resistance is decreased by employing high velocity of solvent relative to feed, with correspondingly great turbulence. Increasing velocity of solvent relative to feed necessitates increasing length of feed bed, in order to provide necessary time of contact. Feed bed length is normally increased by placing several extraction units in series. For example, the extraction of tanbark proceeds rapidly; therefore, velocity of solvent need not be great, and four to six extraction units are usually adequate. In extraction of beet sugar the diffusion processes are slow, the velocity of solvent is great, and from 8 to 14 extraction units are necessary to provide sufficient time of contact.

The System Peanut Meat-Peanut Oil-Hexane

Continuous solvent extraction methods have been adopted to an increasing extent as a means of obtaining oils from various vegetables(85,86,87). This section includes a review of extraction investigations, as well as a review of physical properties of the peanut meat-peanut oil-hexane system. Cottonseed data were reviewed when corresponding data for peanuts were not available.

Density-Composition Relation of Miscellae. The densities of various compositions of peanut oil-hexane and cottonseed oil-hexane miscellae have been determined by

Pollard, Vix, and Gastrock(90). Densities at 30 °C were determined by the pycnometer method and by the Westphal balance method, results agreeing to the fourth decimal place. Table I, page 20, shows experimental results of density determinations. Figure 1, page 21, shows these data as densities plotted against composition. Also, variations of density with temperature for different compositions of peanut oil-hexane miscellae(35) are plotted in Figure 2, page 22.

Viscosity-Composition Relation of Miscellae. Variations of viscosity with temperature for different compositions of peanut oil-hexane miscellae have been investigated by Decossas, Deckbar, and Hecker(36). Viscosity versus temperature curves for weight compositions of 0 to 100 per cent oil in hexane are shown in Figure 3, page 23.

Boiling Point-Composition Relation of Miscellae. Boiling point data for various compositions of vegetable oil-hexane miscellae, over a range of pressures from 160 to 760 millimeters, absolute, have been reported by Pollard, Vix, and Gastrock(90). The results were plotted as miscella composition versus miscella boiling point. Figure 4, page 24, shows experimental results for cottonseed oil-hexane and peanut oil-hexane miscellae, each at five absolute pressures.

TABLE I

Densities of Commercial Hexane-Crude Vegetable Oil Miscellae at 30 °C

Peanut Oil		Cottonseed Oil	
Oil Content, wt %	Density, gm/ml	Oil Content, wt %	Density, gm/ml
100.00	0.9116	100.00	0.9081
91.06	0.883	90.71	0.882
78.81	0.858	79.61	0.852
68.71	0.826	69.38	0.825
52.61	0.801	57.79	0.797
43.33	0.778	49.62	0.777
39.81	0.756	41.33	0.757
29.42	0.733	32.09	0.738
19.80	0.709	19.10	0.710
8.95	0.688	10.24	0.692
(Hexane) 0.00	0.6719	0.00	0.6719

Pollard, E. F., H. L. E. Vix, and E. A. Gastrock: Solvent Extraction of Cottonseed and Peanut Oils, Ind. Eng. Chem., <u>37</u>, 1024 (1945).

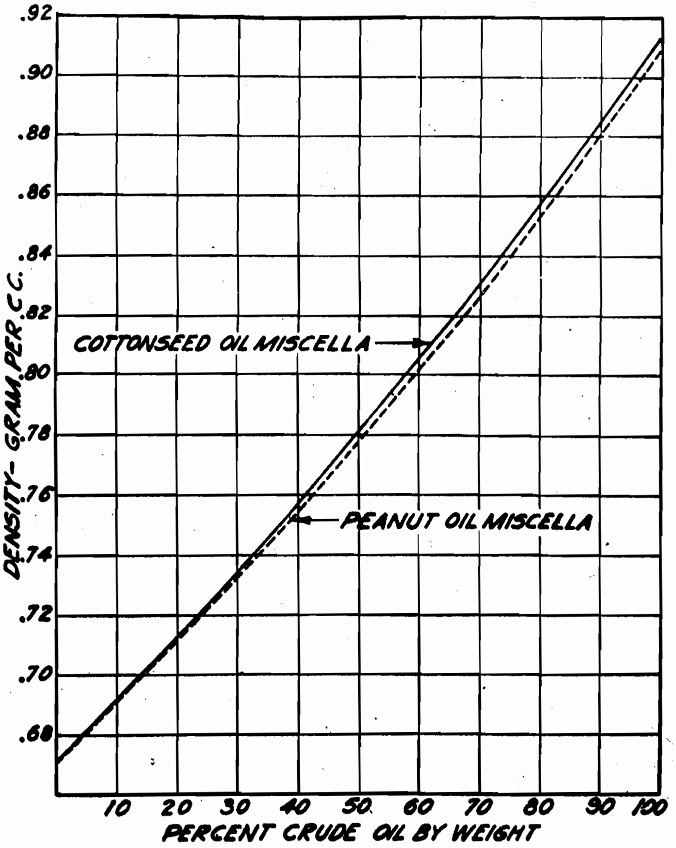


Figure 1. Density-Composition Relations for Miscellae of Crude Cottonseed and Peanut Oils in Commercial Hexane at 30 °C.

Pollard, E. F., H. L. E. Vix, and E. A. Gastrock: Solvent Extraction of Cottonseed and Peanut Oils, Ind. Eng. Chem., 37, 1023 (1945).

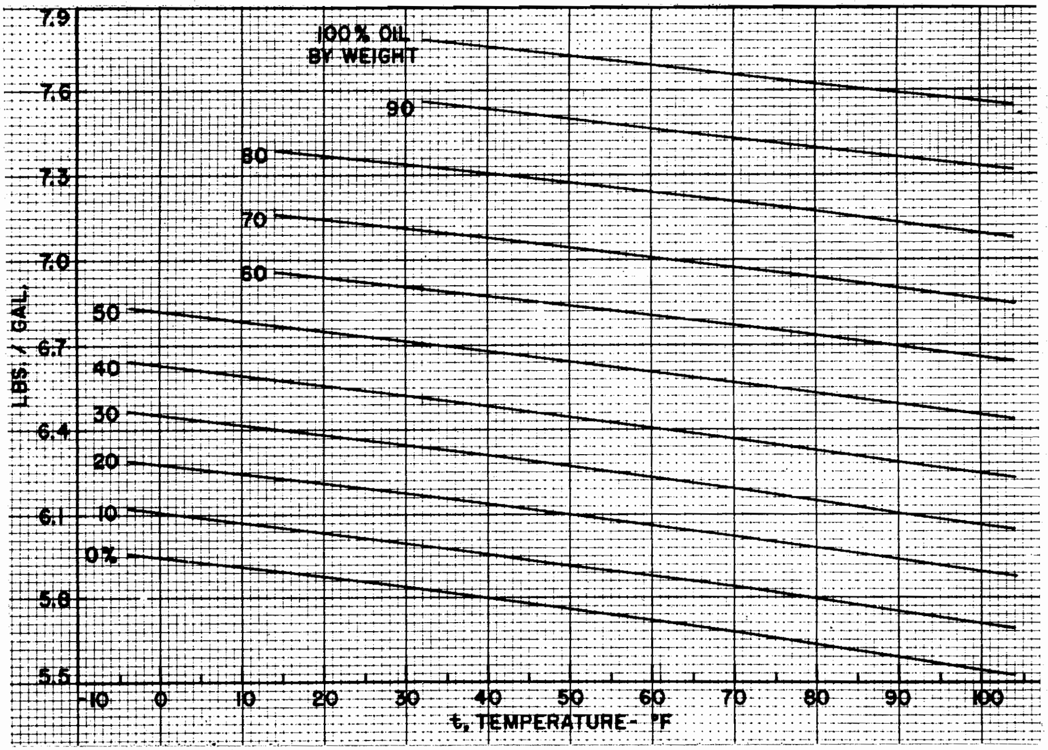


Figure 2. Effect of Temperature and Concentration on Peanut Oil-Hexane Miscellas Densities.

Decossas, K. M., F. A. Deckbar, Jr., and J. L. Hecker: Densities and Gravities of Cottonseed and Peanut Oil Miscellas in English Units, South. Reg. Research Lab., New Orleans, La., Bull. AIC-304, 4 (1950).

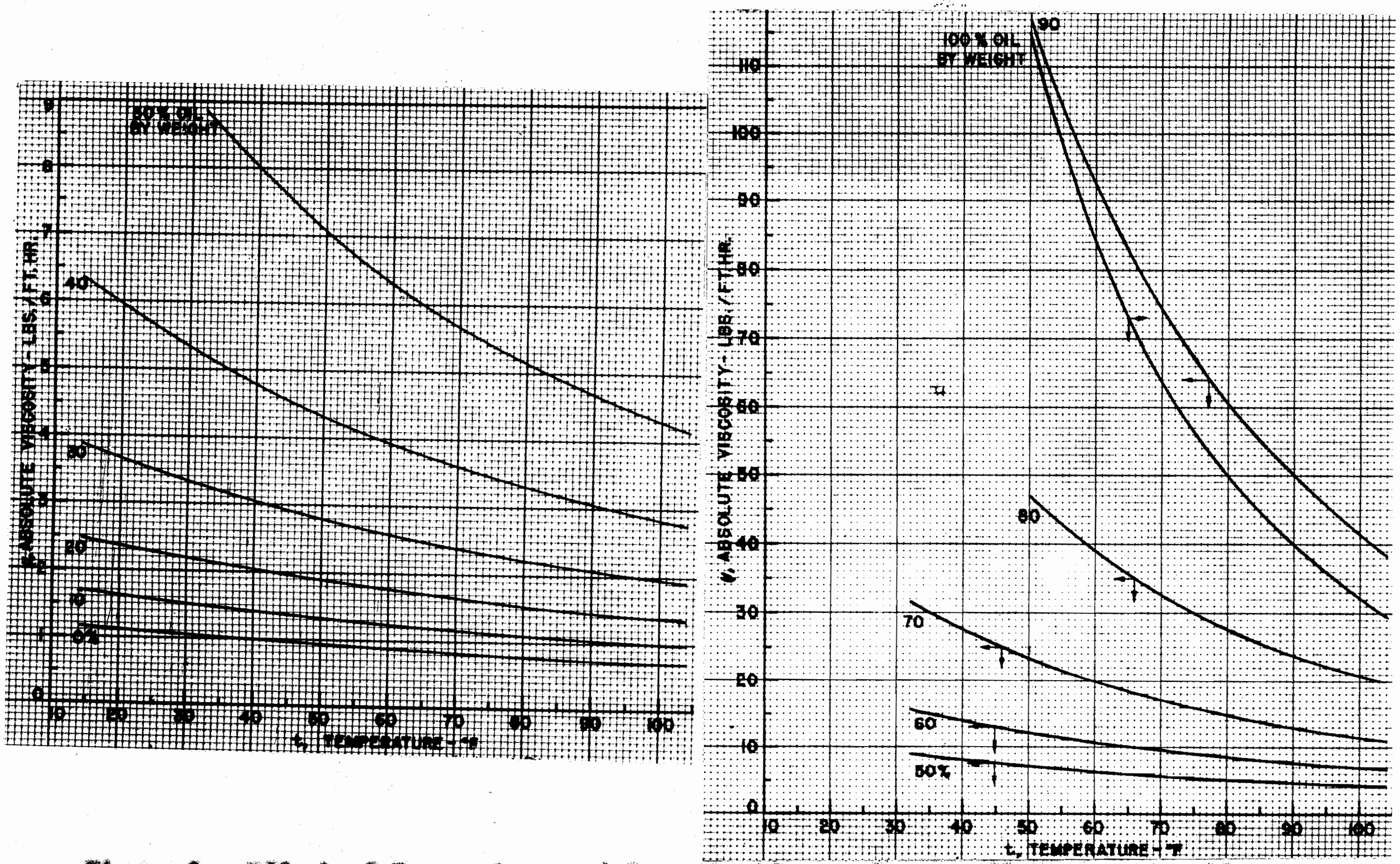


Figure 3. Effect of Temperature and Concentration on Peanut Oil-Hexane Miscellae Viscosities.

Decossas, K. M., F. A. Deckbar, Jr., and J. L. Hecker. Viscosities of Cottonseed and Peanut Oil-Hexane Miscellas in English Units, South. Reg. Research Lab., New Orleans, La., Bull. AIC-292, 4 (1950).

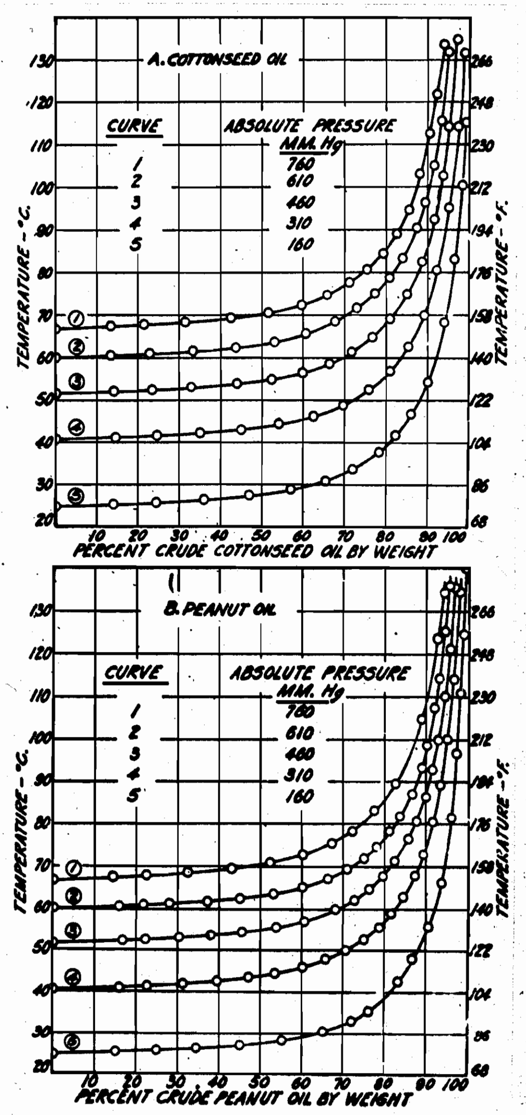


Figure 4. Boiling Point-Composition Curves for Hexane-Vegetable Oil Miscellas at Constant Vapor Pressures.

Pollard, E. F., H. L. E. Vix, and E. A. Gastrock: Solvent Extraction of Cottonseed and Peanut Oils, Inc. Eng. Chem., 37, 1022-6 (1945).

Boiling point curves for cottonseed and peanut oil miscellae at the same absolute pressure are practically congruent, indicating similar physical properties for the two miscellae.

Comparison of Solvents. A comparison of five solvents used commercially for extraction of cottonseed oil has been reported by Eaves, Molaison, Black, Crovetto, and D'Aquin(37). Comparable pilot plant scale extractions were conducted employing each solvent. The solvents hexane, benzene, ethyl ether, acetone, and butanone were compared as to their effect on oil yield and oil quality. None of the solvents investigated compared favorably with hexane for extraction of oil from cottonseed. Ethyl ether, acetone, and butanone were slightly more selective for cottonseed oil than was hexane, as shown in Table II, page 26. However, this increased extraction was achieved at the expense of cottonseed oil quality.

Solvent Extraction Efficiencies. Solvent extraction of four vegetable oils has been investigated by Pominski, Molaison, Crovetto, Westbrook, D'Aquin, and Guilbeau(91). Oil was extracted from cottonseeds, peanuts, okraseeds, and rice bran, employing commercial hexane as the solvent. Quantity of solvent employed for each extraction test was

TABLE II

Efficiency of Five Commercial Solvents for Extracting Oil from Cottonseed

Solvent	Oil in Feed[a], wt %	Crude Oil Yield[b], wt %	Oil in Raffinate[c], wt %	Extracted Oil[d], wt %
Hexane	35.2	35.75	0.99	99.01
Benzene	35.3	36.56	1.49	98.51
Ethyl Ether	35.2	36.39	0.33	99.67
Acetone	35.0	36.77	0.73	99.27
Butanone	35.1	37.44	0.26	99.74

[a] Pounds of solvent-free cottonseed oil yield divided by pounds of feed.

[b] Pounds of solvent-free cottonseed oil yield divided by pounds of moisture-free feed.

[c] Pounds of solvent-free cottonseed oil in raffinate divided by pounds of solvent-free cottonseed oil in feed.

[d] Pounds of solvent-free cottonseed oil in miscella divided by pounds of solvent-free cottonseed oil in feed.

Eaves, P. H., L. J. Molaison, C. L. Black, A. J. Crovetto, and E. L. D'Aquin: A Comparison of Five Commercial Solvents for Extraction of Cottonseed, J. Am. Oil Chemists' Soc., XXIX, 88-94 (1952).

more than sufficient for oil removal, and a high extraction efficiency was obtained in every test. Solvent extraction data and results are given in Table III, page 28.

Ultrasonics

This section is devoted to collection and organization of literature pertinent to the effect of the unique form of energy, ultrasonics, on mass transfer. Generation of ultrasound is briefly discussed, but primary attention has been focused on absorption of sound in various media and on previous investigations of the influence of sonic energy on mass transfer.

Generation of Ultrasonic Energy. Two types of ultrasonic generators, from the theoretical standpoint, are presently being used(113): (1) the vibrating disc or piston and (2) the interrupted gas stream. From the viewpoint of method by which vibrations are produced, the five essential types of generators for producing sonic and ultrasonic energy are(113): (a) mechanical, (b) thermal, (c) electromagnetic, (d) magnetostriction, and (e) piezoelectric generators. The piezoelectric generator has been employed at highest frequencies, and

TABLE III

Batch Solvent Extraction Data

Material	Batch Weight, lb	Flake Thickness, in	Solvent Employed	Extraction Moisture[h], %	Time of Extraction, hr	min	Total Solvent Through Extractor, gal	Solvent per Pound Feed, gal	Temperature of Solvent to Extractor, °F	Extraction Efficiency[i], %
Peanuts[a]	125	0.010	Hexane	9.4	55	-	360	2.88	83	99.0
Cottonseed[b]	120	0.010-0.013	Hexane	7.6	4	38	151	1.26	54	98.1
Cottonseed[c]	118	0.010	Ethyl ether	7.6	12	42	445	3.77	60	99.8
Cottonseed[d]	125	0.007-0.010	Hexane	8.2	6	2	219	1.74	85	98.7
Cottonseed[e]	76	-	Ethyl ether	11.7	8	30	334	4.37	78	-
Okra seed[f]	133	0.009	Hexane	11.5	5	40	222	1.66	66	95.2
Rice bran[g]	120	-	Hexane	11.4	9	55	245	2.04	54	96.2

[a] Peanuts were prepared in standard small-scale equipment, cleaned, stoned, hulled, and flaked.
[b] Cottonseed was prepared in standard small-scale equipment, cleaned, delinted, hulled, and flaked.
[c] Cottonseed was prepared as in b.
[d] Cottonseed was prepared as in b. Average data of six extractions are given.
[e] Defatted cottonseed was re-extracted and the data represent an average of four extractions.
[f] Okra seed was prepared by passing through cracking and flaking rolls. There was no separation of hulls.
[g] Rice bran was used as received from the rice mill. Three extractors were run in series. The data are the average of the three batches processed.
[h] Moisture content of feed prior to extraction.
[i] Pounds of solvent-free oil extracted divided by pounds of oil in pre-extracted feed.

Pominski, J., L. J. Molaison, A. J. Crovetto, R. D. Westbrook, E. L. D'Aquin, and W. F. Guilbeau: Extraction of Cottonseed and Peanut Oils, Oil Mill Gaz., 51, 33-9 (1942).

has been used in a variety of ways in liquid and solid systems. Available transducer types for use at high power levels for various regions of specific acoustic impedance versus frequency are illustrated in Figure 5, page 30, and a spectrum of sonic energy which can be produced by piezoelectric generators is shown in Figure 6, page 31. Resonant frequency variations with temperature for six piezoelectric materials are shown in Figure 7, page 32.

Absorption of Sonic Energy. Whenever a medium is traversed by compressional waves, amplitude of the waves is decreased due to a number of causes. Some of these factors causing decrease in amplitude of vibration are well known while others are not thoroughly understood, but all combine to produce absorption of sonic energy. Viscosity, scattering, and intramolecular processes all play an important part in governing absorption of sound in various media.

Absorption of Sound in Water. Absorption of sonic energy in water over a temperature range of 0 to 95 °C and a frequency range of 7 to 70 megacycles has been measured by Pinkerton(88). Absorption of sonic energy over this temperature range was inversely proportional to square of frequency, as shown in Figure 8, page 33.

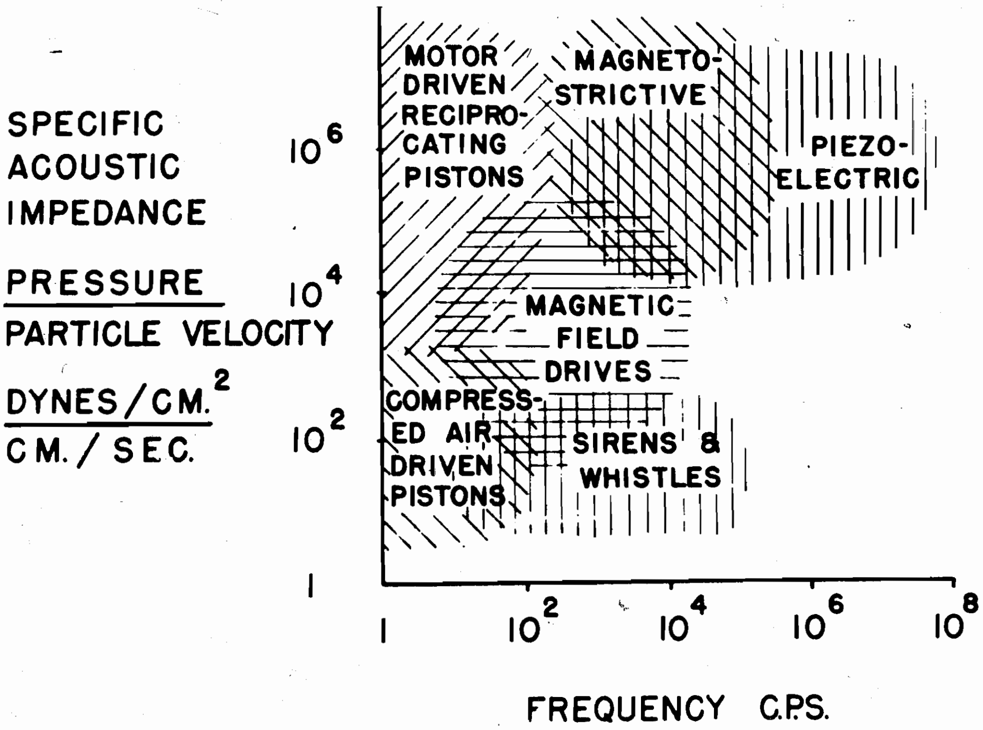


Figure 5. Available Transducer Types Usable at High Power Levels for Various Regions of Specific Acoustic Impedance Versus Frequency.

Samsel, R. W.: Physical and Economic Limitations in the Application of Sonic and Ultrasonic Energy to Industrial Processing, "Ultrasonics--two symposia." Chemical Engineering Progress Symposium Series, No 1, pp. 77-81, 47, American Institute of Chemical Engineers, New York, N. Y., 1951.

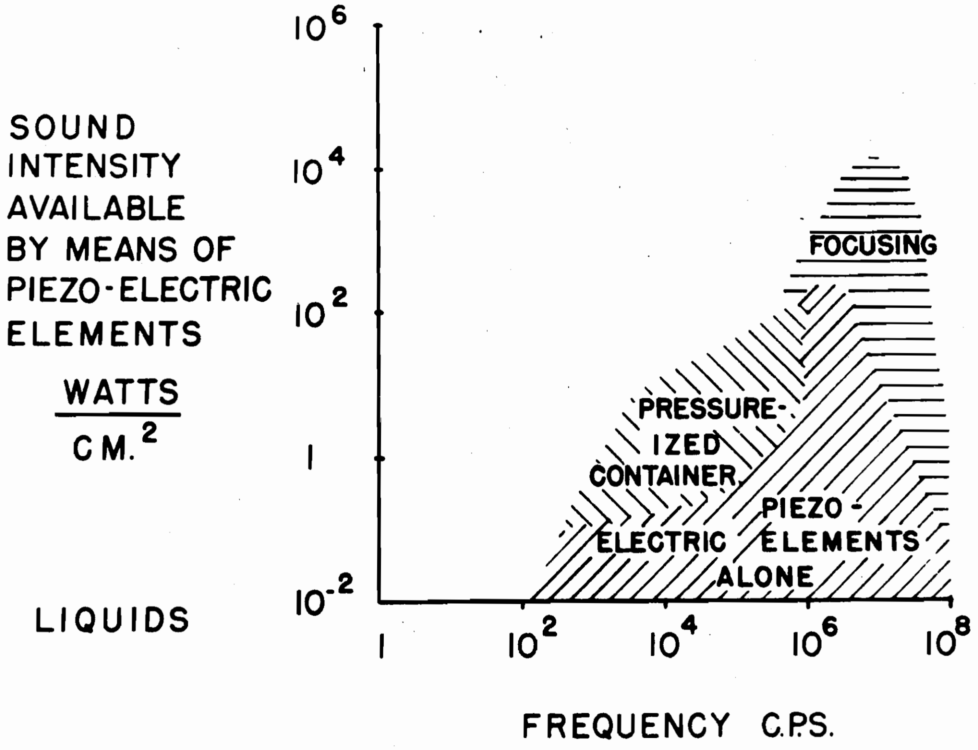


Figure 6. Spectrum of Sounds Produced by Piezoelectric Generators.

Samsel, R. W.: Physical and Economic Limitations in the Application of Sonic and Ultrasonic Energy to Industrial Processing, "Ultrasonics--two symposia." Chemical Engineering Progress Symposium Series, No 1, pp. 77-81, 47, American Institute of Chemical Engineers, New York, N. Y., 1951.

