# THE MEASUREMENT OF THE POSITION OF THE INTERFACE IN A LIQUID-LIQUID EXTRACTOR

by

Irvin A. Jefcoat

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# I. INTRODUCTION

Liquid-liquid extraction is a unit operation that has long demonstrated its usefulness to the chemical industry. The application of liquid-liquid extraction processes in the chemical industry are too numerous to mention.

The effectiveness or efficiency of an extraction process is a function of several variables; i.e., physical and chemical properties of the system, the flow rates of the two streams, the column diameter, the type of packing used, and the extraction height. Once the column is designed and constructed for a particular system, the operational efficiency of the column depends primarily on which phase is continuous and the extraction height. Since the column is operated with one phase continuous and the other discontinuous, the extraction height is the remaining variable which must be controlled in order to minimize its effect on the extraction process.

In universities the extraction height is often controlled by a needle valve that must be set by the experimentor. A system of automatic control of the extraction height would enable the experimentor to treat the extraction height as a constant. Each control system depends primarily on the measuring or detecting element for reliable control.

The purpose of this investigation is to evaluate a differential pressure, an electrical capacitance, and an electrical resistance measuring element to measure the location of the aqueous-organic interface in a liquid-liquid extractor.

# II. LITERATURE REVIEW

The review of the literature covered publications on the operational characteristics of packed pulsed columns, the important physical properties of the system, (nitric acid, tri-n-butyl phosphate, water, and xylene), and liquid-level indicators and their adaptation to a control system.

## Operational Characteristics

The operational characteristics obtained from the literature were qualitative information of flooding and hold-up.

Flooding. Chantry, et al(1), defines the flooding point of a pulse column as the condition at which the hold-up is too large to accommodate any increase in either phase rate. An increase in either phase rate above the flooding point will cause the dispersed phase to become continuous at some point in the column.

As the amplitude(1) of the pulse is increased at a constant frequency the H.E.T.S. decreases to a minimum and then increases as flooding is approached. When the flooding point is reached, the column becomes ineffective because of the high degree of agitation which causes the drop sizes to be too small to coalesce readily.

Thornton(10) reported that he was unable to reproduce either flood point or mass transfer data using a pulsed packed column because of the progressive effect of random packing. The trouble experienced by Thornton was not experienced by other investigators(1,4,11). It was eliminated by retaining the packing in place with various pressure plates, usually a screen at the bottom of the packing and a drilled plate at the top.

Hold-up. The hold-up in a packed column can be defined as the inventory of the dispersed and continuous phases required to fill the column. Gayler, et al(2), give the following empirical formula for the hold-up in a packed tower.

$$V_d + \frac{x}{1-x} V_c = \epsilon v_o \, x \, (1-x)$$

where:

$V_d$ = velocity of dispersed phase, ft/hr.

$V_c$ = velocity of continuous phase, ft/hr.

$v_o$ = the mean droplet velocity relative to the packing at zero flow rates, ft/hr.

$x$ = fractional hold-up

$\epsilon$ = fractional voidage of packing.

This relation was derived for an unpulsed column.

Leva and Wen(4), propose three types of hold-up in a packed column: (1) free hold-up which comprises the dispersed phase drops that rise freely to the interface, (2) operational hold-up which takes in the free hold-up in addition to the drops freed from the packing by pulsation, (3) and total hold-up which is the entire hold-up of the dispersed phase within the effective packing volume. In a pulsed column, operational hold-up is the major contributor to the total hold-up.

Chantry(1) and Wiegandt(11) report that the effect of the continuous phase flow rates on hold-up in a pulsed column is "almost nothing", while the dispersed phase flow rate has "some effect". The hold-up is more a function of the pulsing motion than the flow rates. For example, the dispersed phase hold-up may increase from five percent in an unpulsed column to fifty percent or more in one which is pulsed.

## Physical Properties

The physical properties of the system, tri-n-butyl phosphate-xylene-water-nitric acid, are summarized in Table I, page 6.