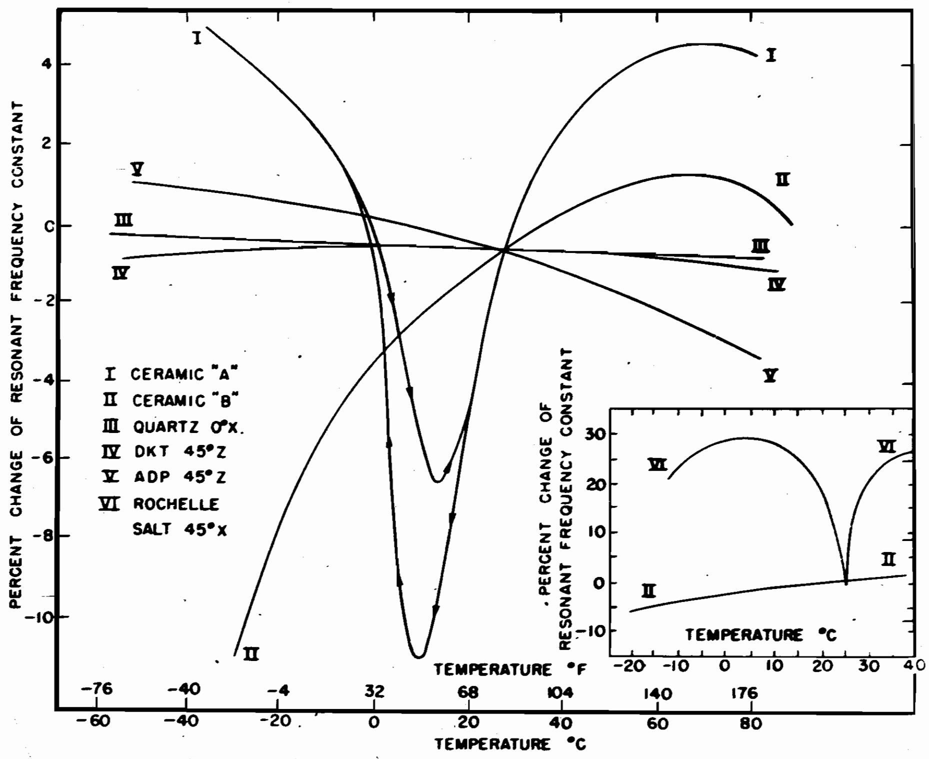


Figure 7. Percentage Change of Frequency Constants with Temperature for Long Slim Free Bar.

Piezotronic Technical Data, Brush Electronics Co., Cleveland, Ohio, Bull., CD43001 6M 2-53, 12-3 (1953).

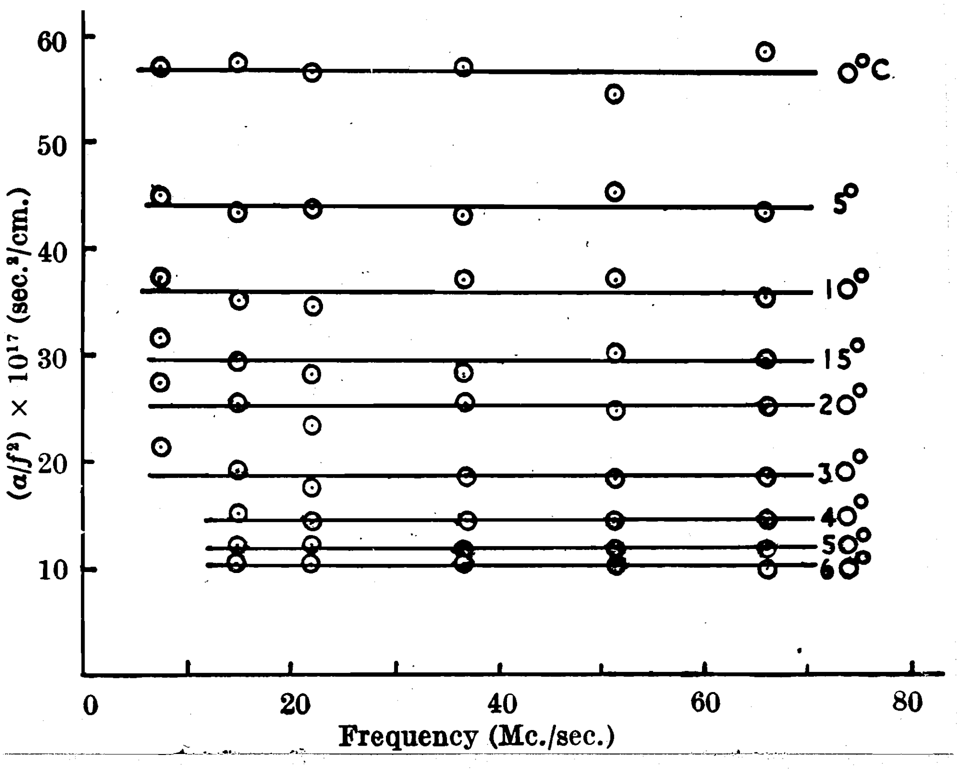


Figure 8. Absorption of Ultrasound in Water as a Function of Temperature and of Frequency.

Pinkerton, J. M. M.: A Pulse Method for the Measurement of Ultrasonic Absorption in Liquids: Results for Water, Nature, <u>160</u>, 128-9 (1947).

The ratio of observed to calculated absorption(89), shown in Table IV, page 35, was approximately constant between 0 and 95 °C although absorption itself varied considerably with temperature. The slight rise in ratio of observed to calculated absorption over the range 0 to 10 °C was accredited to known structural changes of water at this temperature. Other investigators(15,43,93) have obtained data in good agreement with the data of Figure 8 and Table IV.

Absorption of Sound in Pure Liquids. Contradictory values of absorption coefficients of sound for the same liquids have been reported(51,68,69,89). However, investigators are in agreement that experimental values of absorption divided by the square of frequency employed, a/f^2, far exceed theoretical values. Particularly high a/f^2 values have been reported for carbon disulphide, carbon tetrachloride, and benzene.

Values of a/f^2 are not frequency sensitive for most liquids. Only in acetic acid has a decreasing a/f^2 value with frequency been confirmed(68,69,89). Values of a/f^2 at various frequencies for methyl alcohol and acetic acid are compared in Figure 9, page 36.

TABLE IV

Absorption of Ultrasound in Water

Temperature, °C	$10^{14} \cdot a/f^2$ [a] Observed, db sec²/m	$10^{14} \cdot a/f^2$ [a] Calculated, db sec²/m	Rate of Observed to Calculated Absorption	Probable Error, %
0	49.4	14.83	3.33	0.6
5	38.3	11.63	3.29	0.6
10	31.1	9.85	3.15	0.86
15	25.9	8.33	3.11	0.9
20	22.0	7.09	3.10	1.35
30	16.6	5.34	3.11	1.7
40	12.7	4.21	3.01	0.5
50	10.4	3.46	3.01	0.4
60	8.83	2.92	3.02	0.8
70	7.57	2.53	2.99	-
80	6.85	2.24	3.06	-
90	6.29	2.01	3.13	-
100 (b)	5.97	1.83	3.26	-

[a] a = absorption coefficient of ultrasound in water.
f = frequency of insonation.

[b] Extrapolated from 95 °C.

Pinkerton, J. M. M.: The Absorption of Ultrasonic Waves in Liquids and its Relation to Molecular Constitution, Proc. Phys. Soc., <u>62</u>, 129-41 (1949).

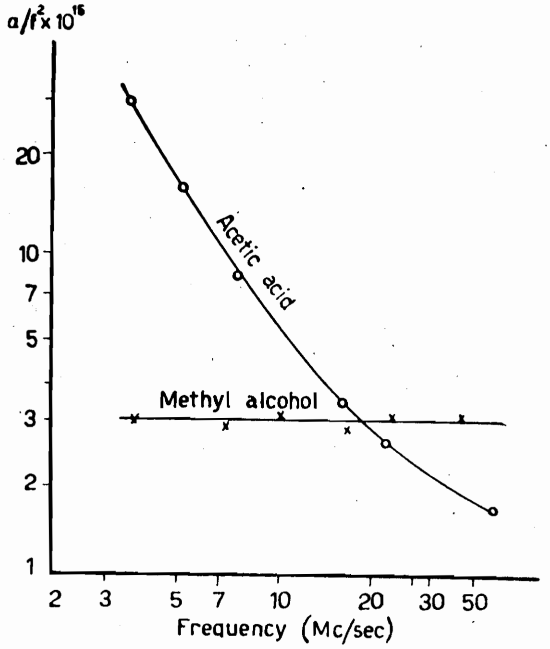


Figure 9. Absorption of Ultrasound as a Function of Frequency in Methyl Alcohol and Acetic Acid.

Pinkerton, J. M. M.: The Absorption of Ultrasonic Waves in Liquids and its Relation to Molecular Constitution, Proc. Phys. Soc., 62, 129-41 (1949).

Comparison of ultrasonic absorption in pairs of organic liquids having symmetry of molecule, such as benzene and toluene, showed no correlation of absorption coefficient(51). The addition of a methyl group caused an increase in ultrasonic absorption, but subsequent additions or rearrangements had little effect.

Absorption of Sound in Liquid Mixtures. Absorption of sonic energy in liquid mixtures has been investigated(31,95,99,122) employing several different systems. The attenuation versus concentration curve for mixtures of two liquids having different densities and viscosities continually increased or decreased from one concentration extreme to the other, providing viscosity of the mixture behaved in the same manner. Variation in absorption of ultrasound with concentration in a mixture of acetone and benzene is shown in Figure 10, page 38. In some mixtures, however, viscosity reached a maximum at some intermediate concentration. Experimental and theoretical data(31) plotted in Figure 11, page 39, illustrate absorption of sonic energy in a mixture of acetone and water. The slight hump in the theoretical absorption curve was due to

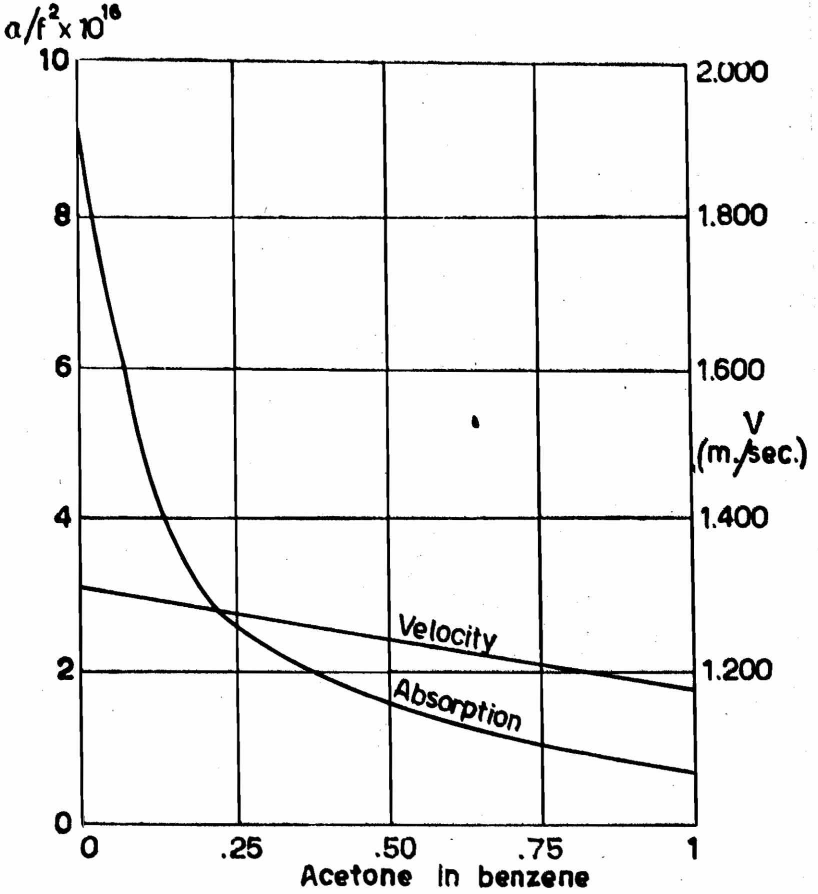


Figure 10. Absorption and Velocity of Ultrasound in Acetone-Benzene Mixtures.

Willard, G. W.: Ultrasonic Absorption and Velocity Measurement in Liquids, J. Acoust. Soc. Am., 12, 438-48 (1941).

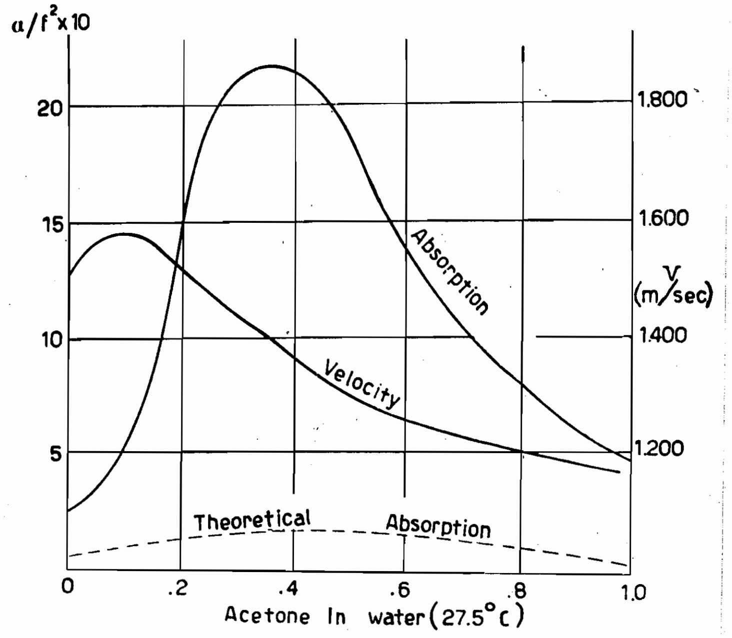


Figure 11. Absorption and Velocity of Ultrasound in Acetone-Water Mixtures.

Burton, C. J.: Ultrasonic Velocity and Absorption in Liquid Mixtures, J. Acoust. Soc. Am., <u>20</u>, 186-99 (1948).

maximum viscosity for the mixture near the 50 per cent concentration mark. Large experimental absorption values are presently not explained by theory.

Aqueous electrolyte solutions showed an almost linear change of a^2 with concentration, although there was a tendency for a/f^2 to decrease at high frequencies(95). The slope of the a/f^2 versus concentration curve depended on the two ions involved. Experimental values of ultrasonic absorption for several molar solutions, relative to water, are listed in Table V, page 41.

Absorption of Sound in Suspensions. Attenuation of an ultrasonic wave traversing a suspension is greatly influenced by scattering produced by suspended particles, particularly when size of suspended particles approaches wave-length. A mathematical expression for absorption of sound waves in suspensions of solid spheres in liquids has been developed by Sewell(105). This equation is valid only under the conditions that particle diameter is "... small compared to wave-length, factor $w/v \cdot d/2$

TABLE V

Absorption of Sound in Molar Solutions Relative to Water

Solution	Absorption Relative to Water
H_2O	1.00
Na_2SO_4	2.54
$CuSO_4$	5.28
$MgSO_4$	4.40
$NaClO_4$	1.37
$NaClO_3$	1.05
CH_3COONa	1.12
$Cu(NO_3)_2$	1.11
$MgCl_2$	1.09
$CuCl_2$	1.93
KBr	0.88
$NaBr$	0.85
$NaCl$	0.89
Na_3PO_4	6.15
$K_4Fe(CN)_6$	2.95
$K_2Cr_2O_7$	2.20
$K_2Sn(OH)_6$	2.30
$NO_2(NO_3)_2$	3.02

Rufer, W.: Absorption Measurements on Ultrasonic Waves in Electrically Conducting Solutions, Ann. Physik, <u>41</u>, 301-12 (1942).

is large, and volume occupied by spheres is small compared to that of the container." Sewell's equation is:

$$a = N\pi d^2/4 \left(12\ v/cd + 3 \quad 2wv/c + 7/144 \cdot w^4 d^4/c^4\right)$$

$$= a_1 + a_2 + a_3$$

where:

a = absorption coefficient, lb/sq ft

N = particle distribution, particles/cu ft

d = particle diameter, ft

v = dynamic viscosity, lb ft/sec

c = sound velocity, ft/sec

w = pulsatance, 1/sec.

The first two bracketted terms of the equation, $\underline{a_1}$ and $\underline{a_2}$, represent incident energy lost by friction. The last term, $\underline{a_3}$, represents energy lost by scattering. The most influential term is the first, and absorption can be approximated by the term $\underline{3N\pi dv/c}$.

Stokes' expression has been adapted to suspensions of flexible spheres(67) such as mists and emulsions. Distortions in flexible suspended matter cause only a slight variation is absorptivity, except in the case of gas bubbles in liquids at some resonant value of frequency.

Investigation of the conditions under which mobility of suspended particles occurs(3,21,49,65) has shown absorption to increase as frequency increases in suspensions containing only small particles. Absorption coefficients in a fungi spore suspension(65) were 3.71, 4.07, and 5.77 theoretically and 2.9, 3.1, and 3.9 experimentally, employing insonation frequencies of 42, 98, and 695, kilocycles, respectively. Further confirmation of the slight increase of ultrasonic absorption with frequency in suspensions containing only small particles has been reported(3), employing a medium of tobacco smoke. The variation of absorption with frequency for suspensions of 50 micron diameter lycopodium(49) is shown in Figure 12, page 44. Experimental values of absorption coefficients much greater than predicted by Sewell's theory were accredited to multiple scattering.

Effect of Pressure on Sound Absorption. Absorption of ultrasonic energy in benzene, toluene, carbon tetrachloride, methyl acetate, and water has been

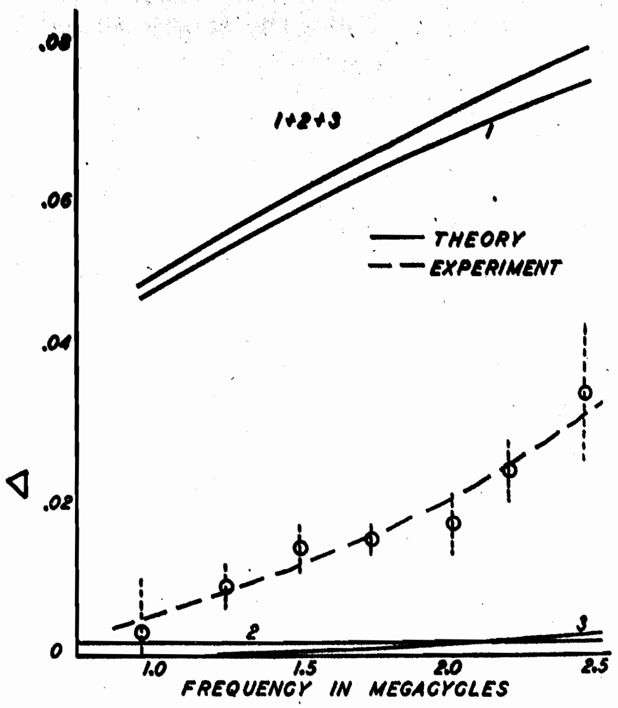


Figure 12. Theoretical and Experimental Absorption Coefficients (Δ) for Suspensions of 50-Micron Diameter Lycopodium in Water (See page 42). Theoretical Curves 1, 2, and 3 (x 100).

Hartmann, G. K. and A. B. Focke: Absorption of Supersonic Waves in Water and in Aqueous Suspensions, Phys. Rev., 57, 221-5 (1940).

investigated(16) employing pressures varying from 0 to 630 kilograms per square centimeter. Figure 13, page 46, shows that $\underline{a/f^2}$ decreased near linear with increasing pressure, in accordance with known viscosity changes with pressure. Values of $\underline{a/f^2}$ decreased near linear only until the liquid neared its point of solidification. Also, experimental and theoretical values of absorption are reported to be in agreement at pressures of several hundred or thousand atmospheres, depending on temperature(55).

Effect of Temperature on Sound Absorption. Interesting changes in absorption occur when temperature of a liquid is varied, most liquids falling into one of the following four classifications:

1. Non-polar liquids of small molecules. Absorption values much greater than theoretical and a positive temperature coefficient are characteristic.

2. Liquids with hydroxyl groups. Absorption values slightly greater than theoretical and a positive temperature coefficient are characteristic.

3. Monatomic liquids characterized by theoretical absorption.

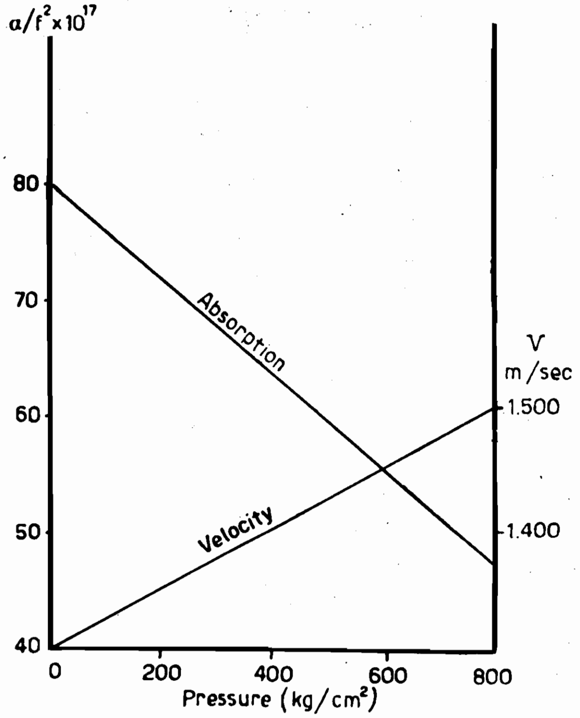


Figure 13. Absorption and Velocity of Ultrasound in Liquids as a Function of Pressure (1 Kilogram Per Square Centimeter Equals 14.22 Pounds Per Square Inch).

Biquard, P.: Etude sur la Vitesse de Propogation et sur L'absorption du son dans les Liquides sons Pression, Rev. d'Acoust., 8, 130 (1939).

4. Viscous liquids. Absorption values slightly greater than theoretical and a negative temperature coefficient are characteristic.

Benzene and methyl iodide, class 1 liquids, have been found to exhibit increases in absorptivity of sonic energy with increasing temperature (Figure 14, page 48). Benzene was characterized by a linear increase in absorption coefficient(92), while values of absorption coefficient for methyl iodide approached a maximum at the boiling point(84). Theoretically, absorption for all four classifications of liquids decreases with increasing temperature, as kinematic viscosity decreases. Experimentally, only class 3 liquids, class 4 liquids, and water exhibited the theoretical positive temperature coefficient.

Application of Sonic Energy to Liquid-Solid Extraction. Various methods of increasing solvent extraction rates have been investigated(8,23,46,100). Investigations pertinent to the effect of insonation on liquid-solid extraction are reviewed in this section.

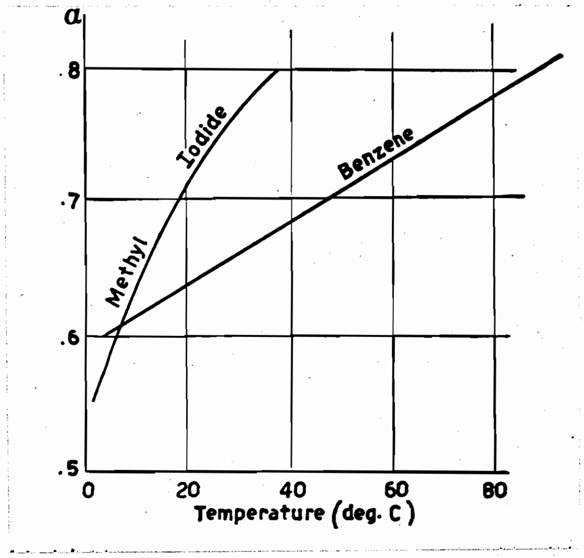


Figure 14. Absorption Coefficients as a Function of Temperature for Methyl Iodide[a] and Benzene[b].

[a] Pellam, J. R. and J. K. Galt: Application of Pulse Technique to Velocity and Absorption Measurements at 15 Megacycles, J. Chem. Phys., <u>14</u>, 608-14 (1946).

[b] Quinn, J.: The Absorption of Ultrasonic Waves in Benzene, J. Acoust. Soc. Am., <u>18</u>, 185-9 (1946).

Solvent Extraction of Protein from Brewer's Yeast Employing Audible Insonation. Solvent extraction of protein from brewer's yeast has been investigated by Grove(48). A five per cent solution of sodium chloride was employed as the solvent, and the system was subjected to the audible insonation of a magnetostriction generator. Data in Table VI, page 50, show that insonation produced slightly higher extraction values than were obtained in the non-insonated control tests. Increased extraction was accredited to the dissolved gases in the water diffusing through the yeast cell membranes, making possible "explosion" of the yeast cells during insonation.

Extraction of Oil from Fish Materials Employing Insonation. Audible and inaudible insonation have been applied to the extraction of oil from fish materials(107). Frequency of insonation was not critical when high power level was maintained, but most of the investigation was conducted employing frequencies low in the audible range. Fish fragments were coarsely ground to produce a pulpy mass, and subjected to compressional wave vibrations at high

TABLE VI

Solubility of Protein from Brewer's Yeast in a Five Per Cent Solution of Sodium Chloride

Elapsed Time of Exposure, min	Weight of Yeast in 500 ml Dispersion, gm	Weight Nitrogen in Filtrate	
		Vibrated, mg	Non-vibrated, mg
20	10.0	4.84	3.78
20	10.0	4.82	3.78
20	12.5	7.00	6.30
20	12.5	7.00	6.30
20	15.0	8.68	7.98
20	15.0	8.75	7.98
20	17.5	9.10	9.03
20	17.5	9.53	9.03
20	20.0	9.52	9.10
20	20.0	9.56	9.10
20	25.0	10.71	10.49
20	25.0	10.98	10.49
20	30.0	13.93	13.37
20	40.0	21.35	20.23

Grove, H. D.: A New Magneto-Striction Oscillator - Construction and Observation of some Chemical and Physical Phenomena, p. 57. Unpublished M. Sc. Thesis, Library, State Univ. of Iowa, Iowa City, Iowa (1948).

power levels. Intensity of insonation was sufficient to produce cavitation within the pulpy mass, causing rupture of cellular fish material. Fish oils were released from the oil cells, and could be removed mechanically without addition of foreign substances.

Extraction of oil from fish material and protein from brewer's yeast have been accelerated by employment of insonation. Conclusions of these investigations are:

1. Audible insonation increased the rate of extraction of protein from brewer's yeast(48).

2. Audible and inaudible insonation increased the rate of extraction of oil from fish material(107).

3. Oil cells of fish materials were ruptured during both audible and inaudible insonation, releasing fish oil from the insonated solid mass(107).

III. EXPERIMENTAL

Solvent extraction of oil from peanuts was investigated employing ultrasonic insonation. The purpose of investigation, plan of experimentation, lists of materials and apparatus, method of procedure, data and results, and sample calculations are presented under their respective headings.

Purpose of Investigation

The purpose of this investigation was to determine the effects of intensity and time of 400-kilocycle insonation on solvent extraction of oil from peanuts.

Plan of Experimentation

In determining the effects of ultrasonic insonation on solvent extraction of oil from peanuts, primary attention was focused on the determination of the per cent of oil extracted employing ultrasonic insonation. Comparison was made with extraction employing mechanical agitation and a control under similar thermal conditions.

Specifically, the plan of experimentation required: reviewing the literature, devising methods of procedure, selecting equipment to use, determining method of analysis, determining oil content of peanuts, preparing peanuts for extraction, conducting preliminary investigations, conducting experimental peanut oil extraction tests employing ultrasonic insonation and mechanical agitation, and conducting control and reproducibility tests.

Materials

Description, source, and use of all materials employed in this investigation are listed below. Details relative to identification that will aid in duplication of materials have been included.

Air, Compressed. Non-purified. Obtained from Sullivan Machinery Co. air compressor in McBryde Building through black iron pipes at maximum pressure of 90 pounds per square inch, gage. Used as source of energy to turn impeller in the air motor.

Carbon Tetrachloride. CP anhydrous, specific gravity at 25/25 °C 1.589, boiling range 77.0 to 77.5 °C, non-volatile matter 0.0000 per cent, iodine consuming substances: none, acidity: none, free chlorine: none. Obtained from Fisher Scientific Co., Silver Spring, Md. Used as the liquid in the bulb of the glass thermostat in the constant temperature bath.

Cement, Varno. Catalog No V-22. Obtained from the Varniton Co., Los Angeles, Calif. Used as a cement to seal thermocouple wires into the ends of glass tubing.

Chloroplatinic Acid. Technical, code No P-154. Distributed by Fisher Scientific Co., Silver Spring, Md. Used as a source of platinum for fusing same into pyrex glass tube when bonding tinned nickel disc to platinized pyrex glass.

Electricity. 120 volts, 60 cycles, alternating current. Obtained from wall receptacle in room A102 in McBryde Building. Used as source of energy for operation of all electrical equipment except air conditioner.

Electricity. 220 volts, 60 cycles, alternating current. Obtained from wall receptacle in room A102 in McBryde Building. Used as source of energy for operation of Carrier air conditioner, model 51H2.

Flux. Liquid, "Tri-Flux", No 3 variety. Manufactured by Wolfe-Kote Co., Sheboygan, Wis.; distributed by Whitehead Metal Products Co., Inc., New York, N. Y. Used as a flux for bonding tinned nickel disc to platinized pyrex glass.

Friction Tape. Width 3/4-inch, code No 8. Manufactured by Appleton Rubber Co., Inc., Boston, Mass.; distributed by Brown Stores Co., Inc., Blacksburg, Va. Used for general purpose electrical insulation.

Hexane. Normal (from petroleum), technical grade, water white, flammable, volatile, shipping: red label, No P 1135, five kilgrams. Obtained from Eastman Organic Chemicals, Rochester, N. Y. Used as solvent for investigations of peanut oil extraction.

Mercury. Obtained from the Department of Chemical Engineering, Virginia Polytechnic Institute, Blacksburg, Va. Purified by investigator. Used as the contact liquid in the glass thermostat in the constant temperature bath.

Peanuts. Unshelled, crude oil content 55 per cent, specific gravity of oil at 30 °C 0.8979, moisture loss after oven drying at 110 °C for 14 hours 2.21 per cent. Obtained from Tidewater Field Station, Holland, Va. Used as feed for all extraction investigations.

Solder. Non-flux, 50-50 per cent lead-tin. Distributed by Noland Co., Roanoke, Va. Used for soldering nickel discs to platinized pyrex glass.

Apparatus

Apparatus used during this investigation is discussed in this section with a more detailed discussion presented for the more complex equipment.

Ultrasonic Generator. An ultrasonic generator is an instrument which converts high frequency electrical power into high frequency mechanical vibrations. The ultrasonic generator employed in this investigation, Crystalab Ultra-Sonorator, model number SL520, consisted of a generator of radio frequency energy and four electromechanical transducers. The signal generator was capable of delivering power between 0 to 720 watts of radio frequency energy. Acoustical power delivered from the transducer was approximately proportional to the product of plate current and plate voltage. The electromechanical transducers, quartz crystals, converted the electrical vibrations generated in the radio frequency generator into mechanical vibrations at frequencies of 400, 700, 1000, or 1500 kilocycles per second.

The unit consisted of the following: The radio frequency generator (Figures 15 through 18, pages 57 through 60, respectively), a coaxial connector cable, an impedance matching unit,

Figure 15. Front View of Ultra-Sonorator High Frequency Generator Showing Plate Voltmeter (left), Plate Ammeter (right), Switches, Powerstat, and Timer (lower center).

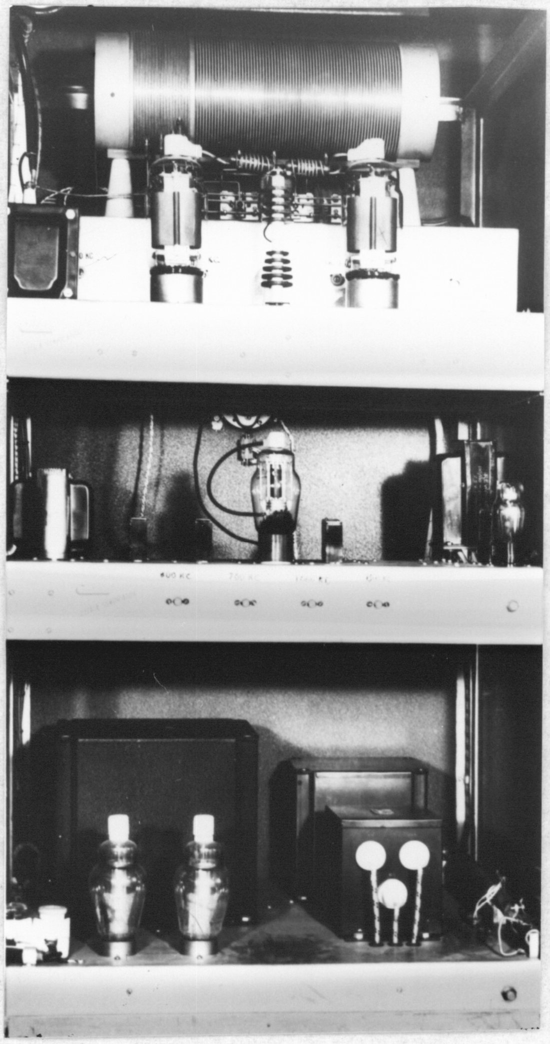


Figure 16. Rear View of Ultra-Sonorator High Frequency Generator Showing Rectification Chassis (bottom), Oscillation Chassis (center), and Amplification Chassis (top).

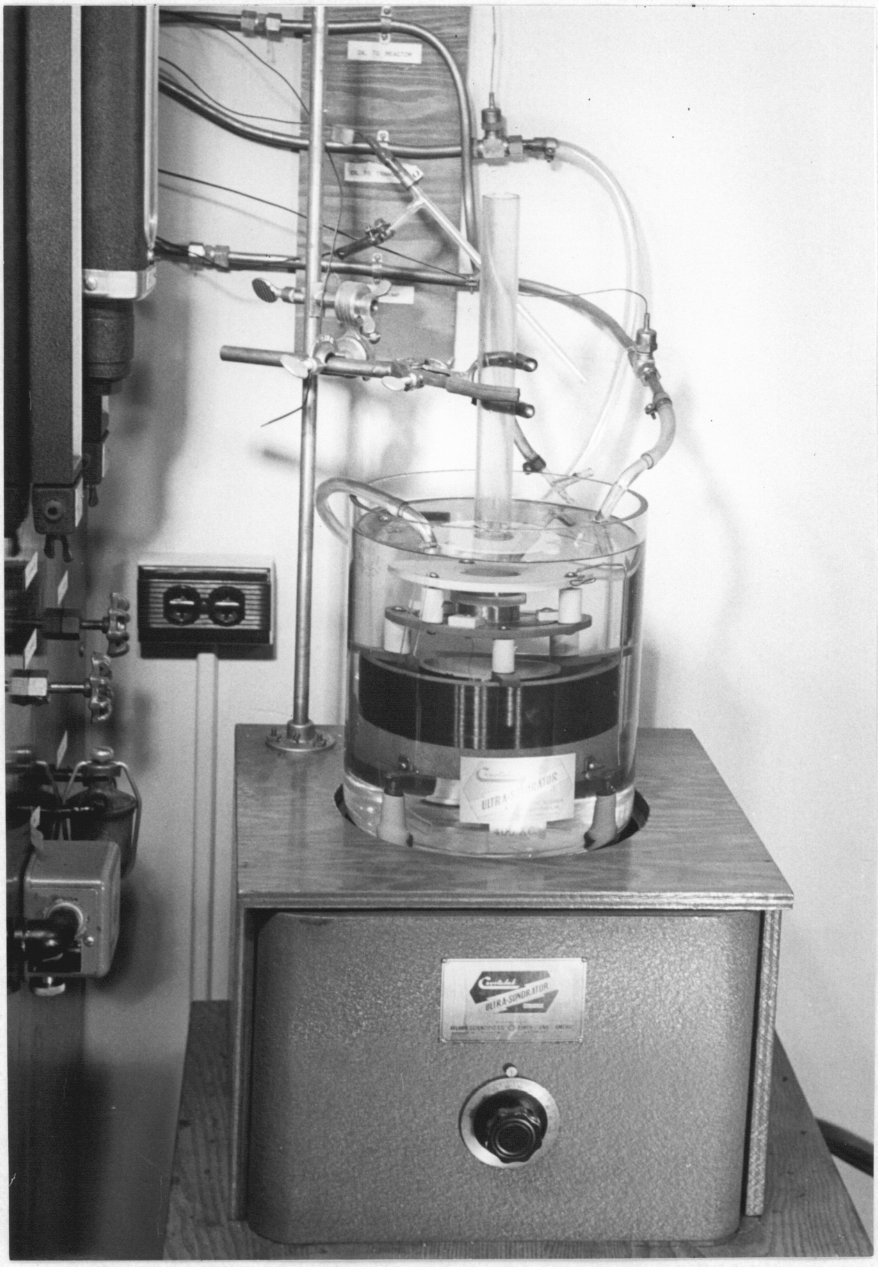


Figure 17. Ultra-Sonorator Transducer Mounted on Impedance Matching Unit with Extractor Vessel Mounted above Flat Transducer Quartz Crystal.

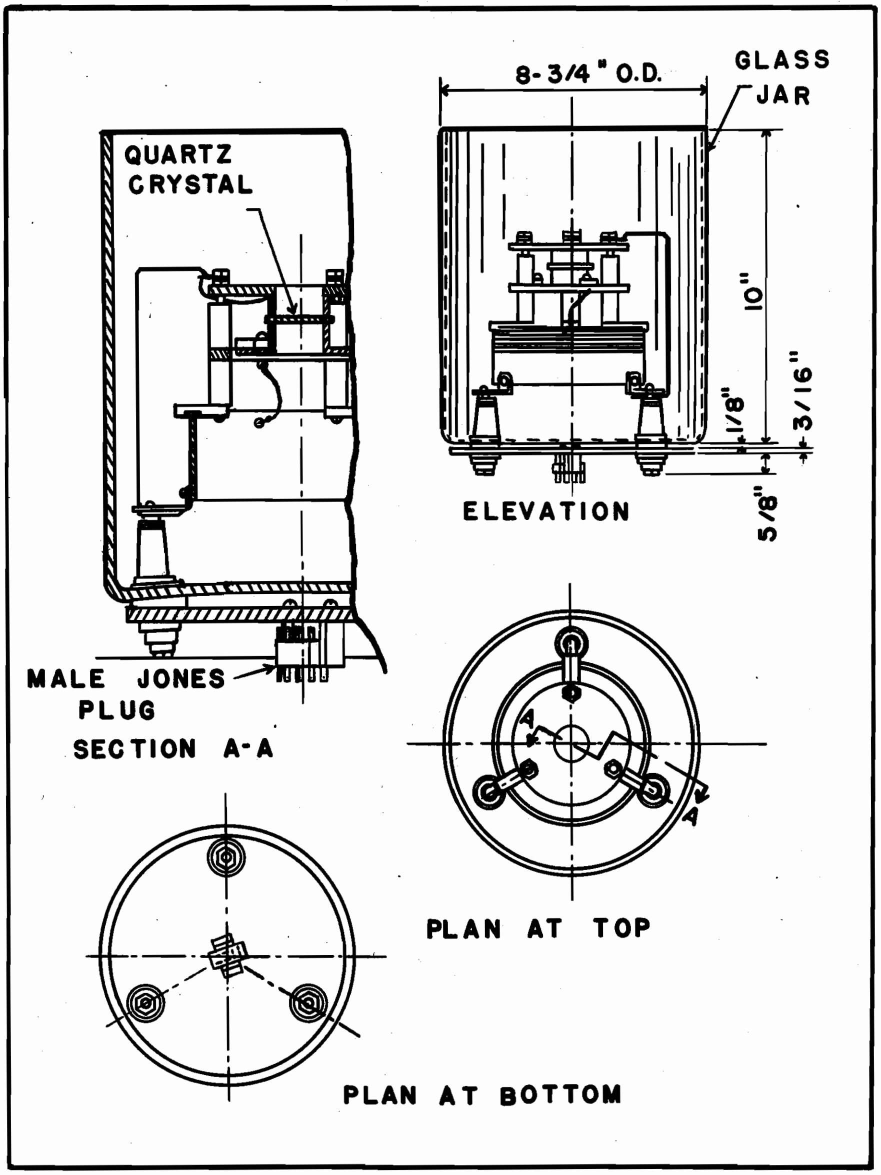


Figure 18. Transducer Design.

Transformer Oil. FS 1866, order No A-4838. Obtained from Fisher Scientific Co., Silver Spring, Md. Used as an insulating and cooling medium in transducer housing of the ultrasonic generator.

Water. Single-distilled. Obtained from the Chemistry Department stock room, Virginia Polytechnic Institute, Blacksburg, Va. Used in calibration of pycnometers and for general purpose cleaning of glassware.

Water. Tap. Obtained from the college water supply, Blacksburg, Va. Used as the cooling medium in the transformer oil heat exchanger, in the condenser of the distillation apparatus used for recovery of hexane, in the reflux condensers of the Soxhlet extractors, and in the air conditioner.

and four oil baths containing mounted crystals that resonate at 400, 700, 1000, and 1500 kilocycles. Each oil bath had a system of coils which connected to the crystal and to the coaxial line through the impedance matching unit.

Air Conditioner. It was necessary to keep humidity of the atmosphere about the generator 60 per cent or lower. When humidity approaches 100 per cent the plastic fiber insulators absorb moisture and are shorted out when high plate voltages are used. When this malfunction occurs it is necessary for factory trained personnel to replace all of the defective relay switches. In order to prevent this situation a model 51H2 Carrier air conditioner, water cooled, for operation on 220 volt, one phase, 60 cycle alternating current, complete with adjustable thermostat was employed.

Mechanical specifications of the air conditioner were as follows: Cooling capacity: 10,600 Btu per hour, based on ASRE standards; compressor: serviceable hermetic type; motor started and operated by one horsepower capacitor; evaporator fan: 4-blade aluminum aerodynamic type; 1/40-horsepower capacitor type reversible motor; speed: 950

revolutions per minute; delivers 400 cubic feet per minute at high speed and 325 cubic feet per minute at low speed; delivers 300 cubic feet per minute in reverse when exhausting from room; outside air delivered: 80 cubic feet per minute while cooling, 350 cubic feet per minute while ventilating; condenser fan: double inlet, centrifugal type, 1/6-horsepower split phase motor, speed of 1725 revolutions per minute, delivers 520 cubic feet per minute; refrigerant: Freon-12, non toxic, non-flamable; discharge air velocity: 700 cubic feet per minute; power consumption: 1450 watts, 7.6 amperes; filters: replaceable, standard size 26-7/8 x 10-1/4 x 1/2-inch; noise level: 44 decibels; dimensions: 40 inches high, 34 inches wide, 21 inches deep; weight: 365 pounds net.

Insonation Extractor. In subjecting the peanut-hexane mixture to ultrasonic insonation, it was necessary to employ a special container, the bottom of which would permit maximum transmission of the ultrasonic waves from the transducer cavity to the peanut-hexane mixture in the container. In a previous investigation, Thompson(112) has shown that a 0.001-inch thick nickel sheet gave best transmission in comparison with a number of other materials such as copper, aluminum, and glass. Also, the diameter of the extractor was limited by the opening of 1-1/2 inches in the transducer cavity through which ultrasonic energy was transmitted from the quartz transducing crystal. Accordingly, the extractor employed in this investigation, Figure 19, page 65, was pyrex glass, 9-1/4 inches high, 1-1/4 inches outside diameter, and 1-1/8 inches inside diameter, with a 0.001-inch nickel disk bonded to the bottom to serve as an acoustic window. Pyrex glass was chosen because of resistivity to corrosion, transparency allowing visual observation of contents of the extractor, and stability to withstand normal handling and the shock of high frequency sound vibrations without fracture of the extractor.

Control Panel for Ultrasonorator. The control panel for the ultrasonorator, shown in Figure 20, page 66, contained the items listed in this section.

Ammeter. Alternating current, 0-15 amps, Type AB-15 switchboard instrument. Obtained from General Electric Co., Roanoke, Va. Used to measure input current to the ultrasonic generator.

Angle Iron. Size 1-1/4 x 1-1/4 x 3/16-inch. Obtained from Noland Co., Roanoke, Va. Used as the main frame and support for the electric, thermometric, and hydraulic control panels.

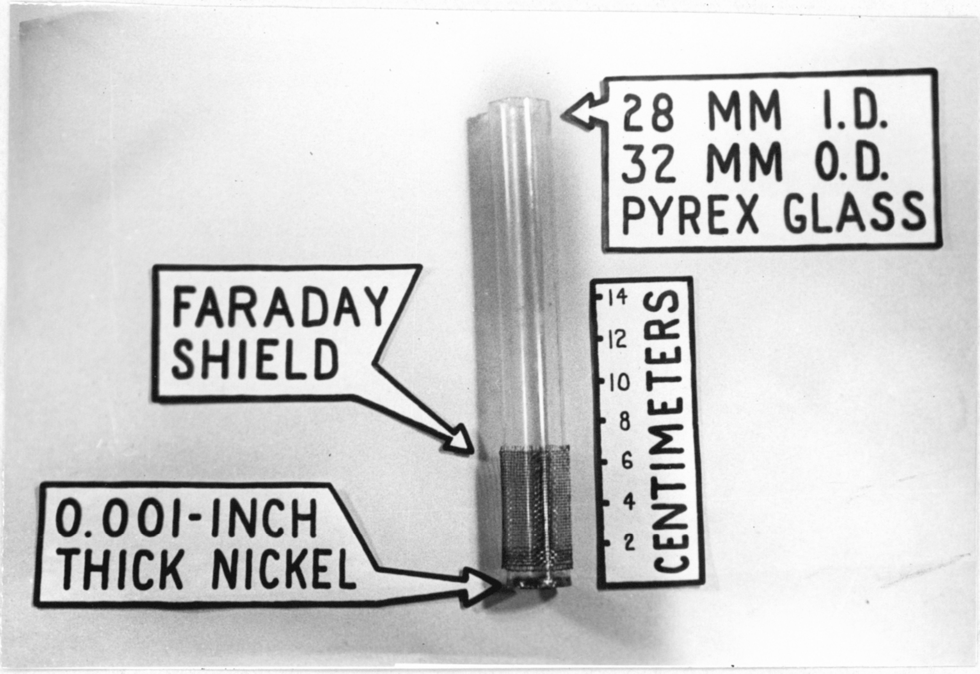


Figure 19. Insonation Extractor

Figure 20. Equipment Layout Showing (Left to Right) Control Panel, Impedance Matching Unit, 400-Kilocycle Transducer, Insonation Extractor, and Radio Frequency Generator.

Bolts, Machine. Square head with nuts, 3/8-inch. Obtained from Brown Stores Co., Blacksburg, Va. Used for the adjustable legs on the main panel frame.

Brush, Varnish. 2-1/2 inches. Obtained from Brown Stores Co., Blacksburg, Va. Used to apply varnish to panel board.

Bushings, Conduit. Size 3/4-inch. Obtained from Electrical Shop, Virginia Polytechnic Institute, Blacksburg, Va. Used in laying the electrical conduit.

Calrod Heater. 1000 watt capacity, 115 volt, 60 cycle, alternating current, copper sheath, catalog No 4A229. Obtained from General Electric Co., Roanoke, Va. Used as the heating unit for the transformer oil constant temperature bath.

Conduit, Electrical. Rigid, 3/4-inch. Obtained from the Electric Shop, Virginia Polytechnic Institute, Blacksburg, Va. Used to convey the controlled voltage electric lines to points of usage.

Condulets. Tees, couplings, and ells, 3/4-inch. Obtained from the Electric Shop, Virginia Polytechnic Institute, Blacksburg, Va. Used to connect electrical conduit.

Connector. Compression male, brass, 3/8-inch, No 68 x 6B. Obtained from W. B. Clements Co., Roanoke, Va. Used in the assembly of copper tubing and instruments for the oil cooling system.

Corner-Irons. Flat, 6-inch. Obtained from Brown Stores Co., Blacksburg, Va. Used for mounting recess boards in the panel board.

Couplings, Conduit. Size 3/4-inch. Obtained from the Electric Shop, Virginia Polytechnic Institute, Blacksburg, Va. Used for connecting the electrical conduit.

Elbow, Compression. Brass, female, 3/8-inch. No 70 x 6. Obtained from W. B. Clements Co., Roanoke, Va. Used in the assembly of copper tubing and instruments for the oil cooling system.

Elbow, Compression. Brass, male, 3/8-inch. No 69 x 6. Obtained from W. B. Clements Co., Roanoke, Va. Used in the assembly of copper tubing and instruments for the oil cooling system.

Filter, Oil. Automobile type, glass, porous ceramic core. Obtained from W. B. Clements Co., Roanoke, Va. Used to filter oil entering the ultrasonic generator transducing unit.

Fittings, Pipe. Galvanized malleable iron, 1/2-inch. Close, 2-inch, 3-inch nipples; ground joint, brass to iron seat unions; straight tees; straight 90° elbows; right hand couplings; square head cored plugs. Obtained from Noland Co., Roanoke, Va. Used in constructing water and air lines.

Hinges, Strap. Size 6-inch. Obtained from Brown Stores Co., Blacksburg, Va. Used for hinging panel board door.

Manometer. Meriam U-type cleanout, Model B-1103 with rear connections. Obtained from Meriam Instrument Co., Cleveland, Ohio. Used to measure the rate of flow of water in the cooling system.

Mixer, Lightnin. Model "L", 110 volt, 60 cycle, alternating current, monel shaft-propeller, tank clamp. Obtained from Mixing Equipment Co., Rochester, N. Y. Used to agitate the cooling water in the external oil cooling tank.

Motor, Air. Type 4AM FCW-10C, V-belt drive, clockwise rotation. Obtained from Gast Manufacturing Co., Benton Harbor, Mich. Used as motor for gear pump used in pumping transformer oil.

Nuts, Lock. Conduit, 3/4-inch. Obtained from the Electric Shop, Virginia Polytechnic Institute, Blacksburg, Va. Used in laying electrical conduit.

Nuts, Hexagonal. Steel, machine screw, No 10, 3/16-inch, 32 threads per inch, catalog No 8474. Obtained from Lucas Motor Co., Blacksburg, Va. Used for fastening plywood panel to angle iron frame.

Paint. Tractor and implement enamel. No 557 Black Sherwin-Williams. Obtained from Brown Stores Co., Blacksburg, Va. Used as the primer and the second coat on the panel board.

Panel Board. Plywood, 1/2-inch, 5-ply. Obtained from New River Lumber Co., Blacksburg, Va. Used for mounting control devices and instruments.

Pipe. Galvanized iron, 1/2-inch. Obtained from Noland Co., Roanoke, Va. Used for water lines.

Pipe Cap. Brass, size 1/4-inch. No 3129 x 4. Obtained from W. B. Clements Co., Roanoke, Va. Used in the assembly of the copper tubing and instruments for the oil cooling system.

Pilot Light and Bracket. No GT 1377. Obtained from General Electric Co., Roanoke, Va. Used as indicator for circuit switches for ultrasonorator, mixer, and calrod heater.

Plastubing. Inside diameter, 3/8-inch. Catalog No 14-169. Obtained from Fisher Scientific Co., Pittsburgh, Pa. Used for connecting glass tubing in transducer unit to copper tubing on oil cooling system.

Plate, Wall. Double opening, Pass and Seymour Chrome-X, catalog No 1791-B. Obtained from General Electric Co., Roanoke, Va. Used as panel front-plate for pilot light and switch.

Power Outlet. Single, T-slot, side wired, 15 amperes, 125 volts, catalog No 1527. Obtained from General Electric Co., Roanoke, Va. Used for coaxial cable connection to the panel board.

Pump, Gear. Capacity 8 gallons per minute, No ROA 203, 3/8-inch inlet and outlet. Obtained from Perfection Pump Co., Milwaukee, Wis. Used to pump transformer oil.

Rotameter. Capacity 0.8 gallon per minute, serial No P4-1101/1. Obtained from Fisher and Porter, Hatboro, Pa. Used to indicate flow of transformer oil to transducer.

Sandpaper. Assorted grits, 20-sheet package. Obtained from Brown Stores Co., Blacksburg, Va. Used for finishing panel board surface.

Screws, Machine. Oval head, No 10, 1-1/2 inches, 32 threads per inch, No 8935. Obtained from Lucas Motor Co., Blacksburg, Va. Used for fastening plywood panel board to angle iron frame.

Shields, Expansion. Machine bolt, 1/4-inch. Obtained from Noland Co., Roanoke, Va. Used for anchoring panel board to wall.

Shut-off Cock. Brass, 1/4-inch, No 6825. Obtained from W. B. Clements Co., Roanoke, Va. Used in the assembly of the copper tubing and instruments for the oil cooling system.

Solder, Wire. Obtained from the Electric Shop, Virginia Polytechnic Institute, Blacksburg, Va. Used for soldering electrical connections and copper tubing fittings.

Straps, Pipe. Size 1/2-inch. Obtained from Noland Co., Roanoke, Va. Used for anchoring compressed air lines.

Switch, Rotary. Ten point, catalog No 8240. Obtained from Leeds and Northrup, Philadelphia, Pa. Used as selector switch for thermocouple circuits.