## TABLE I

## Physical Properties of the System

| Component | Density[a]<br>gm/ml | Dielectric Constant<br>dimensionless | Resistance[a]<br>ohms/cu cm |
|---|---|---|---|
| Nitric Acid | 1.502 | --- | 3.2 (6.2N) |
| Tri-n-butyl phosphate | 0.973-0.983 | 7.95 | --- |
| Water | 1.000 | 78.54 | --- |
| Xylene | 0.86105-0.88020 | 2.270-2.568 | --- |

[a] Handbook of Chemistry and Physics, 34, Chemical Rubber Publishing Co., Cleveland, Ohio, 1952.

[b] Maryott, A. A. and E. R. Smith: Table of Dielectric Constants of Pure Liquids, National Bureau of Standards Circular No. 514, (1951).

## Liquid-Level Indicators

Measurement of liquid level can be accomplished by several means, some of which are:

1. Measurement of the liquid level(6) by using the liquid as the dielectric for a capacitor whose change in capacitance is detected by a resonant frequency circuit or a capacitive bridge circuit.

2. An inductive-type measuring circuit(6) for determining liquid-level employs a float mechanism linked to the core of the coil. The movement of the float changes the position of the core thereby changing the mutual inductance of the coil which in turn changes the coil impedance which causes the current through the coil to vary. A meter connected in series with the coil indicates the change in current.

3. An A-C Wheatstone Bridge circuit(8) used with a conducting liquid as the unknown resistance.

4. A photoelectric tube(6) used to receive a beam of light refracted by the liquid. This is in general an on-off operation.

5. A gamma-ray source(6) radiating into a tank. A detector measures the scattered radiation which is proportional in quantity to the number of molecules encountered by the direct radiation.

6. Measurement of the liquid-level(5) by differential pressure using either the bubbling principle or large diaphragm instruments.

Differential Pressure. The most common type of differential pressure instrument(9) uses a nonmetallic diaphragm to detect small changes in pressure. The detection of these small changes in pressure is accomplished through mechanical linkage of a spring to the diaphragm and applying pressure to both sides of the diaphragm. One side of the diaphragm is maintained at a fixed pressure which is equal in magnitude to the median of the pressure on the other side of the diaphragm. As this pressure varies about its median the movement of the diaphragm is transferred through the spring to an indicator arm that moves across a calibrated scale. This movement can be translated into a signal that can be used to operate a control. The diaphragm type of differential pressure measurement is sensitive to pressure differentials as low as 0.2 inches of water with an accuracy of ± 1/2 to ± 1.0 per cent of the full scale movement of the indicator.

Liquid-Level Measurement Using the Air Bubbling Principle. The gas purge or bubbling system(5) requires a gas source at a fixed flow rate to supply the two lines. One line is vented to the atmosphere and the other is submerged in the liquid. The pressure in the two lines is set with needle valves. The needle valve on the line that is submerged in the liquid is adjusted until a bubble rate of 6 to 8 bubbles per minute is obtained. The two lines are connected to a differential pressure instrument; such as, a manometer, through a tee. The pressure drop in the lines is directly proportional to the liquid level in the tank.

Pressure Instrument Errors. The three most common errors(5) in pressure measuring instruments are: (1) friction, (2) lost motion, (3) hysteresis. Errors caused by friction are reduced by carefully machining pivots and bearings and by making the moving parts light in weight. The effect of friction on the pointer is reduced by making the sensitive element large and powerful so that frictional forces are a smaller percentage of the available forces. Lost motion or play in bearings or mechanical linkage is reduced by careful manufacture of all parts. Hysteresis is the failure of the sensitive element to return to the same position when a given value of the measured variable is approached from opposite directions. In industrial instruments hysteresis, lost motion, and friction are measured together and the combined error is expressed as percentage of the full scale deflection of the instrument. In precision instruments this total error rarely exceeds 1/2 of 1.0 per cent.