Tank, Cooling. Specifications: 1/4-inch steel plate, 16 inches diameter x 30 inches length, with 1/2-inch cover, internal baffle and side outlets for cooling coils. Fabricated by C. L. Fielder Co., Roanoke, Va. Used for cooling transformer oil.

Tee, Compression. Brass, 1/4-inch. No 72 x 6. Obtained from W. B. Clements Co., Roanoke, Va. Used in the assembly of the copper tubing and instruments for the oil cooling system.

Tee, Compression. Brass, 3/8-inch. No 64 x 6. Obtained from W. B. Clements Co., Roanoke, Va. Used in the assembly of the copper tubing and instruments for the oil cooling system.

Thermostat. Automatic with temperature setting device, catalog No CR2992. Capillary tube catalog No 4980281G38 with 120-inch tube. Obtained from

General Electric Co., Roanoke, Va. Used for control of oil cooling tank temperature.

Thermostat Accessories. Brass union, brass squeezing nut, two brass discs, one lead packing, catalog No 490073901. Obtained from General Electric Co., Roanoke, Va. Used for mounting thermostat in cooling tank.

Tubing, Copper. Size 3/8-inch inside diameter. Obtained from W. B. Clements Co., Roanoke, Va. Used in the assembly of the copper tubing and instruments for the oil cooling system.

Tumbler. No GT 1391. Obtained from General Electric Co., Roanoke, Va. Used as switch in pilot light assembly.

Union, Compression. Brass, 3/8-inch. No 62 x 6. Obtained from W. B. Clements Co., Roanoke, Va. Used in the assembly of the copper tubing and instruments for the oil cooling system.

Vacuum Bottle. Thermos brand, model No 2280, filler No 22F. Obtained from American Thermos Bottle Co., Norwich, Conn. Used as cold junction box in thermoelectric circuits.

Valves. Brass, 1-inch globe. Obtained from Noland Co., Roanoke, Va. Used in drainage line from oil cooling tank.

Valves. Brass, 3/8-inch needle. Obtained from Noland Co., Roanoke, Va. Used in compressed air lines, oil lines, and water line.

Varnish. Lin-X, clear gloss. Obtained from Brown Stores Co., Blacksburg, Va. Used for finishing coat on panel board.

Voltage Stabilizer. Rated input voltage 115/120/125, output volts-amperes 1000, catalog No 680955. Obtained from General Electric Co., Roanoke, Va. Used to stabilize electrical voltage to ultrasonic generator.

Voltmeter. Alternating current, 1 to 150 volts, type AB-15 switchboard instrument. Obtained from General Electric Co., Roanoke, Va. Used to measure voltage input to voltage stabilizer and to ultrasonic generator.

Washers, Finishing. Countersunk W. F., brass, No 8663. Obtained from Lucas Motor Co., Blacksburg, Va. Used for fastening plywood panel to angle iron frame.

Wire, Electrical. Solid copper, 1-conductor, No 12 AWG. Obtained from the Electric Shop, Virginia Polytechnic Institute, Blacksburg, Va. Used to convey controlled 115 volt, 60 cycle, alternating current from the voltage stabilizer to the points of usage.

Wire, Themocouple. Copper-constantan. Brown Duplex, No 20 gage with fiberglass braid, catalog No 9B1C2. Obtained from Minneapolis-Honeywell Regulator Co., Philadelphia, Pa. Used in conjunction with the potentiometer for temperature measurement.

Extraction Equipment. Equipment employed in this investigation during extraction of oil from peanuts is presented in this section.

Aluminum Foil. Household type. Obtained from Blacksburg A and P, Blacksburg, Va. Used to support peanuts during their drying period.

Beaker. Pyrex glass, capacity 2000 milliliters. Obtained from Fisher Scientific Co., Pittsburgh, Pa. Used to contain peanuts and distilled water during half-hour soaking period preceding flaying of peanuts.

Clamp Holders. Universal castaloy, catalog No 18-822, six required. Obtained from Phipps and Bird, Inc., Richmond, Va. Used to hold extension clamps to ring stand.

Clamps. Brass, extension, wide range V-jar, catalog No 18-702, six required. Obtained from Phipps and Bird, Inc., Richmond, Va. Used to hold condenser, distilling flask, and extractors for all extraction tests.

Compressor, Air. Class WL 70, model AA, shop No AA-1, size 4-1/2 x 4-1/2 x 4-1/2, maximum pressure 125 pounds per square inch, gage, maximum speed 1200 revolutions per minute. Manufactured by Sullivan Machinery Co., Michigan City, Ind. Used to supply 90 pound per square inch, gage, compressed air to drive air motor.

Filter Paper. Qualitative, semi-crimped, rapid filtering, No 9-795, diameter 11 centimeters. Obtained from Fisher Scientific Co., Silver Spring, Md. Used to filter solids from miscella after extraction tests.

Mortar and Pestle. Porcelain, white, 7-inch inside diameter, wall thickness 1 inch. Obtained from Fisher Scientific Co., Philadelphia, Pa. Used to crush peanuts during preparation of feed for extraction tests.

Motor, Air Compressor. Induction type, model SKF226B3, frame 326, type KF, volts 220/440, 60 cycles, 3 phases, speed F. L. 1740, 15 horsepower continuous 40 °C rise, No KR6152. Obtained from General Electric Co., Schenectady, N. Y. Used to provide power to air compressor.

Oven. Fisher Isotemp, type A, regulated temperature range 35 to 200 °C, 115 volts, 60 cycles, 550 watts, catalog No 13-245. Obtained from Fisher Scientific Co., Pittsburgh, Pa. Used to dry peanuts prior to extraction tests.

Pipet. Transfer type, blue line, 25 milliliter. Obtained from Fisher Scientific Co., Pittsburgh, Pa. Used to pipet hexane to extractors during extraction tests.

Rheostat. Specification No 21405, 1000 ohms, maximum amperage 0.224. Obtained from Ohmite Manufacturing Co., Chicago, Ill. Used to control speed of magnetic stirrer during mechanical agitation extraction tests.

Sieve. U. S. standard sieve series No 16. Manufactured by Newark Wire Cloth Co., Newark, N. J. Used to remove peanut particles larger than 0.0474 inch during sizing of peanut feed.

Sieve. U. S. standard sieve series No 20. Manufactured by Newark Wire Cloth Co., Newark, N. J. Used to retain peanut particles between 0.0336 to 0.0474 inch during sizing of peanut feed.

Sieve Lid. U. S. standard sieve lid. Obtained from Newark Wire Cloth Co., Newark, N. J. Used to cover sieve No 16 during sizing of peanut feed to prevent peanut spillage.

Sieve Pan. U. S. standard sieve pan. Obtained from Newark Wire Cloth Co., Newark, N. J. Used to collect peanut particles smaller than 0.0336 inch during sizing of peanut feed.

Stirrer. Magnetic, variable speed, 110 volts, 60 cycles, alternating current. Rotor constructed of solid steel bar 0.5 centimeter in diameter and 1.7 centimeters long. Rotor was encased in pyrex glass tube 0.55 centimeter inside diameter, 2 centimeters long, and 0.1 centimeter wall thickness. Obtained from Arthur H. Thomas Co., Philadelphia, Pa. Used to provide mechanical agitation during extraction tests.

Strobotac. Type No 631-B, serial No 8936, 115 volts, 60 cycles. Manufactured by General Radio Co., Cambridge, Mass. Used to measure speed of rotor in magnetic stirrer.

Analytical Apparatus. Apparatus employed in this investigation during performance of analytical work are presented in this section.

Adapter. Pyrex glass, 90 ° bend. Obtained from the Chemistry Department stockroom, Virginia Polytechnic Institute, Blacksburg, Va. Used to connect condenser to receiver when distilling peanut oil-hexane miscella.

Can. Tin, 8 inches in diameter and 6 inches high. Obtained from Blacksburg A and P, Blacksburg, Va. Used to contain pycnometers and specimen vials in constant temperature bath during time interval between extraction and analysis.

Condenser. Extraction type, Kimble-Brand, No 24027-ST, size 50 millimeters. Obtained from Department of Chemical Engineering stockroom. Used to condense hexane vapors during Soxhlet extraction.

Condenser. Liebig, water condenser, catalog No 7-720. Obtained from Fisher Scientific Co., Pittsburgh, Pa. Used to condense hexane vapors during recovery of hexane from miscella.

Distilling Flask. Side neck type, capacity 500 milliliters, pyrex glass. Obtained from Chemistry Department stockroom. Used to contain miscella during recovery of hexane from miscella.

Extraction Flask. Kimble-Brand, No 24032, with interchangeable joint, capacity 350 milliliters, three required. Obtained from the Department of Chemical Engineering, Virginia Polytechnic Institute, Blacksburg, Va. Used to contain miscella during Soxhlet extraction.

Extraction Thimble. Whatman type, size 43 x 123 millimeters, No 9-656. Manufactured by W. and R. Balston, Ltd., England. Obtained from Fisher Scientific Co., Silver Spring, Md. Used to contain crushed peanuts during Soxhlet extraction.

Fisher Unitized Bath, Jar, and Base. Unit consisted of 12 x 12-inch cylindrical pyrex glass jar and integral control cox containing relay, condenser, resistor, receptacles, switches, signal light, cord and plug for 110 volt, 60 cycle, alternating current, catalog No 15-444-5. Obtained from Fisher Scientific Co., Pittsburgh, Pa. Used to maintain miscellae at 30 ± 0.1 °C during specific gravity determinations.

Pipet. Measuring type, blue line, 1 and 5 milliliter. Obtained from Fisher Scientific Co., Pittsburgh, Pa. Used to pipet peanut oil and hexane to specimen vials during determination of specific gravity versus miscella concentration data.

Pipet. Transfer type, blue line, 10 milliliter. Obtained from Fisher Scientific Co., Pittsburgh, Pa. Used to pipet peanut oil and hexane to specimen vials during determination of specific gravity versus miscella concentration data.

Ring Stand. Rectangular, cast iron base 6 x 9 inches, with copper plated steel rod 24 inches long, three required. Obtained from Fisher Scientific Co., Silver Spring, Md. Used to support clamps for Soxhlet extractor and distillation apparatus used in recovery of hexane from miscella.

Specimen Vials. Glass, capacity 15 milliliters, size 57 x 27 millimeters, No 3-338. Obtained from Fisher Scientific Co., Pittsburgh, Pa. Used to contain miscella after extraction tests.

Specimen Vial Caps. Plastic, screw type, black. Obtained from Fisher Scientific Co., Pittsburgh, Pa. Used to seal 15 milliliter specimen vials.

Thermometer. Glass, mercury indicating fluid, range - 10 to 110 °C with 1 ° subdivisions, three required. Obtained from Fisher Scientific Co., Silver Spring, Md. Used to measure temperature of constant temperature bath, room, and peanut meat-peanut oil-hexane system before and after treatment.

Waring Blender. 115 volts, 60 cycles, 3 amperes, catalog No 700. Obtained from Waring Products Corp., New York, N. Y. Used to agitate peanut-hexane mixture while extracting oil used during determination of specific gravity versus miscella concentration data.

Weights. Analytical, serial No 4-D-2238. Obtained from Seederer-Kohlbusch, Inc., Jersey City, N. J. Used as weights for the analytical balance.

Method of Procedure

In ascertaining the effect of ultrasonic insonation on solvent extraction of oil from peanuts, attention was focused on determination of the per cent oil extracted employing ultrasonic insonation. Comparison was made with extraction employing mechanical agitation and a control under similar thermal conditions. Methods of procedure were formulated, techniques were developed, experimental tests were conducted, and the data collected were compared.

Literature Survey. A survey of the literature was conducted reviewing mass transfer, liquid-solid extraction, physical properties of the system investigated, and previous works pertinent to this investigation that employed ultrasonic energy. The procedure for reviewing articles consisted of preparing a separate typewritten sheet for each abstract. The sheet was filed in a record binder to serve as a convenient source of information for future investigators, as well as to insure against loss of abstracts. Abstracts could be easily located in the record binder by knowing either the author or subject of the article abstracted.

Record of Data. All original data and notes for this investigation were kept in hard-back, bound notebooks with 60 consecutively numbered pages in each notebook. Curves, on separate, properly identified sheets, were attached to notebook pages.

Extractor Location during Insonation. The insonation extractor was clamped in a vertical position, during insonation, with the center-line over the intersection of the vertical and horizontal axes of the quartz crystal (Figure 17, page 59).

To assure that extractor location did not vary vertically for different tests employing ultrasonic insonation, the following procedure was followed: Oil in the transducer housing was adjusted to four centimeters above the transducing crystal. The acoustic window of the extractor was maintained three centimeters above the quartz transducing crystal and one centimeter below the transducer housing oil level. Height of the extension clamp supporting the extractor was adjusted until the bottom metal prong of the extension clamp was five inches above the bottom of the extractor. Position of the clamp holder on the ring stand was marked on the ring stand adjacent to the bottom of the clamp holder.

Position of the extension clamp on the extractor was marked on the extractor five inches above its bottom. The clamp holder and extension clamp remained in fixed position throughout all tests employing ultrasonic insonation to insure duplication of extractor location.

Horizontal position of the insonation extractor was maintained constant by retaining the double prong side of the extension clamp in fixed position, adjusting only the single prong in removing and replacing extractor. Furthermore, the degree of rotation of the extractor about its vertical axis was maintained constant by insuring that the mark on the extractor was located directly below the tip end of the bottom prong of the extension clamp during all insonation tests.

Transducer Oil Cooling System. Transducer oil was circulated through a cooling unit for the purpose of dissipating heat developed in the transformer oil of the transducer. Transducer oil was pumped through copper tubing from the transducer unit to the coils of a cooling tank. A compressed-air, belt-driven pump was employed. After being cooled by a countercurrent flow of tap water outside the copper coil, the oil was filtered through a ceramic filter and returned to the system. A constant volume of oil, 0.500 ± 0.002 gallon per minute, was circulated through the transducer during all tests employing ultrasonic insonation.

Preparing Peanuts for Extraction. Procedure employed in preparing peanuts for extraction is presented under headings of shelling and flaying peanuts, sizing peanuts, and drying peanuts.

Shelling and Flaying Peanuts. Shells were removed from the peanuts by hand, and the shelled peanuts were allowed to soak in distilled water for half-an-hour to loosen the skins. Peanut kernels were separated from the skins by hand, placed on aluminum foil, and dried in the atmosphere for 24 hours.

Sizing Peanut Feeds. Sizing of peanut feeds was accomplished by screening. Peanuts were ground into flakes in a 7-inch inside diameter porcelain mortar with a pestle. The peanut flakes were placed on a U. S. standard sieve number 16. Crushed peanut masses were forced through the 0.0474-inch aperature in the number 16 sieve with a wooden block. Peanut particles which passed a number 16 sieve and were retained on a number 20 sieve (aperature nominal size 0.0336-inch) were dried.

<u>Drying Peanut Feeds</u>. The sized peanut particles were dried at 110 °C for three hours. Peanut particles were transferred to a desiccator after oven drying, where they were stored for 10 to 50 hours. Prior to extraction, peanut feeds were again screened. Peanut particles which passed a number 16 sieve and were retained on a number 20 sieve were employed for extraction.

<u>Analysis of Miscella</u>. All miscellae from extraction tests were analyzed by specific gravity determinations at 30 °C. Because volumes of miscellae available after extraction tests were only 9 to 11 milliliters, five milliliter capacity pycnometers were used for specific gravity analyses. Specific gravity versus concentration of peanut oil in hexane data are given in Table VII, page 89, and are plotted in Figure 21, page 90.

<u>Oil Content of Feed</u>. Oil content of peanut feed was determined by Soxhlet extraction. Peanut samples were prepared by identical procedure and technique to that employed

TABLE VII

Specific Gravities of Peanut Oil-Hexane Miscellae at 30 °C/4 °C

Test		Composition Oil in Hexane, vol %	wt %	Specific Gravity at 30 °C/4 °C
1	(Peanut Oil)	100.00	100.00	0.8979
2		50.00	56.85	0.7941
3		33.33	39.75	0.7569
4		25.00	31.93	0.7408
5		15.00	19.87	0.7160
6		10.00	13.49	0.7039
7		5.00	6.89	0.6913
8	(Hexane)	0.00	0.00	0.6781

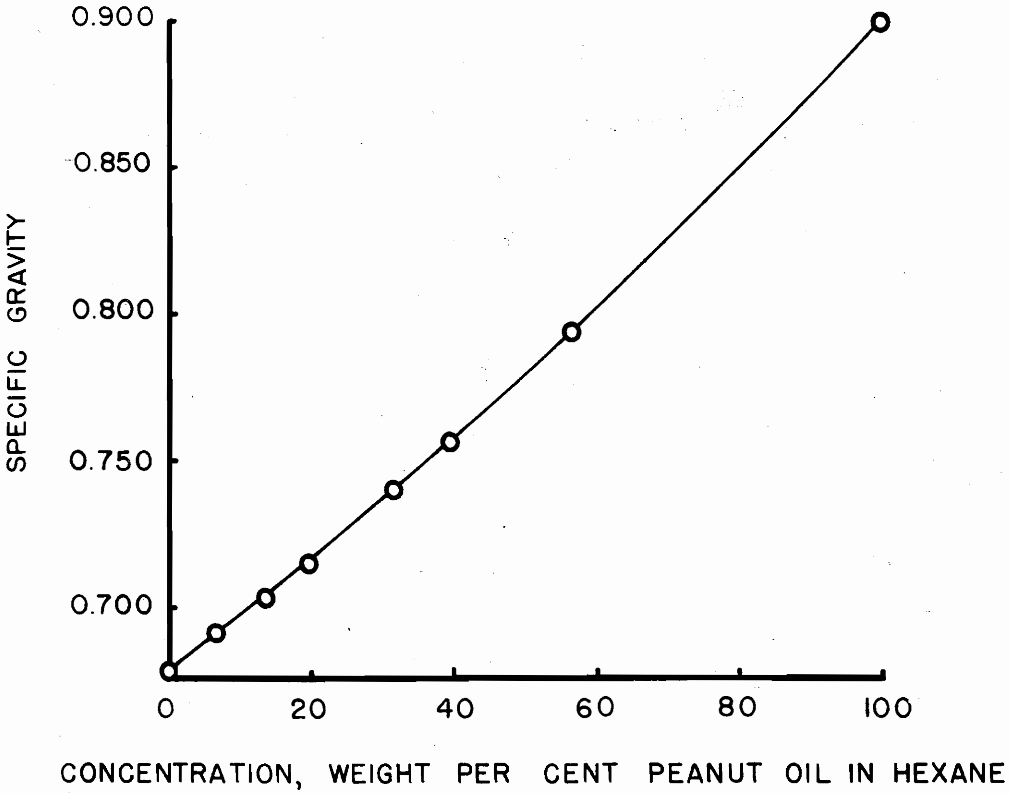


Figure 21. Specific Gravities of Peanut Oil-Hexane Miscellae at 30 °C/4 °C.

in preparing feed for ultrasonic insonation, mechanical agitation, and control tests. Procedure for Soxhlet extraction consisted of weighing Soxhlet thimble both when empty and, subsequently, when containing peanut feed to be tested. Two hundred milliliters of hexane was pipeted into a Soxhlet flask, and the Soxhlet extractor assembled over a laboratory type hot plate. Extraction was allowed to continue for eight hours after the first drop of hexane fell from the reflux condenser. After the eight-hour period the Soxhlet thimble was removed, placed in the atmosphere for one hour, desiccated for 24 hours, and weighed. Difference in filled thimble weight before and after extraction was taken to be the weight of crude oil extracted from the peanuts.

Exploratory Investigations. Exploratory experimentation was necessary to obtain information helpful in fixing certain variables and directing the choice of experimental tests. Exploratory investigations were conducted to determine the following: (1) specific gravity at 30 °C versus concentration curve for peanut oil-hexane miscellae, (2) peanut drying curves, (3) optimum quantity of feed to insonate, (4) optimum solvent-feed ratio, and (5) effect of time of 400-kilocycle insonation at 1000 volts and 160 milliamperes on solvent extraction of peanut oil.

Specific Gravity at 30 °C Versus Concentration of Peanut Oil-Hexane Miscellae. Specific gravities of peanut oil-hexane miscellae varying in concentration were determined. Oil was extracted from peanuts in a Waring blendor, using hexane as the solvent. Distillation of the miscella yielded a quantity of peanut oil sufficient for determination of the specific gravity versus concentration curve. Peanut oil and hexane were mixed in volume ratios of 1:0, 1:1, 1:2, 1:3, 3:17, 1:19, and 0:1. Specific gravity of each miscella at 30 °C was determined, and data are listed in Table VII, page 89, and plotted in Figure 21, page 90.

Drying Peanut Feeds. To insure that moisture content of peanut feeds used in different tests was consistent, drying curves for 0.0336 to 0.0474-inch peanut particles in the atmosphere, oven, and desiccator were determined.

Atmospheric Drying of Peanuts. A sample of sized peanuts weighing 2.6143 grams was allowed to remain in the atmosphere for 94 hours to determine the effect of variation in humidity on moisture content of peanuts. Variations in peanut weights shown in Table VIII, page 94, indicated the necessity for insuring that feeds for all tests had the same moisture content.

Oven Drying of Peanuts. Sized peanut samples were oven dried at 100 °C for 1/4, 1, 3, and 14 hours (Figure 22, page 95). Feed heated three hours became 2.17 per cent lighter, and feed heated 14 hours became 2.21 per cent lighter. A three-hour drying period was adequate in preparation of feeds for extraction investigations, because of the small reduction in weight (0.04 per cent) between 3 and 14 hours of drying.

Desiccation of Peanuts. Sized peanut particles were tested for moisture content by desiccation. Desiccation produced approximately the same reduction in weight of peanut particles (2.25 per cent) as oven drying at 110 °C. The principal difference between desiccation and oven drying at 110 °C was the time rate of weight

TABLE VIII

Data for Peanuts Dried in Atmosphere, Oven, and Desiccator[a]

Test No	Time of Drying, hr	Moisture Loss[b] of Peanut Sample, wt %
Atmospheric Drying:		
1	0	0.00
2	25	0.33
3	53	0.08
4	75	0.21
5	95	0.06
Oven Drying (110 °C):		
6	0	0.00
7	$\frac{1}{4}$	1.93
8	3	2.17
9	14	2.21
Desiccation:		
10	0	0.00
11	25	1.47
12	53	1.90
13	75	2.07

[a] Peanut size was 0.0336 to 0.0474-inch maximum dimension.

[b] Moisture loss was taken to be the reduction in peanut weight during drying.

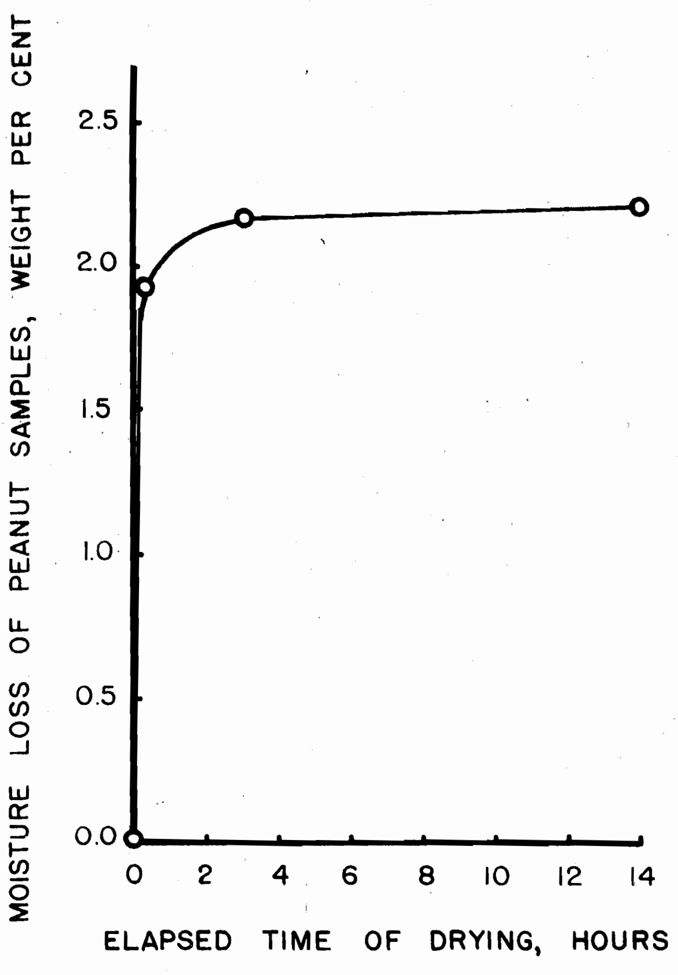


Figure 22. Reduction in Weight of Peanut Particles (0.0336 to 0.0474-Inch) During Drying in a Fisher Isotemp Oven at 110 °C. Moisture Loss Was Taken to Be the Reduction in Peanut Weight During Drying.

reduction as shown in Figure 22, page 95, and Figure 23, page 97.

Optimum Quantity of Feed to Insonate. The optimum quantity of feed to employ during insonation extraction tests was investigated. Employing peanut feed weighing six and ten grams, bulk agitation of the system was not accomplished after five minutes of insonation at a plate potential of 500 volts and a plate current of 80 milliamperes. Five-gram samples of feed, however, were agitated during 500 volt, 80 milliampere insonation. Therefore, all feeds employed in extraction tests weighed approximately five grams.

Optimum Solvent-Feed Ratio. Effects of solvent-feed ratios varying from 1:1 to 5:1 on solvent extraction of oil from peanuts were determined. Four hundred-kilocycle insonation at 1250 volts and 210 milliamperes for two minutes was employed. Using solvent-feed ratios of 1.5:1 and 2:1 necessitated analyses of miscellae with two milliliter pycnometers, because miscellae volumes were less than five milliliters. Miscella volume from investigation of solvent-feed ratio of 1:1 was less than two milliliters and was

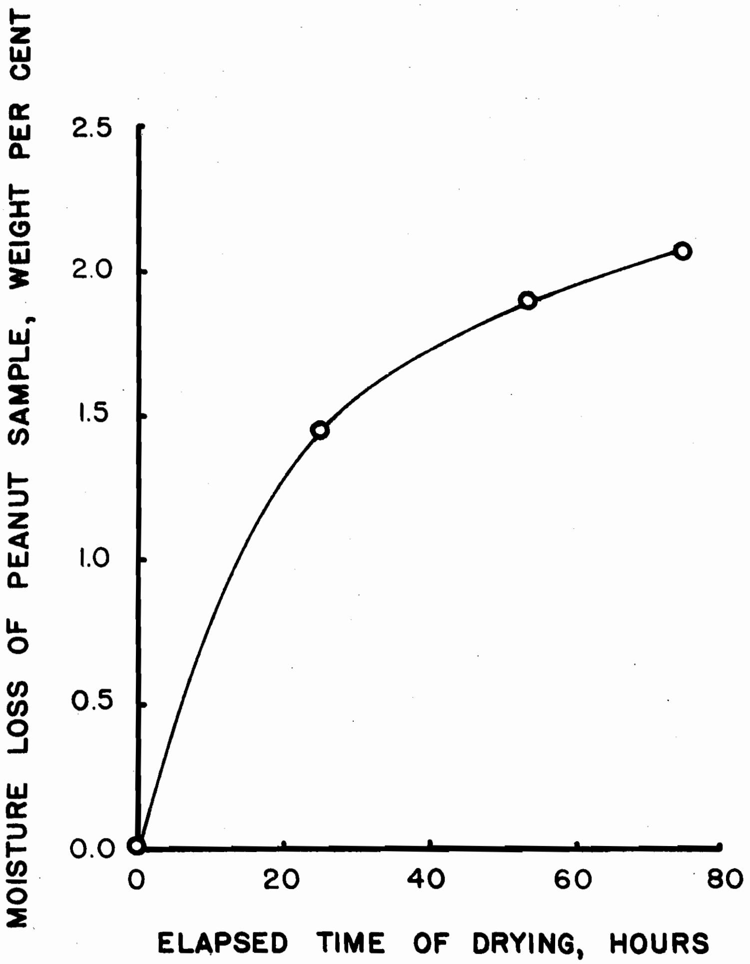


Figure 23. Reduction in Weight of 0.0336 to 0.0474-Inch Peanut Particles During Desiccation at 23.0 ± 1.0 °C. Moisture Loss Was Taken to Be the Reduction in Peanut Weight During Drying.

not analyzed. However, extraction employing a solvent-feed ratio of 3:1 yielded sufficient quantity of miscella to allow analysis in a five-milliliter pycnometer. A solvent-feed ratio larger than 3:1 was not advantageous because of the appreciable decrease in miscella concentration with increasing solvent employed, as shown in Figure 24, page 100.

Effect of Time of 400-Kilocycle Insonation at 1000 Volts and 160 Milliamperes on Solvent Extraction of Peanut Oil. Miscellae concentrations, after 2, 5, 15, and 30 minutes of 400-kilocycle insonation at 1000 volts and 160 milliamperes, are listed in Table X, page 101, and plotted in Figure 25, page 102. The most rapid rate of extraction occurred within the first six minutes of insonation; therefore, attention was focused on this portion of the extraction curves during subsequent tests.

Tests Employing Ultrasonic Insonation. Extraction tests were conducted employing ultrasonic insonation. Peanut particles weighing 5.1220 grams were transferred to the insonation extractor, which was then placed in position as described on page 85. Twenty-five milliliters of hexane

TABLE IX

Concentration of Miscellae for Various Solvent-Feed Ratios Employing Two-minute, 400-Kilocycle Insonation at 1250 Volts Plate Potential and 210 Milliamperes Plate Current

Test No	Solvent-Feed Ratio, wt basis	Specific Gravity at 30 °C/4 °C	Concentration of Miscella, wt % oil in hexane
1	1:1	-	-
2	1.5:1	0.7224	22.8
3	2:1	0.7119	17.5
4	3:1	0.7040	13.5
5	5:1	0.6921	7.3

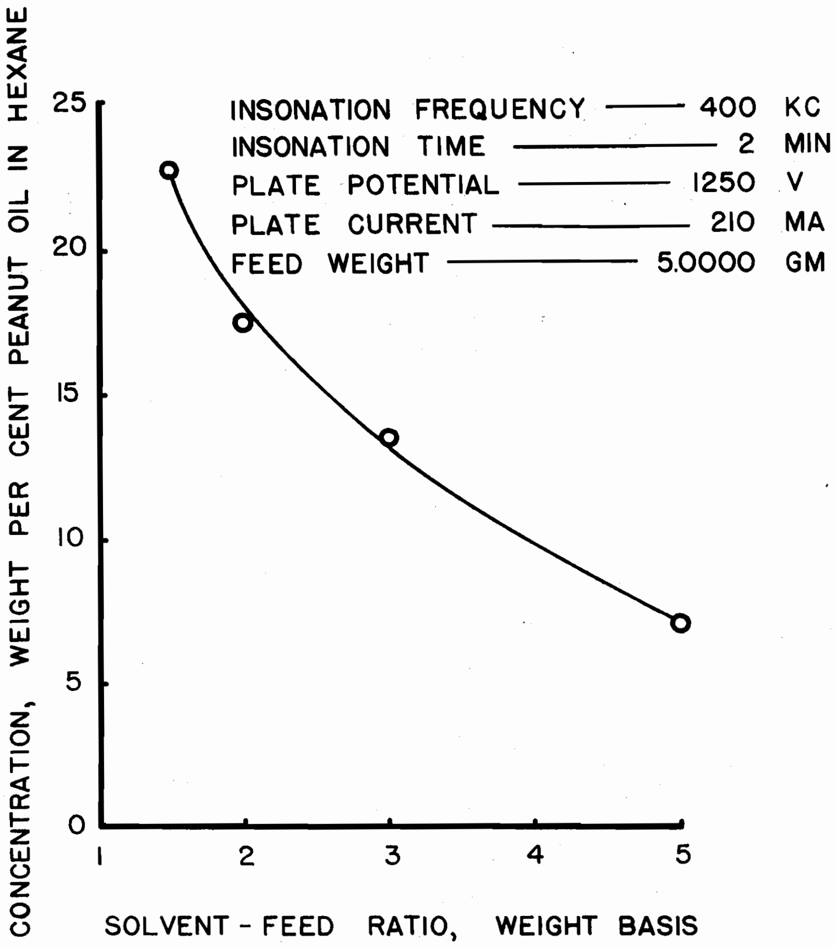


Figure 24. Effects of Solvent-Feed Ratios on Concentration of Miscellas.

TABLE X

Concentration of Miscellas for Various Periods of Insonation Employing a Solvent-Feed Ratio of 3.3:1 and 400-Kilocycle Insonation at 1000 Volts Plate Potential and 160 Milliamperes Plate Current

Test No	Elapsed Time of Insonation min	Specific Gravity at 30 °C/4 °C	Concentration of Miscella wt % oil in hexane
1	0	0.6840	3.1
2	2	0.6949	8.8
3	5	0.6969	9.8
4	15	0.7053	14.2
5	30	0.7160	19.7

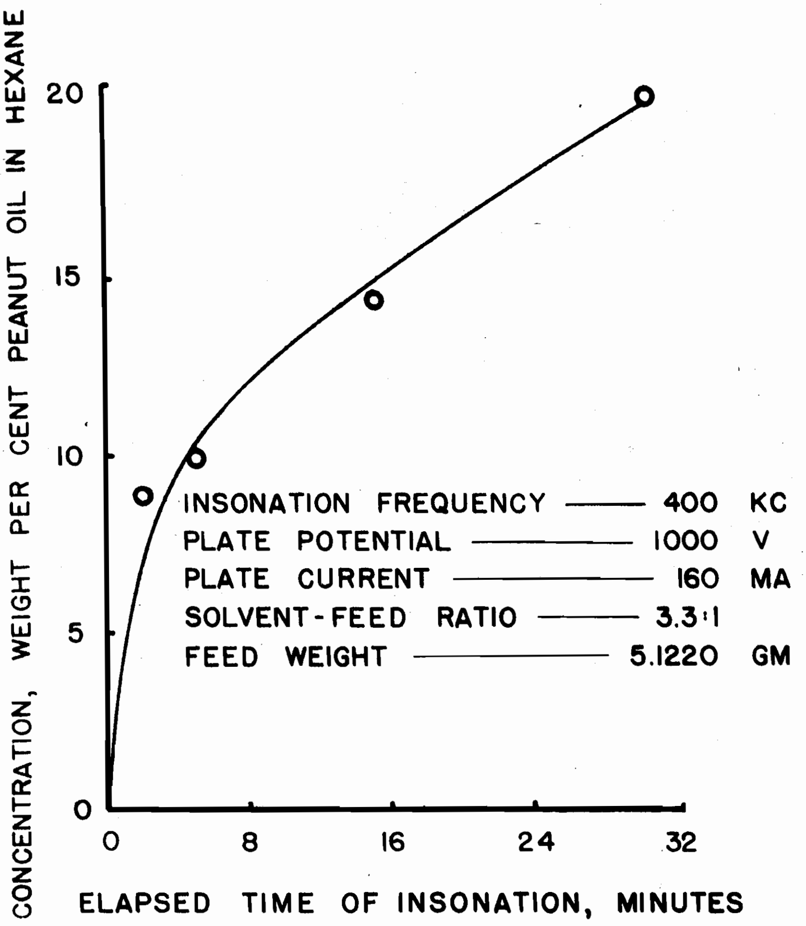


Figure 25. Effects of Elapsed Time of Insonation on Concentration of Miscellae.

at 23.0 ± 1.0 °C were pipeted into the extractor; the time that hexane began to drain from the pipet was recorded as zero time. At the end of 30 seconds, ultrasonic energy was applied at the plate potentials shown in Table XI, page 104. Fifteen seconds after insonation the miscella was filtered, and the filtered miscella was transferred to a 15 milliliter specimen vial. The vial was sealed, and the appropriate test number marked on the vial screw cap.

Tests Employing Mechanical Agitation. Solvent extraction was investigated employing mechanical agitation in the form of magnetic stirring at rotor speed settings of 10 and 50. Strobotac measurements showed a rotor speed of 675 revolutions per minute at a rotor speed setting of 10, and 1242 revolutions per minute at a setting of 50. The agitation extractor was placed in a vertical position on the center of the magnetic stirrer housing. Procedure then followed was identical to the procedure followed for tests employing ultrasonic insonation.

Plan of Evaluation. In the study of the effects of ultrasonic insonation on solvent extraction of oil from peanuts, the variables of intensity and time of 400-kilocycle insonation were investigated. Comparable extraction tests

TABLE XI

Outline of Experimental Tests Conducted

			Elapsed Time of Insonation, min						
			0	¼	½	1	2	4	6
400-Kilocycle Insonation	Plate Potential, v	500							
		1000							
		1250							
		1500							
Mechanical Agitation	Rotor Speed, rpm	675							
		1242							
Thermal Control Corres-	Temperature Range, °C	25.6-44.5							
		25.6-65.8							
		25.6-67.0							
		25.6-67.0							

were conducted employing mechanical agitation. Extraction under thermal conditions similar to those encountered during insonation was investigated. Control and reproducibility tests were also conducted.

Effect of Intensity of Insonation on Extraction. Intensity of insonation, indicated by the product of plate potential and plate current, was varied holding time of insonation constant. Plate potentials and plate currents were read directly during experimental tests, and were controlled by the ultrasonic generator powerstat. Elapsed time of insonation versus oil extracted from peanut feed was plotted for each of the four intensities investigated.

Effect of Time of Insonation on Extraction. Time of insonation was varied holding intensity of insonation constant. Insonation periods of 0, 1/4, 1/2, 1, 2, 4, and 6 minutes were investigated at each of the four plate potentials shown in Table XI, page 104. Elapsed time of insonation was read directly from the calibrated electric timer. Effects of different periods of insonation on extraction for constant intensities of insonation were shown in plots of extraction efficiency versus elapsed time of insonation.

Effect of Mechanical Agitation on Extraction. For mechanical agitation extraction tests, magnetic stirring at 675 and 1242 revolutions per minute was employed for agitation periods of 0, 1/4, 1/2, 1, 2, 4, and 6 minutes. Periods of agitation were ascertained by an electric timer. Effect of time of agitation on extraction was shown by plots of elapsed time of agitation versus oil extracted from peanut feeds for each of the two agitation intensities investigated.

Thermal Effect of Insonation. During determination of the effect of intensity and time of insonation on extraction, temperature of the system was measured both before and after insonation. Plots were made of elapsed time of insonation versus differential between system and ambient temperature for each of the four intensities investigated. Comparison was made between ultrasonic extraction and control extraction under similar thermal conditions.

Data and Results

Data and results of this investigation, effects of ultrasonic insonation on solvent extraction of oil from peanuts, are presented under two main headings: exploratory investigation and experimental data and results.

Exploratory Investigation. Data for the exploratory investigation performed prior to determining effects of ultrasonic insonation on extraction were presented in the procedure section, pages 91 through 103. Specific gravity at 30 °C versus miscella concentration data are listed in Table VII, page 89, and plotted in Figure 21, page 90. Data for the drying of peanuts in atmosphere, oven, and desiccator are given in Table VIII, page 94. Curves for oven drying of peanuts are plotted in Figure 22, page 95, and for desiccation in Figure 23, page 97. Extraction data for different solvent-feed ratios are given in Table IX, page 99, and plotted in Figure 24, page 100. Extraction data for different periods of 400 kilocycle insonation at 1000 volts and 160 milliamperes are given in Table X, page 101, and plotted in Figure 25, page 102.

Experimental Data and Results. The experimental data and results are presented in five sections: ultrasonic insonation tests, mechanical agitation tests, thermal tests, control tests, and reproducibility tests.

Ultrasonic Insonation Tests. The experimental data and results of tests employing ultrasonic insonation are shown in Table XII, page 109, and plotted in Figure 26, page 110. Thermal effects of insonation are also given in Table XII and are plotted in Figure 27, page 111.

Mechanical Agitation Tests. The experimental data and results of tests employing mechanical agitation are shown in Table XIII, page 112, and plotted with ultrasonic insonation curves in Figure 28, page 113.

Thermal Tests. Experimental data and results for tests employing thermal conditions similar to those encountered in ultrasonic insonation tests are given in Table XIII, page 112, and plotted in Figure 29, page 114.

Control Tests. Experimental information for the control tests at 23.0 ± 1.0 °C are presented in Table XIII, page 112. Control data are plotted with

TABLE XII

Operational Data and Results for Ultrasonic Solvent Extraction of Peanut Oil[a]

Test No	Insonation						Analysis	
	Elapsed Time[b], min	Plate			Powerstat Setting	Temperature Differential[d], °C	Specific Gravity at 30 °C/4 °C	Crude Oil Extraction Efficiency[e], wt %
		Potential, v	Current, ma	$E \cdot I$[c], v-a				
1	0	500	80	40	31.5	2.6	0.6839	18.8
2	¼	500	80	40	31.5	-	0.6920	46.7
3	½	500	80	40	31.5	-	0.6955	59.6
4	1	500	80	40	31.5	9.1	0.6966	63.9
5	2	500	80	40	31.5	13.0	0.6983	70.6
6	4	500	80	40	31.5	18.8	0.6994	74.8
7	6	500	80	40	31.5	21.5	0.7002	77.9
8	0	500	80	40	31.5	-	0.6840	19.1
9	¼	500	80	40	31.5	-	0.6922	47.5
10	½	500	80	40	31.5	-	0.6955	59.6
11	1	500	80	40	31.5	-	0.6966	63.9
12	2	500	80	40	31.5	-	0.6982	70.2
13	4	500	80	40	31.5	-	0.6993	74.6
14	6	500	80	40	31.5	-	0.7003	77.9
15	0	500	80	40	31.5	-	0.6838	18.7
16	¼	500	80	40	31.5	-	0.6922	47.1
17	½	500	80	40	31.5	-	0.6953	59.0
18	1	500	80	40	31.5	-	0.6968	64.5
19	2	500	80	40	31.5	-	0.6983	70.4
20	4	500	80	40	31.5	-	0.6995	74.9
21	6	500	80	40	31.5	-	0.7002	77.8
22	0	500	80	40	31.5	-	0.6838	18.7
23	¼	500	80	40	31.5	-	0.6920	47.0
24	½	500	80	40	31.5	-	0.6952	59.0
25	1	500	80	40	31.5	-	0.6967	63.9
26	2	500	80	40	31.5	-	0.6983	70.8
27	4	500	80	40	31.5	-	0.6995	74.8
28	6	500	80	40	31.5	-	0.7003	78.2
29	0	1000	160	160	60.0	2.6	0.6839	18.8
30	¼	1000	160	160	60.0	-	0.6929	50.4
31	½	1000	160	160	60.0	-	0.6960	61.8
32	1	1000	160	160	60.0	14.4	0.6987	72.0
33	2	1000	160	160	60.0	24.0	0.7007	79.7
34	4	1000	160	160	60.0	36.8	0.7018	84.3
35	6	1000	160	160	60.0	42.8	0.7020	85.1
36	0	1000	160	160	60.0	-	0.6838	18.4
37	¼	1000	160	160	60.0	-	0.6927	49.5
38	½	1000	160	160	60.0	-	0.6960	61.9
39	1	1000	160	160	60.0	-	0.6987	72.2
40	2	1000	160	160	60.0	-	0.7006	79.6
41	4	1000	160	160	60.0	-	0.7017	84.1
42	6	1000	160	160	60.0	-	0.7020	85.0
43	0	1000	160	160	60.0	-	0.6839	18.7
44	¼	1000	160	160	60.0	-	0.6926	49.4
45	½	1000	160	160	60.0	-	0.6961	62.1
46	1	1000	160	160	60.0	-	0.6986	71.5
47	2	1000	160	160	60.0	-	0.7007	79.9
48	4	1000	160	160	60.0	-	0.7018	84.3
49	6	1000	160	160	60.0	-	0.7020	85.1
50	0	1000	160	160	60.0	-	0.6839	18.7
51	¼	1000	160	160	60.0	-	0.6928	50.0
52	½	1000	160	160	60.0	-	0.6959	61.5
53	1	1000	160	160	60.0	-	0.6985	71.3
54	2	1000	160	160	60.0	-	0.7008	80.2
55	4	1000	160	160	60.0	-	0.7017	83.9
56	6	1000	160	160	60.0	-	0.7020	85.2
57	0	1250	210	263	75.0	2.6	0.6839	18.7
58	¼	1250	210	263	75.0	-	0.6935	52.3
59	½	1250	210	263	75.0	12.8	0.6964	63.4
60	1	1250	210	263	75.0	25.8	0.6994	74.6
61	2	1250	210	263	75.0	41.7	0.7011	81.7
62	4	1250	210	263	75.0	43.5	0.7021	85.7
63	6	1250	210	263	75.0	44.0	0.7024	86.7
64	0	1250	210	263	75.0	-	0.6840	19.0
65	¼	1250	210	263	75.0	-	0.6932	51.2
66	½	1250	210	263	75.0	-	0.6966	63.9
67	1	1250	210	263	75.0	-	0.6992	74.0
68	2	1250	210	263	75.0	-	0.7011	81.7
69	4	1250	210	263	75.0	-	0.7021	85.7
70	6	1250	210	263	75.0	-	0.7024	86.9
71	0	1250	210	263	75.0	-	0.6840	19.1
72	¼	1250	210	263	75.0	-	0.6934	51.8
73	½	1250	210	263	75.0	-	0.6965	63.7
74	1	1250	210	263	75.0	-	0.6991	73.8
75	2	1250	210	263	75.0	-	0.7012	82.0
76	4	1250	210	263	75.0	-	0.7020	85.4
77	6	1250	210	263	75.0	-	0.7024	86.5
78	0	1250	210	263	75.0	-	0.6840	19.0
79	¼	1250	210	263	75.0	-	0.6936	52.8
80	½	1250	210	263	75.0	-	0.6964	63.2
81	1	1250	210	263	75.0	-	0.6990	73.4
82	2	1250	210	263	75.0	-	0.7011	81.5
83	4	1250	210	263	75.0	-	0.7021	85.8
84	6	1250	210	263	75.0	-	0.7024	87.1
85	0	1500	260	390	85.0	2.6	0.6839	18.8
86	¼	1500	260	390	85.0	-	0.6936	52.7
87	½	1500	260	390	85.0	14.8	0.6970	65.4
88	1	1500	260	390	85.0	29.8	0.7000	76.8
89	2	1500	260	390	85.0	41.8	0.7018	84.2
90	4	1500	260	390	85.0	43.5	0.7029	89.0
91	6	1500	260	390	85.0	44.0	0.7032	90.0
92	0	1500	260	390	85.0	-	0.6838	18.6
93	¼	1500	260	390	85.0	-	0.6934	52.0
94	½	1500	260	390	85.0	-	0.6970	65.2
95	1	1500	260	390	85.0	-	0.6999	76.4
96	2	1500	260	390	85.0	-	0.7018	84.1
97	4	1500	260	390	85.0	-	0.7028	88.5
98	6	1500	260	390	85.0	-	0.7033	90.4
99	0	1500	260	390	85.0	-	0.6838	18.7
100	¼	1500	260	390	85.0	-	0.6938	53.5
101	½	1500	260	390	85.0	-	0.6970	65.7
102	1	1500	260	390	85.0	-	0.7001	77.2
103	2	1500	260	390	85.0	-	0.7018	84.2
104	4	1500	260	390	85.0	-	0.7028	88.7
105	6	1500	260	390	85.0	-	0.7033	90.2
106	0	1500	260	390	85.0	-	0.6840	19.0
107	¼	1500	260	390	85.0	-	0.6934	51.8
108	½	1500	260	390	85.0	-	0.6971	66.1
109	1	1500	260	390	85.0	-	0.7002	77.6
110	2	1500	260	390	85.0	-	0.7019	84.5
111	4	1500	260	390	85.0	-	0.7028	88.3
112	6	1500	250	390	85.0	-	0.7033	90.4

---

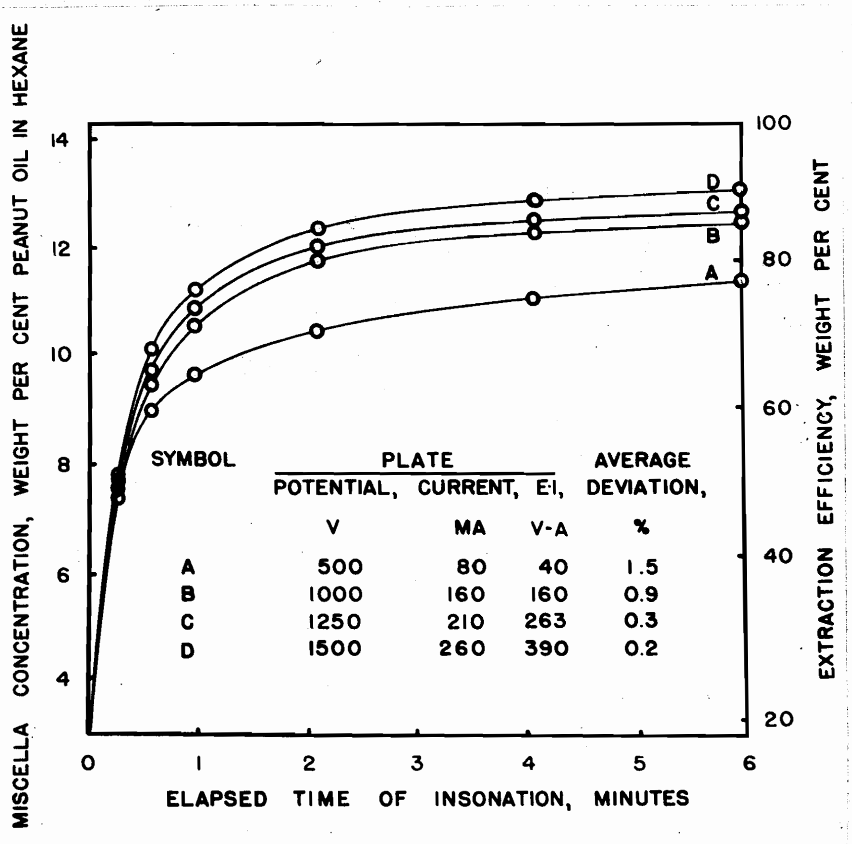
[a] Following conditions maintained for all tests: Crystalab ultra-sonorator model SL 520 employed; insonation frequency, 400 kilocycles; impedance setting, 40.5; line potential, 119-21 volts; transducer oil flow rate, 0.500 ± 0.002 gallons per minute; cooling water flow rate to heat exchanger, 8.8 ± 0.4 gallons per minute; room temperature 23.0 ± 1.0 °C during extraction and 22.2 ± 0.2 °C during analysis.

[b] Insonation preceded by 30-second period to allow solvent to drain from pipet, and followed by 15-second period to allow transfer of miscella to specimen vial.

[c] Product of plate potential and current (apparent power).

[d] Computed by subtracting ambient temperature from temperature of system at conclusion of insonation.

[e] Computed by dividing oil yield from ultrasonic tests by oil yield from soxhlet determinations (weight basis).



SYMBOL	PLATE POTENTIAL,	PLATE CURRENT,	PLATE E·I,	AVERAGE DEVIATION,
	V	MA	V-A	%
A	500	80	40	1.5
B	1000	160	160	0.9
C	1250	210	263	0.3
D	1500	260	390	0.2

Extraction efficiency represents weight of peanut oil extracted during insonation divided by weight of peanut oil extracted during Soxhlet extraction.

E·I represents product of plate potential and plate current. For a power factor of one the volt-ampere (v-a) values represent power.

Average deviation represents deviation of experimental from calculated extraction values.

Figure 26. Effect of Intensity and Time of Insonation on Solvent Extraction of Oil from Peanuts.