Electrical Capacitance. Measurements based upon variations of capacitance(6) are achieved by any one or any combination of the following mechanisms:

1. Variation of capacitance by a change in the separation of the plates.

2. Variation of capacitance by a change in the area of the plates.

3. Variation of capacitance by a change in the inductive capacity of a dielectric.

The first of these mechanisms has received the widest application in engineering gaging systems. In using this method in liquid level measurement(3,6), one plate of the capacitor is suspended above the liquid and the liquid itself is made the other plate. As the liquid level rises or falls this will create a change in the capacitance of the system and this change can be detected by a suitable electric circuit.

Liquid level measurement and control(3) by electrical capacitance is achieved by a capacitance probe or probes in the liquids. As the liquid level varies, the dielectric constant of the capacitance probe changes. This change in capacitance is detected by an electronic instrument operating at either audio or radio frequencies. The output of the instrument can be transformed into a signal for control.

The probe can be constructed from a bare or coated metal strip. This structure is suspended in the liquid by attaching it to the vessel with some form of insulating material. The capacitance of a parallel plate capacitor with two plates(7,6) can be calculated by use of the formula:

$$C = 0.225\, K \frac{A}{D}$$

where:

$C$ = capacitance, micromicrofarads

$A$ = plate area, sq.in.

$d$ = distance between plates, in.

$K$ = dielectric constant, micromicrofarads, per inch.

Often the application requires cylindrical instead of flat plates as electrodes. A formula for the capacitance of a condenser formed with concentric cylindrical plates is:

$$C = \frac{0.614\ KL}{\log_{10} \frac{b}{a}}$$

where:

C = capacitance, micromicrofarads

K = dielectric constant of material between plates, micromicrofarads per inch.

L = length of cylinder, inches

b = inner diameter of outer electrode, inches

a = outer diameter of inner electrode, inches.

Commercial instruments(7) using these types of probes are available. For a probe capacitance of ten micromicrofarads a full scale deflection can be obtained from some of these instruments with a change of probe capacitance of 0.01 micromicrofarads.

Several disadvantages(6) of the two-plate capacitance measuring element are: change in sensitivity with change of plate spacing, or non-linearity; change in zero setting with change in temperature, or zero drift; change in calibration with change in temperature, or temperature error; and danger of random and extraneous effects being recorded, or instability. These disadvantages are all reduced by careful mechanical design.

Resistance. The change(6) in resistance of an element may be accomplished by changing either or both the temperature and physical dimensions of the element. These changes occur primarily from the change in resistivity of the material of which the element is composed; that is, the material has either a temperature coefficient of resistivity or a stress coefficient of resistivity. (Resistivity is defined as the resistance between opposite faces of a centimeter cube of the material.) Resistance is increased by an increase in the length of the conductor or by decreasing the cross-sectional area of the conductor. Changes in dimension and properties may occur simultaneously; i.e., an expansion or shrinkage of the material caused by a change in its temperature.

In variable-resistance elements in which an electrolytic liquid(6) is the resistance material, movement of the electrodes changes the length and perhaps the distribution of the current paths while not affecting the resistivity. This same effect can be produced by moving the liquid or changing the amount of liquid between the electrodes. This principle can be applied to liquid-level measurement.

Electrical resistance or conductivity measurements(8) of liquid-levels can be accomplished by the use of an A-C Wheatstone Bridge using a voltage source of either 60 or 1000 cycles per second. Two electrodes placed in the liquid form the unknown leg of the bridge. The detector is an electronic instrument whose output can be used as the signal for a control system.

## Electric Circuits

The types of electric circuits most often used to detect changes in capacitance and resistance are presented here.

Capacitance Detecting Circuits. The function of an electric circuit used with a capacitance measuring element(6) is to convert change in capacitance into a change in voltage, current, or frequency and to indicate or record these changes. There are two general methods by which this conversion can be made. One makes use of two electrically oscillating circuits coupled together, Figure 1, page 15, where the capacitance of one oscillator is determined by the measuring element; the resonant frequency of this circuit depends upon the capacitance of this element according to the relation:

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

where:

$f$ = resonant frequency, cycles per second

$L$ = inductance, henries

$C$ = capacitance, farads.