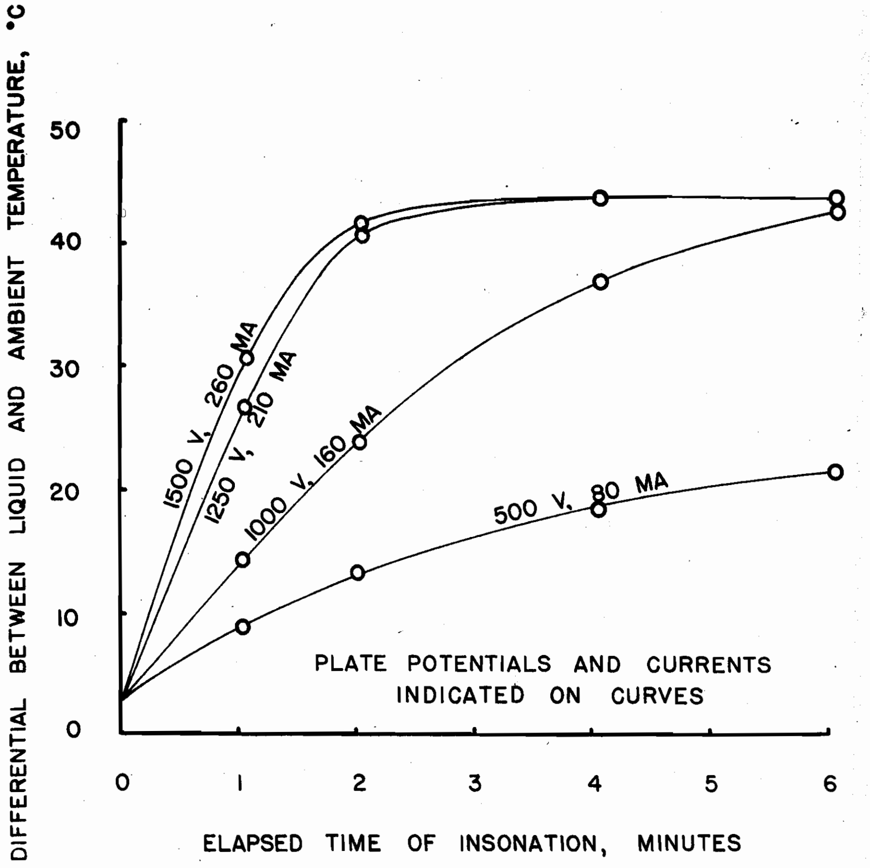


Figure 27. Effect of Intensity and Time of Insonation at 400 Kilocycles on Temperature Variation of Peanut Meat-Peanut Oil-Hexane System.

TABLE XIII

Operational Data and Results for Mechanical Agitation and Control Solvent Extraction of Peanut Oil

Test No	Agitation: Elapsed Time[a], min	Agitation: Rotor Speed[b], rpm	Agitation: Temperature Differential[c], °C	Analysis: Specific Gravity at 30 °C/4 °C	Analysis: Crude Oil Extraction Efficiency[d], wt %
Mechanical Agitation:					
113	0	675	1.2	0.6839	18.8
114	¼	675	-	0.6908	42.2
115	½	675	-	0.6932	51.6
116	1	675	1.4	0.6954	59.7
117	2	675	1.6	0.6972	66.3
118	4	675	2.0	0.6982	70.2
119	6	675	2.4	0.6989	72.7
120	0	675	-	0.6839	18.8
121	¼	675	-	0.6907	42.0
122	½	675	-	0.6931	50.9
123	1	675	-	0.6953	59.2
124	2	675	-	0.6972	66.3
125	4	675	-	0.6983	70.5
126	6	675	-	0.6989	72.7
127	0	675	-	0.6838	18.6
128	¼	675	-	0.6908	42.4
129	½	675	-	0.6932	51.2
130	1	675	-	0.6954	59.6
131	2	675	-	0.6973	66.6
132	4	675	-	0.6984	70.7
133	6	675	-	0.6987	72.3
134	0	675	-	0.6839	18.8
135	¼	675	-	0.6909	42.4
136	½	675	-	0.6932	51.6
137	1	675	-	0.6952	58.7
138	2	675	-	0.6973	66.8
139	4	675	-	0.6983	70.6
140	6	675	-	0.6988	72.5
141	0	1242	1.2	0.6839	18.8
142	¼	1242	-	0.6993	74.7
143	½	1242	-	0.7016	83.5
144	1	1242	1.6	0.7026	87.5
145	2	1242	1.7	0.7034	90.5
146	4	1242	2.5	0.7037	92.1
147	6	1242	3.3	0.7041	94.0
148	0	1242	-	0.6840	19.1
149	¼	1242	-	0.6997	75.6
150	½	1242	-	0.7018	84.5
151	1	1242	-	0.7025	87.1
152	2	1242	-	0.7035	91.4
153	4	1242	-	0.7038	92.6
154	6	1242	-	0.7041	93.7
155	0	1242	-	0.6838	18.7
156	¼	1242	-	0.6993	74.7
157	½	1242	-	0.7016	83.7
158	1	1242	-	0.7026	87.5
159	2	1242	-	0.7034	90.5
160	4	1242	-	0.7038	92.5
161	6	1242	-	0.7041	94.1
162	0	1242	-	0.6838	18.7
163	¼	1242	-	0.6997	76.0
164	½	1242	-	0.7014	83.0
165	1	1242	-	0.7027	88.1
166	2	1242	-	0.7033	90.4
167	4	1242	-	0.7039	92.7
168	6	1242	-	0.7042	94.2
Control (23.0 ± 1.0 °C):					
169	0	-	1.2	0.6838	18.7
170	¼	-	-	0.6842	20.0
171	½	-	-	0.6844	20.8
172	1	-	1.2	0.6848	22.0
173	2	-	1.2	0.6854	23.9
174	4	-	1.2	0.6867	28.6
175	6	-	1.2	0.6879	32.7
176	0	-	-	0.6839	18.8
177	¼	-	-	0.6842	19.9
178	½	-	-	0.6844	20.9
179	1	-	-	0.6849	22.4
180	2	-	-	0.6855	24.1
181	4	-	-	0.6868	28.6
182	6	-	-	0.6878	32.6
183	0	-	-	0.6839	18.8
184	¼	-	-	0.6842	20.1
185	½	-	-	0.6842	20.1
186	1	-	-	0.6849	22.1
187	2	-	-	0.6855	24.0
188	4	-	-	0.6867	28.3
189	6	-	-	0.6880	32.9
190	0	-	-	0.6839	18.9
191	¼	-	-	0.6841	19.5
192	½	-	-	0.6843	20.5
193	1	-	-	0.6849	22.2
194	2	-	-	0.6855	24.2
195	4	-	-	0.6867	28.1
196	6	-	-	0.6878	32.5
Control (25.6 °C to 44.5 °C):					
197	0	-	2.6	0.6839	18.9
198	¼	-	-	-	-
199	½	-	-	-	-
200	1	-	9.1	0.6850	22.6
201	2	-	13.0	0.6858	25.0
202	4	-	18.8	0.6869	29.0
203	6	-	21.5	0.6881	33.2
Control (25.6 °C to 65.8 °C):					
204	0	-	2.6	0.6838	18.7
205	¼	-	-	-	-
206	½	-	-	-	-
207	1	-	14.4	0.6859	25.6
208	2	-	24.0	0.6876	31.7
209	4	-	36.8	0.6909	42.8
210	6	-	42.8	0.6936	52.7
Control (25.6 °C to 67.0 °C):					
211	0	-	2.6	0.6839	18.9
212	¼	-	-	-	-
213	½	-	12.8	-	-
214	1	-	25.8	0.6897	38.8
215	2	-	41.7	0.6929	50.2
216	4	-	43.5	0.6950	58.2
217	6	-	44.0	0.6960	61.4
Control (25.6 °C to 62.0 °C):					
218	0	-	2.6	0.6840	19.1
219	¼	-	-	-	-
220	½	-	14.8	-	-
221	1	-	29.8	0.6912	43.5
222	2	-	41.8	0.6934	52.0
223	4	-	43.5	0.6950	58.1
224	6	-	44.0	0.6960	61.4

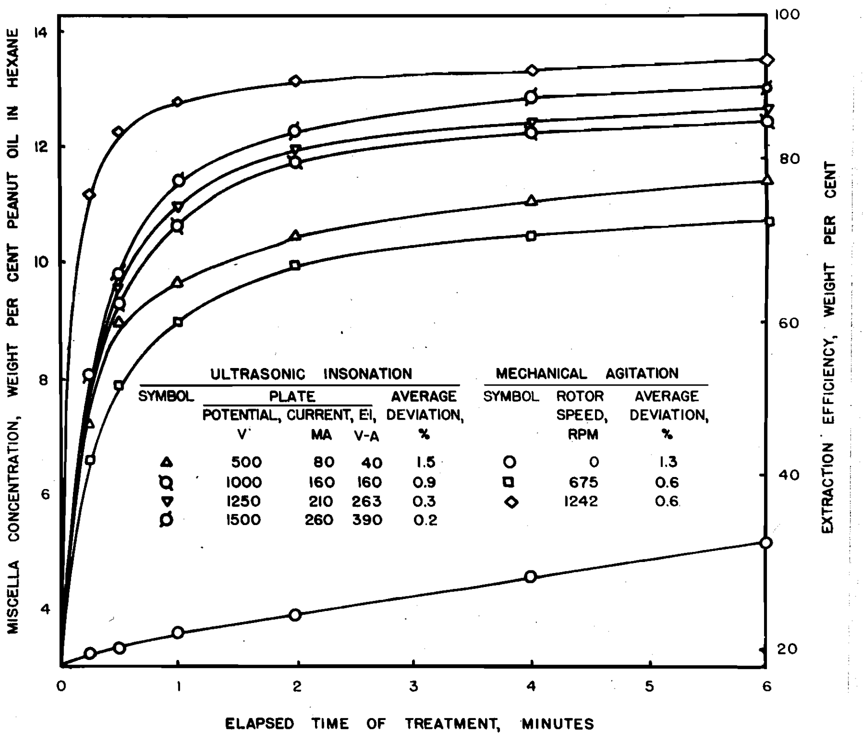
---

[a] Period of time indicated was preceded by 30-second period to allow solvent to drain from pipet and followed by 15-second period to allow transfer of miscella to specimen vial.

[b] Mechanical agitation accomplished by employing a magnetic stirrer (rotor 0.75 millimeter in diameter and 2 millimeters long) at stirrer setting of 10 (675 revolutions per minute) and 50 (1242 revolutions per minute).

[c] Computed by subtracting ambient temperature from temperature of system at conclusion of test.

[d] Computed by dividing oil yield from test by oil yield from soxhlet determination.



Extraction efficiency represents weight of peanut oil extracted during insonation or agitation divided by weight of peanut oil extracted during Soxhlet extraction.

E·I represents product of plate potential and plate current. For a power factor of one the volt-ampere (v-a) values represent power.

Average deviation represents deviation of experimental from calculated extraction values.

Figure 28. Comparison of Ultrasonic Insonation, Mechanical Agitation, and 23.0 ± 1.0 °C Control Test for Solvent Extraction of Oil from Peanuts.

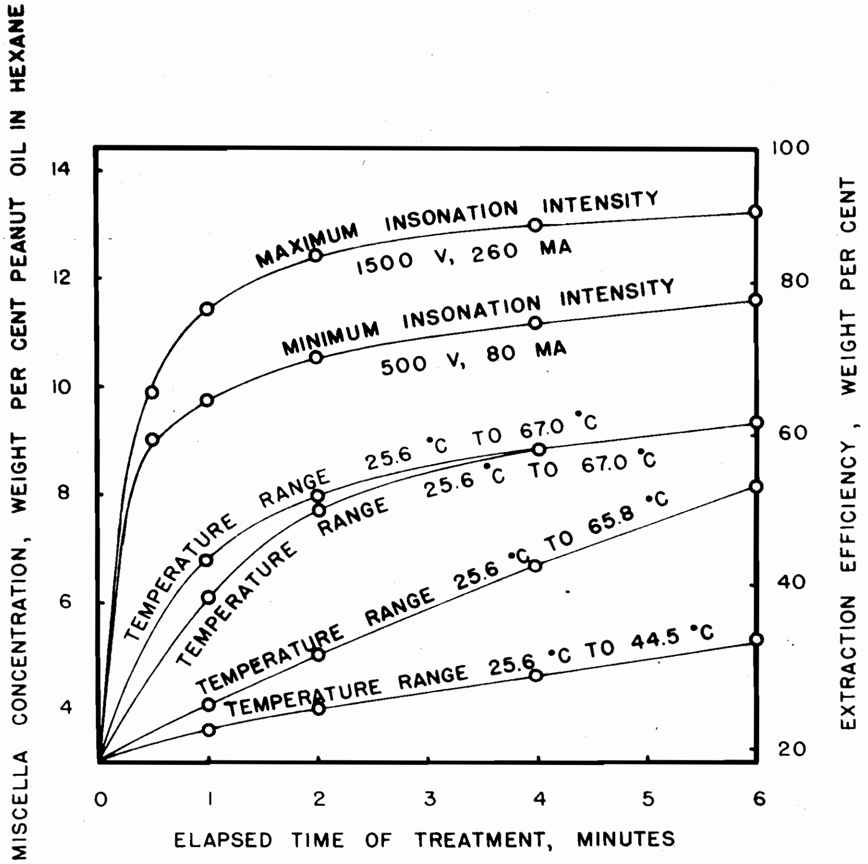
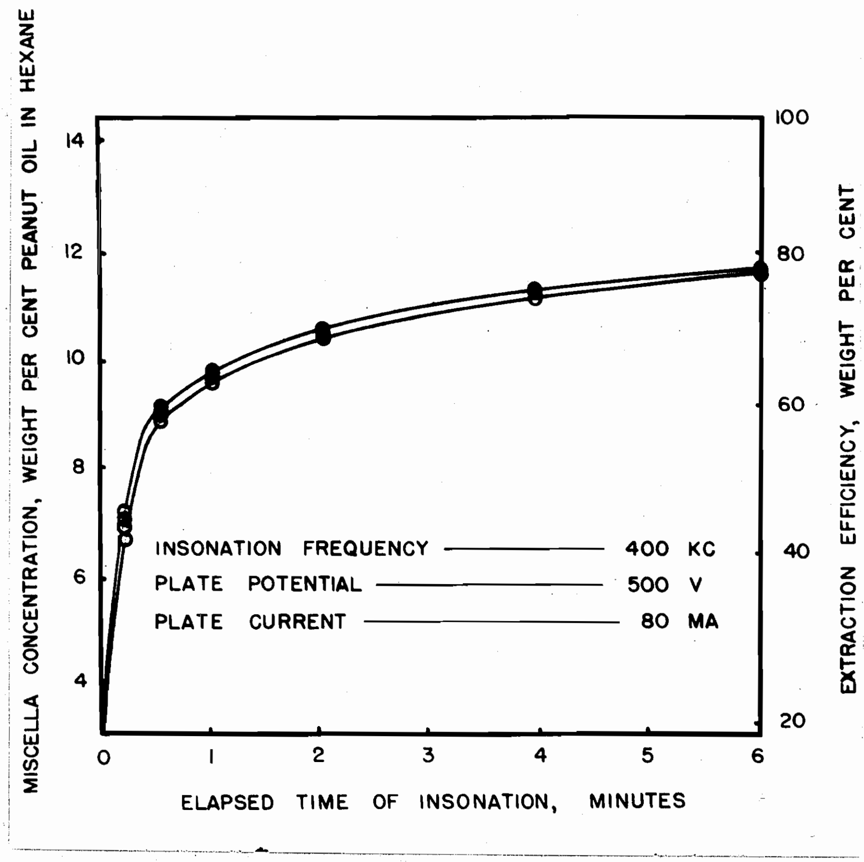


Figure 29. Comparison of Ultrasonic Insonation and Control Tests Under Similar Thermal Conditions.

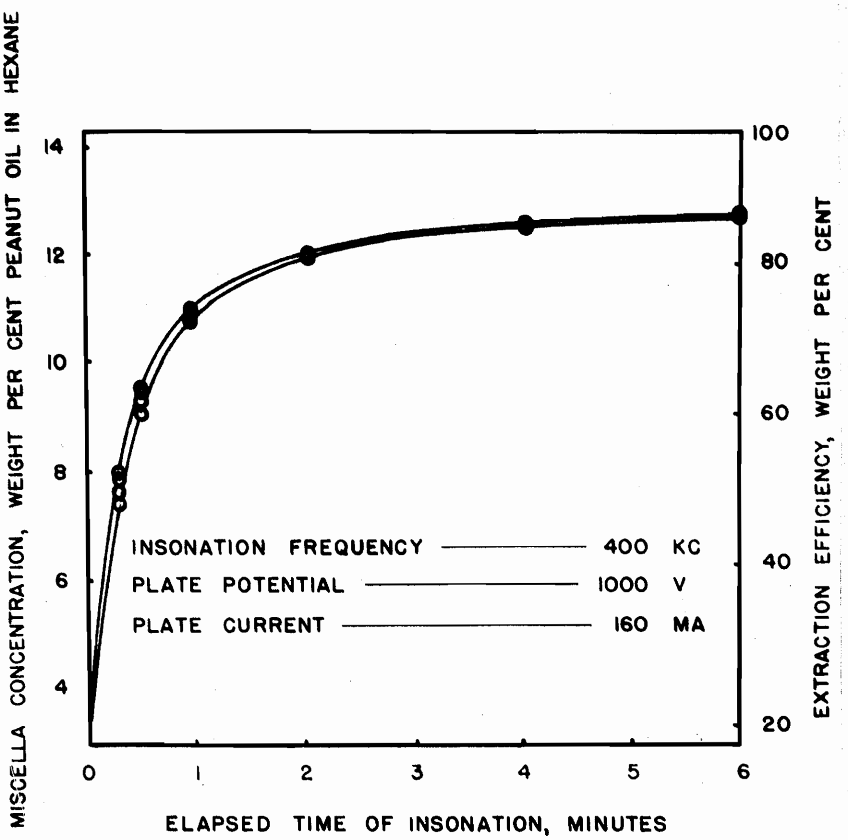
ultrasonic insonation and mechanical agitation extraction data in Figure 28, page 113.

<u>Reproducibility Tests</u>. Reproducibility data for ultrasonic insonation tests are listed in Table XII, page 109, and plotted in Figures 30, 31, 32, and 33, pages 116, 117, 118, and 119. Data for mechanical agitation and control reproducibility tests are listed in Table XIII, page 112. Reproducibility of mechanical agitation and control test data are plotted in Figure 34, page 120.



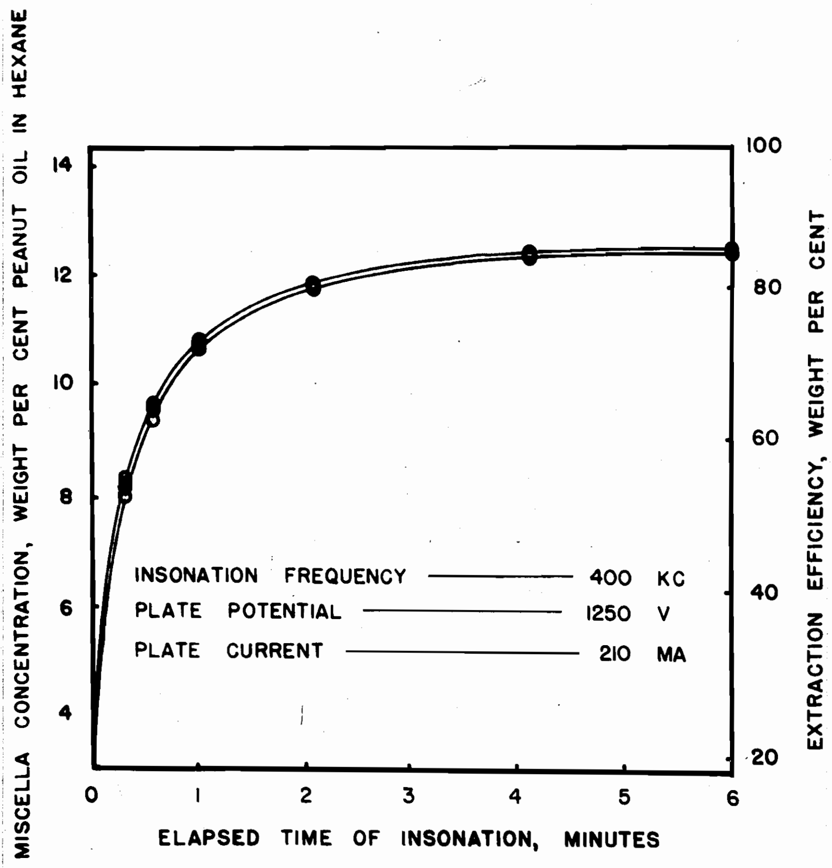
<u>Extraction efficiency</u> represents weight of peanut oil extracted during insonation divided by weight of peanut oil extracted during Soxhlet extraction.

Figure 30. Reproducibility of Peanut Oil Extraction Data for 400-Kilocycle Insonation at 500 Volts and 80 Milliamperes.



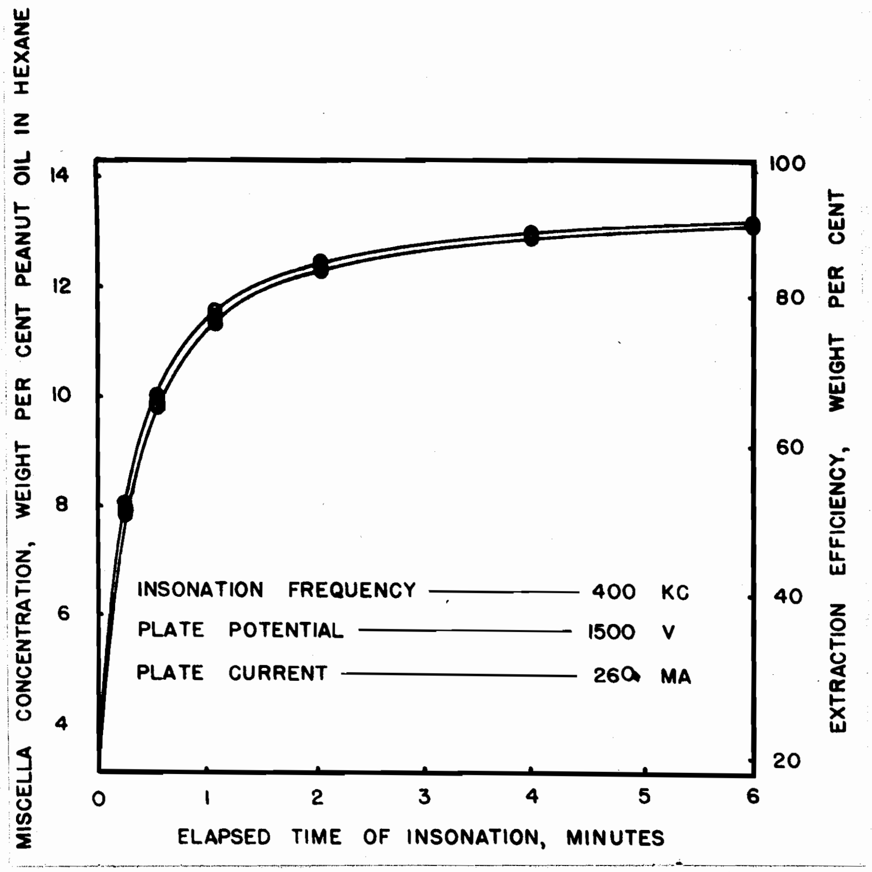
<u>Extraction efficiency</u> represents weight of peanut oil extracted during insonation divided by weight of peanut oil extracted during Soxhlet extraction.

Figure 31. Reproducibility of Peanut Oil Extraction Data for 400-Kilocycle Insonation at 1000 Volts and 160 Milliamperes.



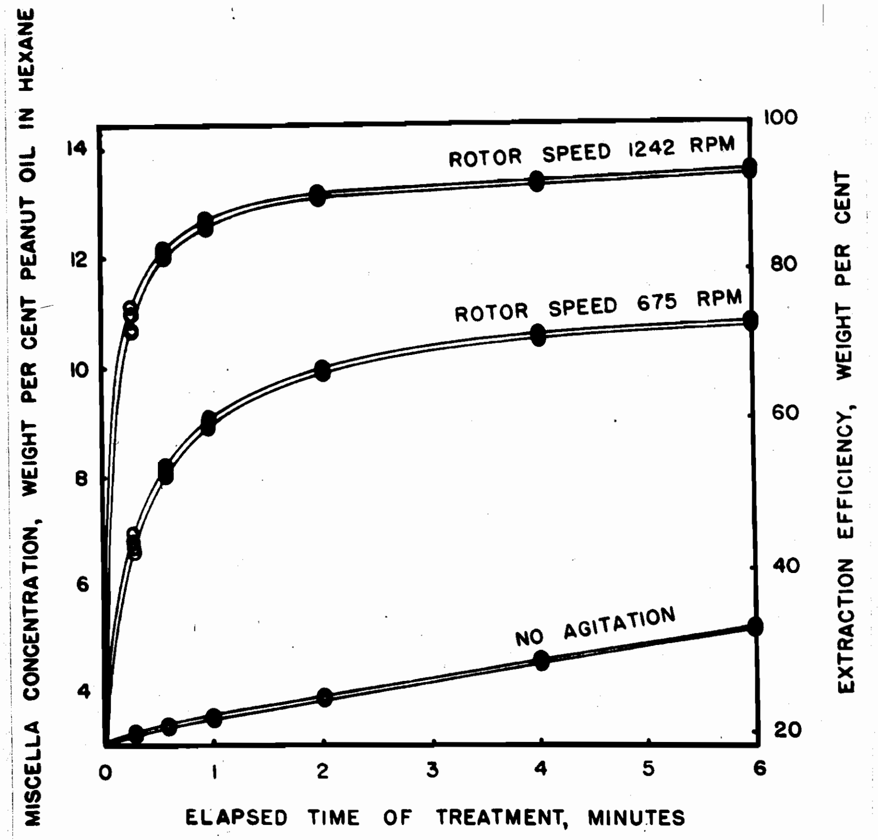
Extraction efficiency represents weight of peanut oil extracted during insonation divided by weight of peanut oil extracted during Soxhlet extraction.

Figure 32. Reproducibility of Peanut Oil Extraction Data for 400-Kilocycle Insonation at 1250 Volts and 210 Milliamperes.



<u>Extraction efficiency</u> represents weight of peanut oil extracted during insonation divided by weight of peanut oil extracted during Soxhlet extraction.

Figure 33. Reproducibility of Peanut Oil Extraction Data for 400-Kilocycle Insonation at 1500 Volts and 260 Milliamperes.



<u>Extraction efficiency</u> represents weight of peanut oil extracted during mechanical agitation divided by weight of peanut oil extracted during Soxhlet extraction.

Figure 34. Reproducibility of Peanut Oil Extraction Data for Mechanical Agitation at 23.0 ± 1.0 °C.

Sample Calculations

Sample calculations used in determination as well as interpretation of results are presented in this section.

Weight of Oil Extracted from Feed. Quantity of oil extracted from peanuts during ultrasonic insonation, mechanical agitation, and control tests was determined by specific gravity analysis of miscella at 30 °C. Specific gravity was converted to miscella concentration by use of the specific gravity versus miscella concentration relationship plotted in Figure 21, page 90. The following assumptions were made: (1) the system was composed of a single soluble solute, a solvent, and an inert carrier solid, (2) solubility of the carrier solid was negligible, (3) solute was removed solely by physical solution in the solvent, (4) concentration of miscella retained by the solid was identical to that of the miscella withdrawn, and (5) solute was nonvolatile.

Quantity of oil extracted from the peanuts was calculated by the following relationship:

$$W_o = V_s \rho_s C_e/(1 - C_e) \qquad (9)$$

where:

W_o = weight of oil extracted from peanuts, gm

V_s = volume of solvent employed, ml

ρ_s = density of solvent at 30 °C, gm/ml

C_e = concentration of extract, gm oil/gm hexane.

<u>Oil Content of Feed</u>. Oil content of peanut feeds was measured by employing Soxhlet extraction. The following assumptions were made: (1) the system was composed of a single soluble solute, a solvent, and an inert carrier solid, (2) solubility of the carrier solid was negligible, (3) solute was removed solely by physical solution in the solvent, (4) concentration of miscella retained by the solid was identical with that of the miscella withdrawn, (5) solute was non-volatile, (6) infinite stages accomplished in the Soxhlet extractor resulted in maximum extraction, and (7) quantity of hexane escaping into the room during Soxhlet extraction was negligible.

The mathematical relationship for calculating oil content of peanuts was as follows:

$$W_{op} = (W_{p1} - W_{p2})/W_{p1} \qquad (10)$$

where:

W_{op} = ratio of oil weight to total peanut weight, gm oil/gm peanut

W_{p1} = weight of peanut feed before Soxhlet extraction, gm

W_{p2} = weight of peanut feed after Soxhlet extraction, gm.

Percentage of Oil Extracted from Feed during Ultrasonic Insonation, Mechanical Agitation, and Control Tests. Percentage oil extracted from peanut feeds during ultrasonic insonation, mechanical agitation, and control tests was calculated from values of $\underline{W_o}$, Equation 9, and $\underline{W_{op}}$, Equation 10.

The following relationship was employed:

$$P_c = C\,W_o/W_{op} \tag{11}$$

where:

P_c = percentage oil extracted from peanuts, wt per cent

C = 100/5.1220, wt per cent peanut oil in hexane/gm peanuts

= 19.524

W_o = weight of oil extracted from peanuts, gm oil

W_{op} = ratio of oil weight to total peanut weight, gm oil/gm peanut.

Mathematical Relations Describing Extraction Data. Extraction curves for tests employing ultrasonic insonation and mechanical agitation were exponential when plotted on ordinary graph paper, while extraction curve for control tests was linear. The method followed in determination of mathematical relations describing extraction data is presented in this section.

Insonation and Agitation Data. Because extraction curves for ultrasonic insonation and mechanical agitation tests belonged to the same family, hyperbolic functions, identical procedure was followed in determining mathematical relations describing these curves. Data for extraction tests employing 400-kilocycle insonation at 500 volts and 80 milliamperes were arbitrarily selected for this illustration.

Data were rectified to linear relation when $\underline{T/(E - E_o)}$ versus $\underline{T}$ were plotted on Cartesian coordinates (Figure 35, page 126, and Figure 36, page 127). The equation describing the data was:

$$T/(E - E_o) = a + bT \qquad (12)$$

where:

T = elapsed time of insonation, min

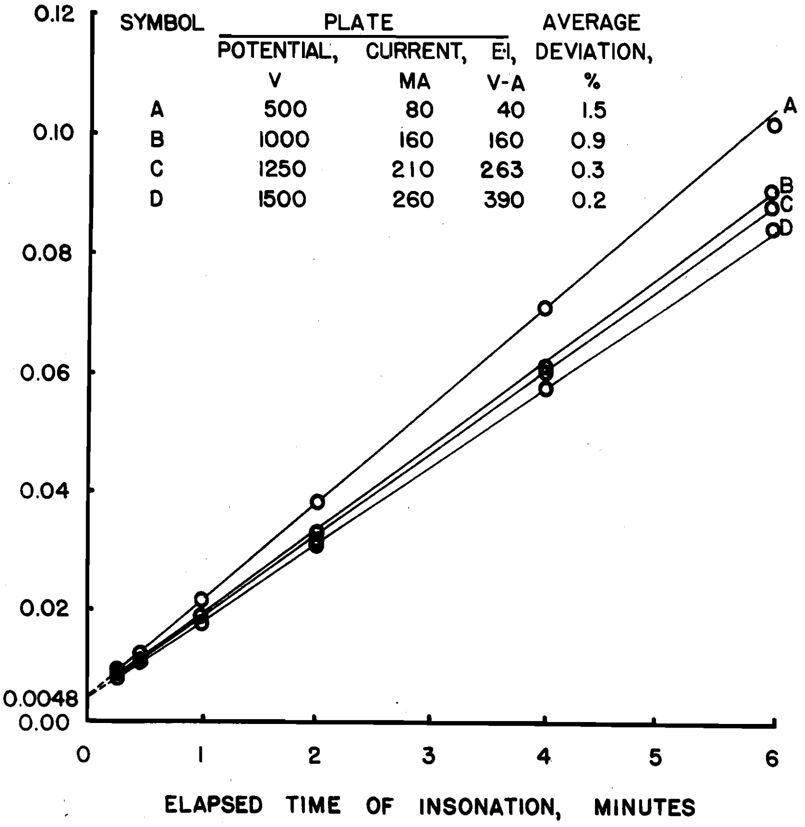
E = peanut oil extraction efficiency, wt per cent

E_o = peanut oil extraction efficiency at zero time of insonation, wt per cent

= 18.8

a = ordinate intercept after reduction to linear form, min/wt per cent

b = slope of curve after reduction to linear, 1/wt per cent.



SYMBOL	PLATE			AVERAGE
	POTENTIAL, V	CURRENT, MA	EI, V-A	DEVIATION, %
A	500	80	40	1.5
B	1000	160	160	0.9
C	1250	210	263	0.3
D	1500	260	390	0.2

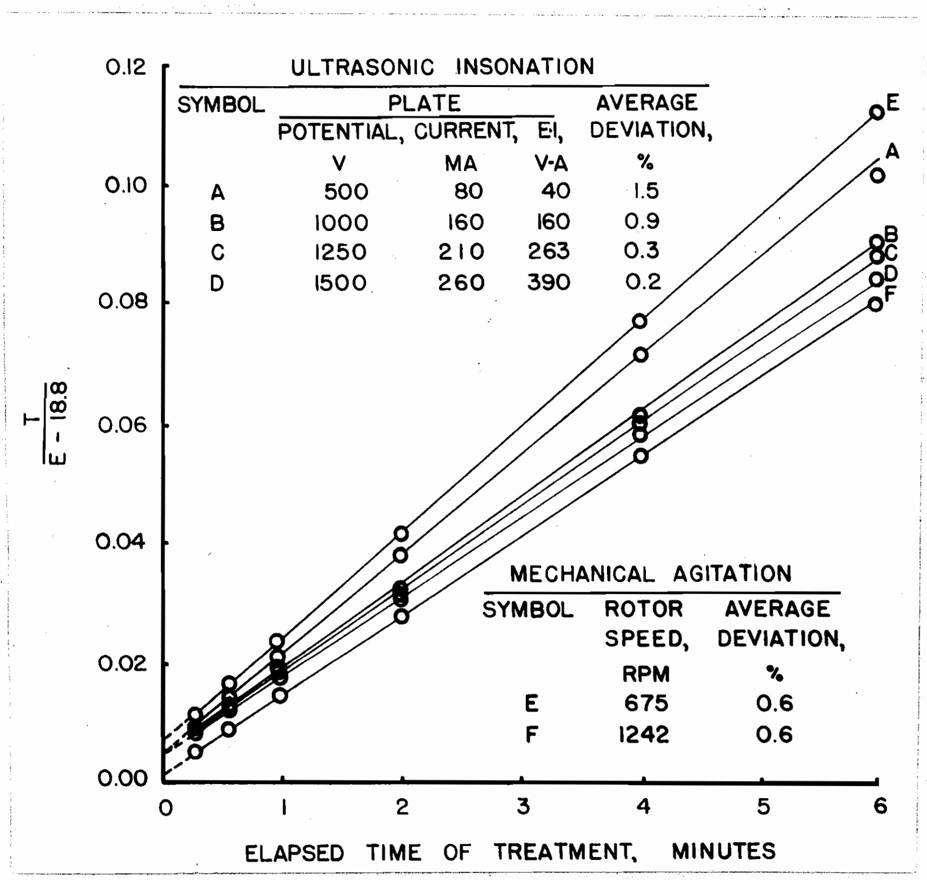
T represents elapsed time of insonation, minutes.

E represents extraction efficiency, weight peanut oil extracted during insonation tests divided by weight of peanut oil extracted during Soxhlet extraction.

E·I represents product of plate potential and plate current. For a power factor of one the volt-ampere (v-a) values represent power.

Average deviation represents deviation of experimental from calculated extraction values.

Figure 35. Rectification of Ultrasonic Insonation Extraction Data to Linear Relation.



ULTRASONIC INSONATION

SYMBOL	PLATE POTENTIAL, V	PLATE CURRENT, MA	E·I, V-A	AVERAGE DEVIATION, %
A	500	80	40	1.5
B	1000	160	160	0.9
C	1250	210	263	0.3
D	1500	260	390	0.2

MECHANICAL AGITATION

SYMBOL	ROTOR SPEED, RPM	AVERAGE DEVIATION, %
E	675	0.6
F	1242	0.6

T represents elapsed time of insonation, minutes.

E represents extraction efficiency, weight peanut oil extracted during insonation or agitation tests divided by weight peanut oil extracted during Soxhlet extraction.

E·I represents product of plate potential and plate current. For a power factor of one the volt-ampere (v-a) values represent power.

Average deviation represents deviation of experimental from calculated extraction values.

Figure 36. Rectification of Ultrasonic Insonation and Mechanical Agitation Extraction Data to Linear Relation.

Substitution of data for 1/4, 1/2, 1, 2, 4, and 6 minutes of insonation into Equation 12 yielded the following six equations:

$$0.00897 = a + b\,(1/4) \qquad (13)$$

$$0.01238 = a + b\,(1/2) \qquad (14)$$

$$0.02200 = a + b\,(1) \qquad (15)$$

$$0.03891 = a + b\,(2) \qquad (16)$$

$$0.07140 = a + b\,(4) \qquad (17)$$

$$0.10170 = a + b\,(6) \qquad (18)$$

The constants, <u>a</u> and <u>b</u>, were determined by simultaneous solution of these six equations, yielding:

$$a = 0.00489 \qquad (19)$$

and

$$b = 0.01645 \qquad (20)$$

The equation for the curve becomes

$$E = T/(0.00489 + 0.01645\,T) + 18.8 \qquad (21)$$

Equation 21 was tested by calculating values of <u>E</u> for 1/4, 1/2, 1, 2, 4, and 6 minutes of insonation and checking these values with experimental results. These data are included in Table XIV, page 129.

TABLE XIV

Relation of Experimental and Calculated Values of Peanut Oil Extraction Efficiency for Tests Employing Ultrasonic Insonation at 500 Volts Plate Potential and 80 Milliamperes Plate Current

E^a (experimental)	T^b	$E - 18.8$	$\frac{T}{E - 18.8}$	E (calculated)	Average Deviation[c]
18.8	0	0.0	-	18.8	0.0
47.1	$\frac{1}{4}$	27.9	0.00897	46.5	+ 1.3
59.3	$\frac{1}{2}$	40.4	0.01238	57.7	+ 2.7
64.1	1	45.4	0.02200	65.7	- 2.4
70.5	2	51.4	0.03891	71.7	- 1.7
74.8	4	56.1	0.07140	75.4	- 0.8
78.0	6	59.0	0.10170	76.7	+ 1.7
					1.5 av.

---

[a] Extraction efficiency, weight peanut oil extracted during insonation test divided by weight peanut oil extracted during Soxhlet extraction.

[b] Elapsed time of insonation, minutes.

[c] Deviation of experimental from calculated extraction values.

Control Data. Data from control tests were linear when plotted in graph of crude oil extraction efficiency versus elapsed time of insonation; therefore, the equation describing the data was:

$$E = a + b\,T \qquad (22)$$

where:

E = crude oil extraction efficiency, wt per cent

a = ordinate intercept, wt per cent

b = slope of curve, wt per cent/min

T = elapsed time of insonation, min.

Constants were calculated by simultaneous solution of equations, as in calculation of constants for insonation and agitation tests. The equation for the curve was thus:

$$E = 19.7 + 2.205\,T \qquad (23)$$

Deviation of Equation 23 was determined as for insonation and agitation tests, and these data are included in Table XV, page 131.

TABLE XV

Relation of Experimental and Calculated Values of Peanut Oil Extraction Efficiency for Control Tests at 23.0 ± 1.0 °C

E[a] (experimental)	T[b]	E[a] (calculated)	Average Deviation[c]
18.8	0	19.7	4.6
19.9	$\frac{1}{4}$	20.2	1.4
20.6	$\frac{1}{2}$	20.8	0.9
22.2	1	21.9	1.4
24.1	2	24.1	0.0
28.4	4	28.5	0.4
32.7	6	32.9	0.6
			1.3 av.

[a] Extraction efficiency, weight peanut oil extracted during control test divided by weight peanut oil extracted during Soxhlet extraction.

[b] Elapsed time of treatment, minutes.

[c] Deviation of experimental from calculated extraction values.

IV. DISCUSSION

Analysis and discussion of results are included in this section of the thesis. Recommendations for future work and a summary of the limitations imposed on the investigation are also included.

Discussion of Results

Experimental variables investigated in different series of extraction tests were as follows: (1) time of insonation, (2) intensity of insonation, (3) time of mechanical agitation, (4) intensity of mechanical agitation, (5) time of control sample, and (6) temperature of control sample. These variables and experimental results are discussed in this section.

Effect of Time of Insonation on Extraction. Quantity of extracted peanut oil increased as time of insonation was increased. However, the greatest percentage of oil in the peanut feed was extracted during the first 30 seconds of insonation. Rate of extraction gradually decreased with increasing time of insonation, as shown in Figure 26, page 110. Extraction rate decrease with time was credited primarily to the decrease in quantity of oil available for extraction. A second reason for the decrease in extraction rate with time was the increase of oil in the hexane, which decreased the peanut oil concentration gradient between the peanut meat and miscella(6,25). For example, consider the data for tests employing maximum insonation (1500 volts, 260 milliamperes). After the first minute of insonation, 77.0 weight per cent of the oil originally in the peanut feed had been extracted. Only 23.0 per cent of the original peanut oil remained for extraction during the final five minutes of insonation. After six-minute insonation, 90.2 per cent of the peanut oil present in the peanuts before insonation had been extracted. Complete extraction of oil from the peanut feeds was not possible under the experimental conditions of the extraction tests, because of oil in the portion of miscella wetting the peanuts. Theoretical limit to peanut oil extraction was 98.7 weight per cent.

Peanut feeds contained 2.8427 grams of oil. Quantity of peanut oil extracted during the first minute of maximum insonation (1500 volts, 260 milliamperes) was 2.1889 grams. Peanut oil quantities extracted during the second, third, fourth, fifth, and sixth minutes of insonation were 0.2075, 0.0824, 0.0370, 0.0256, and 0.0227 gram, respectively. Raffinate contained 0.2786 gram of peanut oil. Extraction curves for tests employing mechanical agitation (Figure 27, page 111) and for tests employing thermal conditions similar to those observed in insonation tests (Figure 29, page 114) also exhibited decreasing rate of peanut oil extraction with increasing time of treatment.

Effect of Intensity of Insonation on Extraction. Peanut oil extraction increased as intensity of insonation was increased, as shown in Figure 26, page 110. Insonation at maximum intensity (1500 volts, 260 milliamperes) for six minutes produced extraction of 90.2 per cent of the oil in the peanut feed. Six-minute insonation at 500 volts and 80 milliamperes (minimum intensity), 1000 volts and 160 milliamperes, and 1250 volts and 210 milliamperes produced peanut oil extractions of 78.0, 85.2, and 86.9 per cent, respectively.

Insonation at minimum intensity (500 volts, 80 milliamperes) was found to produce slightly lower peanut oil extraction values than expected, considering extraction data for the three greater levels of insonation intensity. High intensities that produced the disruptive action of cavitation near the free surface of miscella were accompanied by fog formation. Mass transfer of the oil from the inert solids to the solvent was facilitated by the selective micro-agitation at the peanut-miscella interface. Absorption of ultrasound increased proportionally as interfacial area was increased(3,105). Solid-liquid and liquid-gas interfaces were present in the system during, and increased by, insonation. Increased solid-liquid interface was provided by dispersion of peanut particles in the miscella during insonation. Increased liquid-gas interface was provided by formation of cavities and degassing of the liquid in the miscella. Cavities formed and collapsed at the rate of 400,000 times per second. Fog formation at the free surface of the miscella (cavitation) was not present during insonation at minimum intensity (500 volts, 80 milliamperes). An acoustic intensity of approximately 0.3 watt per square centimeter was necessary before cavitation occurred. Absence of cavitation at the miscella free surface indicated that the peanut meat-peanut oil-hexane system was not exposed to the disruptive action of cavitation throughout its entire mass. Cavitation could have occurred beneath but not at the interface, during low intensity (500 volts, 80 milliamperes) insonation.

The curve representing intensity of insonation with limited cavitation (500 volts, 80 milliamperes) was proportionally lower than the position of other extraction curves. Interfacial agitation increased the rate of extraction by aiding the replacement of peanut oil-rich miscella with solvent-rich miscella. Gross turbulence, or mixing, of the system during insonation accomplished a degree of interfacial agitation. It was inferred though not demonstrated directly that selective interfacial agitation produced by the high frequency compressional waves was a greater aid to extraction than agitation resulting from gross turbulence.

Comparison of Ultrasonic Insonation, Mechanical Agitation and Thermal Extraction. Higher extraction values were produced by ultrasonic insonation at each level of intensity of insonation investigated than by control extraction under similar thermal conditions (Figure 29, page 114). Quantity of peanut oil extracted after six minutes of insonation at minimum intensity (500 volts, 80 milliamperes) was 2.3142 times greater than control extraction under similar thermal conditions (25.6 °C to 44.5 °C). Six-minute insonation at maximum intensity (1500 volts, 260 milliamperes) produced extraction 1.4637 times greater than the corresponding thermal control (25.6 °C to 67.0 °C). Depletion of peanut oil in the feed and decrease of concentration gradient of oil between peanut meat and miscella were responsible for decreasing extraction with increasing intensity.

Six-minute extraction employing maximum-mechanical agitation (1242 revolutions per minute) was slightly greater, 1.042 times, than extraction employing maximum-intensity insonation (1500 volts, 260 milliamperes). Each of the four insonation intensities produced extraction values greater than were produced by mechanical agitation at 675 revolutions per minute. Data for insonation and mechanical agitation extraction tests are plotted together with data for control extraction at 23.0 ± 1.0 °C in Figure 27, page 111.

Power Consumption During Ultrasonic Insonation, Mechanical Agitation, and Thermal Control Tests. The product of plate potential and plate current was taken as a criterion of energy input to the insonation extractor. Loss of radio frequency energy, loss of energy in the transducer and in the extractor, loss of energy in the media surrounding the extractor, and absorption of energy in the peanut meat-peanut oil-hexane system, all contribute to the total power consumption required for insonation. The ratio of electrical energy generated by the ultrasonorator to acoustical energy received by the peanut meat-peanut oil-hexane system may have been in the order of ten to one or possibly greater. Under the foregoing assumption, product of plate potential and plate current indicated that 39.0 watts of acoustical energy were delivered at maximum intensity of insonation (1/10)(1500 volts, 260 milliamperes), as shown in Table XVI, page 139. Four watts were delivered at minimum intensity of insonation (1/10)(500 volts, 80 milliamperes). The product of plate potential and plate current divided by the upper effective area of the transducing crystal (5.06 square centimeters) was taken as the power density. During low intensity insonation (500 volts, 80 milliamperes), power density was 0.78 watt per square centimeter. Acoustic intensity at the free surface of miscella was less than 0.3 watt per square centimeter.

Maximum power requirement during mechanical agitation was 10.7 watts at 1242 revolutions per minute. Maximum mechanical agitation power consumption was 2.68 times the minimum power required for insonation, and 27.5 per cent

TABLE XVI

Power Consumption for Ultrasonic Insonation, Mechanical Agitation, and Thermal Control Tests

Treatment	Power, watts	Oil Extracted Per Unit Power[a], gm/watt
Ultrasonic Insonation[b]:		
500 v, 80 ma	4.0	0.547
1000 v, 160 ma	16.0	0.151
1250 v, 210 ma	26.3	0.094
1500 v, 260 ma	39.0	0.066
Mechanical Agitation[c]:		
675 rpm	9.0	0.229
1242 rpm	10.7	0.290
Thermal Control[d]:		
25.6 °C to 44.5 °C	2.3	0.420
25.6 °C to 65.8 °C	4.3	0.350
25.6 °C to 67.0 °C	4.5	0.385
25.6 °C to 67.0 °C	4.5	0.385

[a] Extraction values after six minutes of treatment were employed. Quantity of peanut oil extracted increased as time of treatment was increased.

[b] Power of insonation expressed as watts was read as volt-amperes (v-a). Power factor of one was assumed. One-tenth of the indicated electrical power produced was assumed to be converted into acoustical energy that entered the extraction mass.

[c] Watt-meter readings at input to agitator motor were employed as energy utilized for mechanical agitation.

[d] Control extraction tests represent similar thermal conditions to those encountered during insonation.

of the maximum power required for insonation. Minimum mechanical agitation requirement was 9.0 watts at 675 revolutions per minute. Maximum thermal control power consumption was 1.12 times the minimum power required for insonation, and 11.5 per cent of the maximum power required for insonation. Minimum power requirement during thermal control tests was 2.3 watts.

Theoretically, quantity of peanut oil extracted per unit of thermal energy applied to the peanut meat-peanut oil-hexane system decreases with increasing applied thermal energy. However, extraction values corresponding to insonation at 1250 volts and 210 milliamperes (0.3907 gram per watt) and 1500 volts and 260 milliamperes (0.3907 gram per watt) were greater than at 1000 volts and 160 milliamperes (0.3437 gram per watt). Miscella boiled (67.0 °C) during thermal control tests corresponding to insonation at 1250 volts, 210 milliamperes and 1500 volts, 260 milliamperes. Boiling of miscella caused a slight degree of mechanical agitation which provided greater solvent-feed contact than possible with no agitation. Also, values of extraction for controls corresponding thermally to 1250 volts, 210 milliamperes and 1500 volts, 260 milliamperes were identical

(0.3907 gram per watt). Identical values resulted because controls were conducted at the same temperature (67.0 °C).

Recommendations

Investigation of the effects of 400-kilocycle insonation on solvent extraction of peanut oil suggested a number of questions to which no answer was provided, indicating possibilities for future investigations. Specific recommendations for future investigations are presented in this section.

Measurement of Ultrasonic Intensity. Ultrasonic intensity at various points within the extractor should be measured directly, either by using a probe incorporating a calibrated piezoelectric crystal or by using a probe incorporating a thermocouple. In the case of the former, the probe would give a direct indication of absolute pressure amplitude; in the case of the latter, the probe would give an indication of acoustical energy absorbed and converted to thermal energy.

Localized Mechanical and Thermal Effects. The extent to which localized mechanical and thermal effects at the solvent-feed interface aid extraction should be investigated. A comprehensive investigation of localized mechanical and thermal effects produced by insonation will require development of instrumentation.

Frequency of Insonation. Solvent extraction of oil from peanuts should be investigated employing insonation at low ultrasonic frequencies (30 to 40 kilocycles). Greater amplitude of vibration can be produced employing insonation frequencies of 30 to 40 kilocycles than at the 400-kilocycle frequency level employed in this investigation.

Attenuation. Attenuation of ultrasonic energy in the peanut meat-peanut oil-hexane system for various intensities and times of 400-kilocycle insonation should be determined. Attenuation should be experimentally determined within the system for various concentrations.

Extraction Employing Mechanical and Thermal Energy. It is recommended that mechanical agitation extraction tests be conducted at thermal conditions similar to those encountered during ultrasonic insonation.

Insonation Extractor. Although not of major concern, a study should be made to determine the effect of various supports and clamping assemblies for the insonation extractor on peanut oil extraction. The optimum diameter of the insonation extractor should also be determined.

Limitations

Limitations of the specific fields of work and the quantitative boundaries of the variables investigated are presented in this section.

System. The investigation was limited to the system peanut meat-peanut oil-hexane.

Quantity of Material Insonated. Peanut feeds for extraction tests weighed 5.1220 grams. A solvent-feed ratio of 3.3:1 was employed.

Size of Peanut Particles in Feed. Size of peanut particles in feed was 0.0336 to 0.0474-inch maximum dimension.

Intensity of Insonation. Measurement of compressional wave intensity was restricted to measurement of ultrasonic generator plate potential and plate current. Plate potentials and plate currents of 500 volts and 80 milliamperes (minimum intensity), 1000 volts and 160 milliamperes, 1250 volts and 210 milliamperes, and 1500 volts and 260 milliamperes (maximum intensity) were an indication of ultrasonic intensities investigated.

Time of Insonation. Insonation periods of 0, 1/4, 1/2, 1, 2, 4, and 6 minutes were employed during conduct of this investigation.

Frequency of Insonation. Frequency of insonation was restricted to 400 kilocycles per second.

Reproducibility of Data. Minimum deviation of experimental extraction data from calculated extraction data was 0.2 per cent, and maximum deviation was 1.5 per cent. Arithmetical average of deviation for extraction tests was 0.8 per cent.

V. CONCLUSIONS

Solvent extraction of oil from peanut particles, (0.0336 to 0.0474-inch), employing n-hexane as the solvent, peanut feeds weighing 5.1220 grams, a solvent-feed ratio of 3.3:1, and extraction periods varying from zero to six minutes, led to the following conclusions.

A. Four-hundred kilocycles compressional waves were generated by a Model SL520 Ultra-Sonorator.

1. At 500 volts plate potential and 80 milliamperes plate current (minimum intensity), peanut oil extraction efficiencies for 0, 1/4, 1/2, 1, 2, 4, and 6 minutes of insonation were 18.8, 47.4, 59.2, 64.2, 70.1, 74.7, and 77.7, respectively.

2. At 1000 volts plate potential and 160 milliamperes plate current, peanut oil extraction efficiencies for 0, 1/4, 1/2, 1, 2, 4, and 6 minutes of insonation were 18.8, 48.5, 61.8, 71.5, 79.9, 83.9, and 85.2, respectively.

3. At 1250 volts plate potential and 210 milliamperes plate current, peanut oil extraction efficiencies for 0, 1/4, 1/2, 1, 2, 4, and 6 minutes of insonation were 18.8, 50.6, 63.5, 73.9, 81.8, 85.4, and 86.9, respectively.

4. At 1500 volts plate potential and 260 milliamperes plate current (maximum intensity), peanut oil extraction efficiencies for 0, 1/4, 1/2, 1, 2, 4, and 6 minutes of insonation were 18.8, 52.5, 65.5, 77.0, 84.3, 88.5, and 90.1, respectively.

B. Thermal energy during control tests was provided by immersing extractor in a heated water bath. Temperature of the water bath was that necessary to produce rates of heating similar to insonation tests.

1. Under thermal conditions varying from 25.6 °C to 44.5 °C in six minutes (similar to 500 volt, 80 milliampere insonation), peanut oil extraction efficiencies for 0, 1/4, 1/2, 1, 2, 4, and 6 minutes of treatment were 18.8, 20.0, 21.1, 22.6, 24.9, 28.7, and 33.3, respectively.

2. Under thermal conditions varying from 25.6 °C to 65.8 °C in six minutes (similar to 1000 volt, 160 milliampere insonation), peanut oil extraction efficiencies for 0, 1/4, 1/2, 1, 2, 4, and 6 minutes of treatment were 18.8, 20.7, 22.4, 25.6, 31.6, 42.7, and 52.7, respectively.

3. Under thermal conditions varying from 25.6 °C to 67.0 °C in six minutes (similar to 1250 volt, 210 milliampere insonation), peanut oil extraction efficiencies for

0, 1/4, 1/2, 1, 2, 4, and 6 minutes of treatment were 18.8, 24.6, 29.7, 38.7, 50.1, 58.1, and 61.4, respectively.

4. Under thermal conditions varying from 25.6 °C to 67.0 °C in six minutes (similar to 1500 volt, 260 milliampere insonation), peanut oil extraction efficiencies for 0, 1/4, 1/2, 1, 2, 4, and 6 minutes of treatment were 18.8, 28.0, 34.7, 43.5, 51.9, 58.1, and 61.4, respectively.

C. Mechanical agitation was provided by a variable-speed magnetic stirrer.

1. At 675 revolutions per minute (minimum intensity), peanut oil extraction efficiencies for 0, 1/4, 1/2, 1, 2, 4, and 6 minutes of agitation were 18.8, 42.3, 51.2, 59.2, 66.3, 70.6, and 72.5, respectively.

2. At 1242 revolutions per minute (maximum intensity), peanut oil extraction efficiencies for 0, 1/4, 1/2, 1, 2, 4, and 6 minutes of agitation were 18.8, 75.4, 83.9, 87.7, 90.9, 92.5, and 94.1, respectively.

D. Extraction efficiency for six-minute ultrasonic insonation at 500 volts and 80 milliamperes (minimum intensity), 1000 volts and 160 milliamperes, 1250 volts and 210 milliamperes, and 1500 volts and 260 milliamperes (maximum intensity) were 2.29, 1.60, 1.43, 1.48 times greater than for six-minute control extraction under similar thermal conditions.

E. Extraction efficiency for six-minute mechanical agitation at 1242 revolutions per minute was slightly greater, 1.042 times, than for six-minute ultrasonic insonation at maximum intensity (1500 volts, 260 milliamperes).

VI. SUMMARY

A summary of the investigation, the effects of intensity and time of 400-kilocycle insonation on solvent extraction of oil from peanuts, is given in the following paragraphs.

Purpose of Investigation. Various methods of accomplishing intimate solvent-feed contact in solvent extraction operations have been investigated. This investigation employed ultrasonic insonation in the extraction process. Selective micro-agitation at interfacial areas has been reported, but the application of ultrasonic insonation to mass transfer operations is not thoroughly understood. The purpose of this investigation was to determine the effects of intensity and time of 400-kilocycle insonation on solvent extraction of oil from peanuts.

Plan of Experimentation. Oil was extracted from 0.0336 to 0.0474-inch peanut particles using n-hexane as the solvent. Peanut feeds weighed 5.1220 grams, and a solvent feed ratio of 3.3:1 was employed. Insonation extraction tests and control extraction tests under similar thermal conditions were conducted. Extraction tests were also conducted employing mechanical agitation. Variables investigated were: time of insonation, intensity of insonation, time of mechanical agitation, intensity of mechanical agitation, time of control sample, and temperature of control sample.

Results and Conclusions. Rate of peanut oil extraction was increased by employment of ultrasonic insonation. Quantity of peanut oil extracted after six minutes of insonation at maximum intensity (1500 volts, 260 milliamperes) was 2.709 times greater than six-minute control extraction at 23.0 ± 1.0 °C. Control extraction under thermal conditions similar to those encountered during 1500 volt, 260 milliampere insonation (25.6 °C to 67.0 °C) was 1.850 times greater than control extraction at 23.0 ± 1.0 °C. Quantity of peanut oil extracted employing maximum-intensity mechanical agitation (1242 revolutions per minute) was slightly greater, 1.042 times, than extraction employing maximum intensity insonation (1500 volts, 260 milliamperes). Bulk agitation and selective micro-agitation at the solvent-feed interface aided extraction by replacing peanut oil-rich miscella surrounding peanut particles with hexane-rich miscella.

VII. BIBLIOGRAPHY

1. Algar, W. H. and H. W. Giertz: Fiber Structure and Pulp Properties. II. The Influence of Ultrasonic Treatment on Wood Cellulose Fibers, Svensk Papperstidning, 54, 693-700 (1951).

2. Allis-Chalmers Plant Flow Sheet, Soybean Oil Extraction with n-Hexane, Chem. Met. Eng., 48, 128-31 (1941).

3. Altberg, W. and M. Holtzman: Physik. Z., 26, 149 (1925). Richardson, E. G.: "Ultrasonic Physics," pp. 256-7. Elsevier Publishing Co., Houston, Texas, 1952. 1 ed.

4. Ament, W. S.: Sound Propogation in Gross Mixtures, J. Acoust. Soc. Am., 25, 638-41 (1953).

5. Austerweil, M. G. V.: L'auxiliaire des Chemins de fer et de O'industrie, Fr. Pat. 841,359 (May 17, 1939).

6. Badger, W. L. and W. L. McCabe: "Elements of Chemical Engineering," pp. 4-6. McGraw-Hill Book Co., Inc., New York, N. Y., 1936. 2 ed.

7. ibid, pp. 428-34.

8. ibid, pp. 434-36.

9. Baker, E. M.: Simplifying the Calculation of Countercurrent Extractions, Chem. Met. Eng., 42, 669-71 (1935).

10. Bazhulin, P. A.: Absorption of Ultrasonic Waves in Liquids, Sc. A., <u>55</u>, 600 (1952); Akademii Nauk S S S R, 5, 261-338 (1950).

11. Bergmann, L. and H. S. Hatfield: "Ultrasonics and their Scientific and Technical Applications," pp. 128-36. John Wiley and Sons, Inc., New York, N. Y., 1938. 1 ed.

12. ibid, pp. 151-60.

13. ibid, pp. 202-16.

14. Bilbe, C. W.: Continuous Solvent Extraction of Vegetable Oils, Mech. Eng., <u>63</u>, 357-60 (1941).

15. Biquard, P.: Absorption of Ultrasonic Waves by Liquids, Ann. Phys., <u>6</u>, 195-304 (1936).

16. __________: Etude sur la vitesse de propogation et sur l'absorption du son dans les liquides sons pression, Rev. d'Acoust., <u>8</u>, 130 (1939).

17. __________: Sur l'absorption des ondes ultra-sonores par les liquides, Theses de Doctorat, Paris (1935); Vigoureux, P.: "Ultrasonics," pp. 73-7. John Wiley and Sons, Inc., New York, N. Y., 1951. 1 ed.

18. Bordoni, P. G.: Absorption of Ultrasound in Solids, Nuovo Cimento, <u>7</u>, 144-60 (1950).

19. Boutaric, A. and J. Bouchard: Action of Agitation and of Ultrasonic Waves on the Speed of Flocculation of Unstable Colloids, C. A., <u>26</u>, 4225 (1932); Bull. Soc. Chim., <u>51</u>, 543-9 (1932).

20. Boyd, C. A. and R. J. Zentner: Experiments on the Ultrasonic Unmixing of Liquid Solutions, Phys. Rev., <u>83</u>, 1059 (1951).

21. Brandt, O., H. Freund, and E. Hiedemann: Suspended Particles in a Sound Field, Z. Physik, <u>104</u>, 511-33 (1937).

22. Brown, G. G., et al: "Unit Operations," p. 277. John Wiley and Sons, Inc., New York, N. Y., 1950. 1 ed.

23. ibid, pp. 277-81.

24. ibid, p. 510.

25. ibid, pp. 510-1.

26. ibid, pp. 514-6.

27. ibid, p. 528.

28. Bull, F. W. and G. J. Coli: Graphical Methods as Applied to Extraction Problems, Va. Poly. Inst., Blacksburg, Va., Eng. Expt. Sta., Bull., No 72, 10 (1950).

29. Bull, F. W. and G. J. Coli: Graphical Methods as Applied to Extraction Problems, Va. Poly. Inst., Blacksburg, Va., Eng. Expt. Sta., Bull., No 72, 10-3 (1950).

30. ibid, pp. 45-9.

31. Burton, C. J.: Ultrasonic Velocity and Absorption in Liquid Mixtures, J. Acoust. Soc. Am., 20, 186-99 (1948).

32. Colburn, A. P.: Simplified Calculations of Diffusional Processes, Ind. Eng. Chem., 33, 459-67 (1941).

33. ______________ and R. L. Pigford: "Chemical Engineers' Handbook," (J. H. Perry, Editor), pp. 549-50. McGraw-Hill Book Co., Inc., New York, N. Y., 1950. 3 ed.

34. Cypers, J. F., G. Scheu, and W. T. Jones: Continuous Solvent Extraction Apparatus, U. S. Pat. 2,278,647 (Apr. 7, 1942).

35. Decossas, K. M., F. A. Deckbar, Jr., and J. L. Hecker: Densities and Gravities of Cottonseed and Peanut Oil Miscellas in English Units, South. Reg. Research Lab., New Orleans, La., Bull. AIC-304, 4 (1950).

36. Decossas, K. M., F. A. Deckbar, Jr., and J. L. Hecker: Viscosities of Cottonseed and Peanut Oil-Hexane Miscelles in English Units, South. Reg. Research Lab., New Orleans, La., Bull. AIC-292, 4 (1950).

37. Eaves, P. H., L. J. Molaison, C. L. Black, A. J. Crovetto, and E. L. D'Aquin: A Comparison of Five Commercial Solvents for Extraction of Cottonseed, J. Am. Oil Chemists' Soc., XXIX, 88-94 (1952).

38. Elgin, J. C.: Graphical Calculations of Leaching Operations, Trans. Am. Inst. Chem. Engrs., 32, 451-71 (1936).

39. ____________ and R. Wynkoop: Solvent Extraction, "Chemical Engineers' Handbook," (J. H. Perry, Editor), pp. 716-8. McGraw-Hill Book Co., Inc., New York, N. Y., 1950. 3 ed.

40. ibid, pp. 737-9.

41. Evans, T. W.: Countercurrent and Multiple Extraction, Ind. Eng. Chem., 26, 860-4 (1936).

42. Fox, F. E.: Dependence of Ultrasonic Absorption on Intensity and the Phenomenon of Cavitation, Nuovo Cimento, 7, 198-203 (1950).

43. Fox, F. E. and G. D. Rock: Compressional Viscosity and Sound Absorption in Water at Different Temperatures, Phys. Rev., 70, 68-73 (1946).

44. Gold, L.: Ultrasonic Wave Propogation in Materials: Guide to Theoretical Results, American Society for Testing Materials, Symposium Ultrasonic Testing, 3-13 (1951).

45. Goss, W. H.: Modern Practice in Solvent Extraction, Chem. Met. Eng., 48, 80-3 (1941).

46. ____________: Processing Oil Seeds and Oils in Germany, Oil and Soap, 23, 241-4 (1946).

47. Gralen, N. and S. Berg: Treatment of Wood with Ultrasonic Waves, J. Polymer Sci., 6, 503-7 (1951).

48. Grove, H. D., Jr.: A New Magneto-Striction Oscillator - Construction and Observation of some Chemical and Physical Phenomena, pp. 42-3. Unpublished M. Sc. Thesis, Library, State Univ. of Iowa, Iowa City, Iowa (1948).

49. Hartmann, G. K. and A. B. Focke: Absorption of Supersonic Waves in Water and in Aqueous Suspensions, Phys. Rev., 57, 221-5 (1940).

50. Hawley, L. F.: Numerical Relation Between Cells and Treatments in Extraction Processes, Ind. Eng. Chem., 12, 493-6 (1920).

51. Hazzard, G. W.: Ultrasonic Absorption in Liquids, J. Acoust. Soc. Am., 22, 29-32 (1950).

52. Heitmann, H.: Apparatus for the Continuous Extraction of Material, U. S. Pat. 2,043,409 (June 9, 1936).

53. Herzfeld, K. F.: Origin of the Ultrasonic Absorption in Liquids. II., J. Chem. Phys., 20, 288-9 (1952).

54. Hildebrandt, K.: The Continuous Extraction of Seeds and the Equipment for its Accomplishment, Fette u. Seifen, 46, 350-2 (1939).

55. Holtol, G. J.: Propogation of Sound in Liquids Under High Pressure, Phys. Rev., 73, 543 (1948).

56. Horvath, A. A.: "The Soybean Industry," pp. 42-58. Chemical Publishing Co. of New York, Inc., 1939. 2 ed.

57. Hougen, O. A.: "Teaching of Chemical Engineering," chapter on drying, Proc. Chem. Eng. Div., Soc. Prom. Eng. Ed., 2nd Summer School in Chem. Eng., June, 1939.

58. Hunter, T. G. and A. W. Nash: Liquid-Liquid Extraction Systems, Ind. Eng. Chem., 27, 836-45 (1935).

59. Janssen, W. H.: Equipment for Generating Ultrasonic Energy, Chem. Eng. Prog., 46, 541 (1950).

60. Kinney, G. F.: Leaching Calculations, Ind. Eng. Chem., 34, 1102-4 (1942).

61. Kirchhoff, G.: Uber den Einfluss der Warmeleitung in einem Gase auf die Schallbewegung, Pogg. Ann., 134, 177 (1868).

62. Krause, R.: Measurement of Ultrasonic Fields in Fluids, Phys. A., 54, 20 (1951); Zeitschrift angewandte fuer Physik, 2, 370-3 (1950).

63. Kress, H.: Alleviation of Stream Pollution from Semi-Chemical Pulping Process. Unpublished Ph. D. Thesis, Library, Va. Poly. Inst., Blacksburg, Va. (1950).

64. Labaw, L. W. and A. O. Williams: Absorption of Supersonic Waves in Water near One Megacycle, J. Acoust. Soc. Am., 19, 30-4 (1947).

65. Laidler, T. J. and E. G. Richardson: The Absorption of Supersonics in Smokes, J. Acoust. Soc. Am., 9, 217-23 (1928).

66. Lal, K. C.: Ultrasonic Velocity in Organic Solutions. II., Indian Journal of Physics, 25, 73-8 (1951).

67. Lamb, H.: "Hydrodynamics," p. 337. Dover Publications, New York, N. Y., 1945. 6 ed.

68. Lamb, J. and D. H. A. Huddart: The Absorption of Ultrasonic Waves in Propionic Acid, Trans. Faraday Soc., 46, 540 (1950).

69. ________ and J. M. M. Pinkerton: Absorption and Dispersion of Ultrasonic Waves in Acetic Acid, Proc. Roy. Soc., (London), A199, 114-30 (1949).

70. Lambert, J. D. and J. S. Rowlinson: Ultrasonic Dispersion in Organic Vapours, Proc. Roy. Soc. (London), 204, 424-34 (1950).

71. Levine, A. A., O. E. Sweeney, and C. E. Kircher, J.: Apparatus for Extraction, U. S. Pat. 2,264,390 (Dec. 2, 1941).

72. Makinson, K. R.: Transmission of Ultrasonic Waves through a Thin Solid Plate at the Critical Angle for the Dilatational Wave, J. Acoust. Soc. Am., 24, 202-6 (1952).

73. Maloney, J. O. and A. E. Schubert: The Application of Rectangular Coordinate Methods to Solvent Extraction Design, Trans. Am. Inst. Chem. Engrs., 36, 741-57 (1940).

74. Matheiu-Sicaud, A.: Study of Dispersions with the Electron-Microscope. II. Ultrasonics, Brit. A., BI, 563 (1952); Memorial des Services Chimiques de L'etat, 35, 33-78 (1952).

75. Maxwell, J. C.: "Encyclopedia Britannica," pp. 369-74. Encyclopedia Britannica, Inc., New York, N. Y., 1938. 14 ed.

76. McDonald, D.: Solvent Extraction of Oil from Oleaginious Material, U. S. Pat. 2,198,413 (Apr. 23, 1940).

77. McKinney, R. S., W. G. Rose, and A. B. Kennedy: Continuous Process for Solvent Extraction of Tung Oil, Ind. Eng. Chem., 36, 138-40 (1944).

78. Moen, C. J.: Ultrasonic Absorption in Liquids, J. Acoust. Soc. Am., 23, 62-70 (1951).

79. Molle, A. W. and P. W. Sieck: Longitudal and Transverse Ultrasonic Waves in a Synthetic Rubber, J. Applied Phys., 23, 888-94 (1952).

80. Neppiras, E. A. and B. E. Noltingk: Cavitation Produced by Ultrasonics: Theoretical Conditions for the Onset of Cavitation, Proc. Phys. Soc. (London), 64, 1032-8 (1951).

81. Olcott, H. S.: Solvent Extraction of Cottonseed Oil, Ind. Eng. Chem., 33, 611-5 (1941).

82. Othmer, R. E.: McCabe-Thiele Diagram Useful in Extraction Problems, Chem. Eng., 43, 325-8 (1936).

83. Parthasarathy, S., D. Srinivasan, and S. S. Chari: Absorption of Ultrasonics in Liquids from Thermal Considerations, Nature, 166, 828-9 (1950).

84. Pellam, J. R. and J. K. Galt: Application of Pulse Technique to Velocity and Absorption Measurements at 15 Megacycles, J. Chem. Phys., 14, 608-14 (1946).

85. Persell, R. M., E. F. Pollard, and E. A. Gastrock: Solvent Extraction of Cottonseed, Cotton Gin Oil Mill Press, 51, 9-11 (1950).

86. ibid, pp. 24-5.

87. ibid, pp. 30-2.

88. Pinkerton, J. M. M.: A Pulse Method for the Measurement of Ultrasonic Absorption in Liquids: Results for Water, Nature, 160, 128-9 (1947).

89. Pinkerton, J. M. M.: The Absorption of Ultrasonic Waves in Liquids and its Relation to Molecular Constitution, Proc. Phys. Soc., 62, 129-41 (1949).

90. Pollard, E. F., H. L. E. Vix, and E. A. Gastrock: Solvent Extraction of Cottonseed and Peanut Oils, Ind. Eng. Chem., 37, 1022-6 (1945).

91. Pominski, J., L. J. Molaison, A. J. Crovetto, R. D. Westbrook, E. L. D'Aquin, and W. F. Guilbeau: Solvent Extraction of Cottonseed and Peanut Oils, Oil Mill Gaz., 51, 33-9 (1947).

92. Quinn, J.: The Absorption of Ultrasonic Waves in Benzene, J. Acoust. Soc. Am., 18, 185-9 (1946).

93. Rapuano, R. A.: Ultrasonic Absorption from 75 to 280 Mc/s., Phys. Rev., 72, 78-9 (1947).

94. Ravenscroft, E. A.: Extraction of Solids with Liquids, Ind. Eng. Chem., 28, 851-5 (1936).

95. Rea, H. E., Jr. and A. C. Wamble: The Extraction of Oil from Oil-Bearing Materials by Prepressing followed by Solvent Extraction, Texas A & M College, College Station, Texas, Eng. Expt. Sta., Research Report, No 11, 1-6 (1950).

96. Richardson, E. G.: "Ultrasonic Physics," pp. 178-85. Elsevier Publishing Co., Houston, Texas, 1952. 1 ed.

97. ibid, pp. 252-3.

98. Roberts, E. N.: Recovery of Solvents from Oil, U. S. Pat. 2,081,844 (May 25, 1937).

99. Rufer, W.: Absorption Measurements on Ultrasonic Waves in Electrically Conducting Solutions, Ann. Physik, 41, 301-12 (1942).

100. Schmid, A.: The Extractor, Based on the Hansa-Muhle System, Its Construction and Manner of Operation, Fette u. Seifen, 46, 464-5 (1939).

101. Sette, D.: Experimental Results of Measurement of the Ultrasonic Absorption Coefficients in Certain Liquid Mixtures, Ricerca scientifica resostruzione, 21, 1999-2005 (1951).

102. ________: Results of Experiments to Measure the Absorption Coefficient of Ultrasonic Waves in Mixtures of Liquids, Nuovo Cimento, 7, 318-27 (1950).

103. ________: On the Ultrasonic Absorption in Binary Mixtures of Unassociated Liquids, J. Chem. Phys., 18, 1592-4 (1950).

104. Sette, D.: On the Ultrasonic Absorption in Binary Mixtures of Unassociated Mixtures, Ricerca scientifica recostruzione, 21, 2006-9 (1951).

105. Sewell, C. J. T.: Phil. Trans. Roy. Soc. (London), A210, 269 (1910). Richardson, E. G.: "Ultrasonic Physics," pp. 252-3. Elsevier Publishing Co., Houston, Texas, 1952. 1 ed.

106. Sherwood, T. K. and R. L. Pigford: "Absorption and Extraction," pp. 1-28. McGraw-Hill Book Co., Inc., New York, N. Y., 1952. 2 ed.

107. Shropshire, R. F.: Methods of Extracting Oil from Fish Material, U. S. Pat. 2,473,453 (June 14, 1949).

108. Simpson, F. W. and S. G. Mason: Treatment of Cellulose Fibers with Ultrasonic Waves, Pulp & Paper Mag. Can., 51, 70-2 (1950).

109. Spadaro, J. J., E. J. McCourtney, and H. L. E. Vix, Solvent Extraction of Cottonseed and Peanut Oils. VII. Effect of Drying Flaked Prime Cottonseed on Color of Oil and Meal Properties, J. Am. Oil Chemists' Soc., XXVII, 394-6 (1950).

110. Stokes, G. G.: On the Theories of the Internal Friction of Fluids in Motion and of the Equilibrium and Motion of Elastic Solids, Cambr. Trans. Phil. Soc., 8, 287 (1845).

111. Thiele, E. W.: Application of Graphical Method of Ponchon to Distillation and Extraction, Ind. Eng. Chem., 27, 392-6 (1935).

112. Thompson, A. D.: Transparency of Materials to Ultrasonic Waves, pp. 39-40. Unpublished B. Sc. Thesis, Library, Va. Poly. Inst., Blacksburg, Va. (1951).

113. Thompson, D.: Ultrasonic Coagulation of Phosphate Tailing, Va. Poly. Inst., Blacksburg, Va., Eng. Expt. Sta., Bull., No 75, 16-63 (1950).

114. Varteressian, K. A. and M. R. Fenske: Liquid-Liquid Extraction, Ind. Eng. Chem., 28, 928-33 (1936).

115. Verma, D. S.: Ultrasonic Absorption in Organic Liquids, Brit. A., AI, 860 (1951); Proc. Natl. Acad. Sci. India, 19A, 34-44 (1950).

116. Verma, G. S.: Effects of Humidity on Ultrasonic Absorption in Air at 1.46 Megacycles, J. Acoust. Soc. Am., 22, 861-2 (1950).

117. Vigoureux, P.: "Ultrasonics," pp. 26-44. John Wiley and Sons, Inc., New York, N. Y., 1951. 1 ed.

118. ibid, pp. 73-7.

119. Wilbrandt, F. C., et al: "Notes on Thesis Writing," Dept. of Chem. Eng., Va. Poly. Inst., Blacksburg, Va. 1950.

120. Von Schmid, G. and H. Knapp: Transmission of Ultrasound through Porous Bodies in Liquids, Nuovo Cimento, 7, 296-301 (1950).

121. Weissler, A.: Sonochemistry: The Production of Chemical Changes with Sound Waves, J. Acoust. Soc. Am., 25, 651-7 (1953).

122. ____________: Physio-Chemical Effects of Ultrasonics, "Ultrasonics -- two symposia." Chemical Engineering Progress Symposium Series, No 1, 47, pp. 22-7. American Institute of Chemical Engineers, New York, N. Y., 1951.

123. Willard, G. W.: Ultrasonic Absorption and Velocity Measurement in Liquids, J. Acoust. Soc. Am., 12, 438-48 (1941).

124. Zimmerman, O. T. and I. Lavine: "Scientific and Technical Abbreviations, Signs and Symbols," pp. 3-306. Industrial Research Service, Dover, N. H., 1949. 2 ed.

125. Zimmerman, O. T. and I. Lavine: "Scientific and Technical Abbreviations, Signs and Symbols," pp. 513-27. Industrial Research Service, Dover, N. Y., 1949. 2 ed.

VIII. ACKNOWLEDGMENTS

The author wishes first to express his gratitude to his understanding parents, whose strengthful guidance and financial aid made completion of this thesis possible.

The author wishes to express his sincere appreciation to Dr. Dudley Thompson, Associate Professor, Department of Chemical Engineering, Virginia Polytechnic Institute, for his advice, guidance, encouragement, criticism, and patience during the course of this investigation.

The author is also indebted to Dr. Frank C. Vilbrandt, Head of the Department of Chemical Engineering, Virginia Polytechnic Institute, and other members of the staff for their valuable suggestions and criticisms.

The author wishes also to express his appreciation to Mr. Melvin Price for aid in assembling the control panel for the ultra-sonorater.

IX. VITA

The author, Donald Gene Sutherland, was born in Omar, West Virginia, on August 20, 1930. His first year in high school was at St. Petersburg High School, St. Petersburg, Florida. He received the remainder of his high school education at Logan High School, Logan, West Virginia.

In September, 1948, he enrolled at the Virginia Polytechnic Institute, Blacksburg, Virginia, in the

Department of Chemical Engineering. Here he was a member of the Student A. I. Ch. E., Phi Lambda Upsilon, O. D. K., and German Club. He was also President of the Corps of Cadets and class treasurer. He received his Bachelor of Science Degree in Chemical Engineering in June, 1952. He returned to Virginia Polytechnic Institute the following August to pursue a Master of Science Degree in Chemical Engineering and held a research assistantship in that department. He completed his degree requirements in November, 1953.

D. G. Sutherland