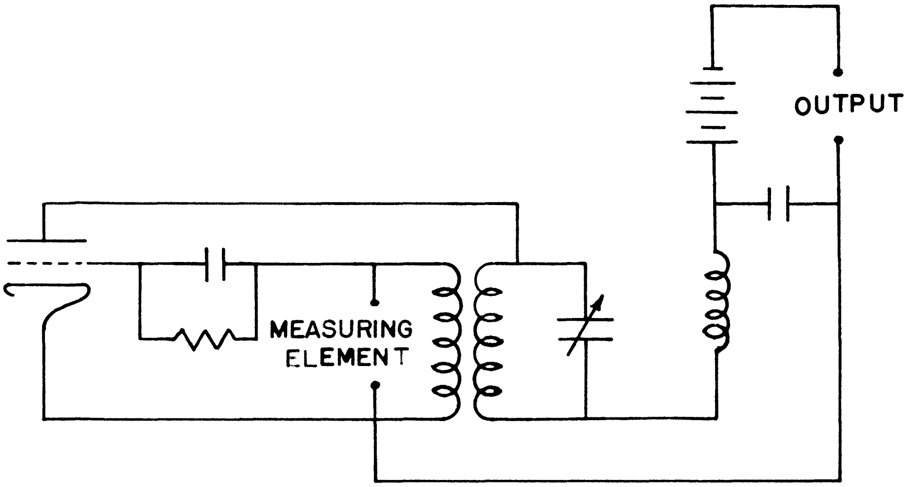


FIGURE 1. RESONANCE PRINCIPLE CAPACITANCE MEASURING CIRCUIT

The two circuits are adjusted to oscillate at equal frequencies with the capacitance measuring element in its median position. Since the two circuits are inductively coupled together, a change in the frequency of one will cause a change in the plate current of the oscillating electron tube. By careful adjustment of the two circuits the plate current can be made to follow the capacitance of the measuring element.

The other method uses the measuring element as a reactance in a bridge circuit, Figure 2, page 17. If the circuit is balanced to zero output for the static condition, a change in the capacitance element will cause a proportional change in the output voltage. The output of the bridge is amplified and recorded by a graphic instrument, or indicated by a pointer-type instrument.

<u>Resistance Detecting Circuits</u>.(6) The two most popular methods for measuring resistance are the null bridge and A-C bridge circuit. The null bridge circuit, Figure 3, page 18, is the most sensitive and has the greatest accuracy. This type of bridge is known as a Wheatstone. The operation of the circuit can be deduced from Figure 3, page 18; at balance, no current flows between points A and B, the potential drop across $R_2$, equals that across $R_3$, and that

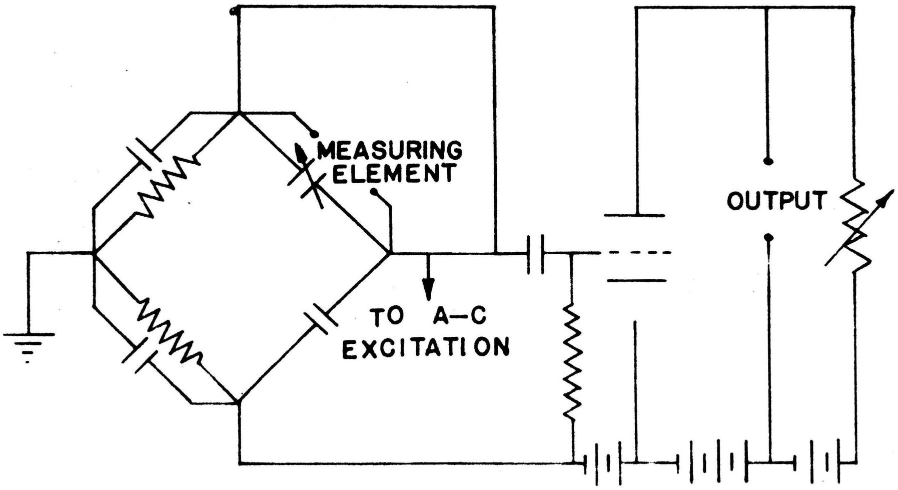
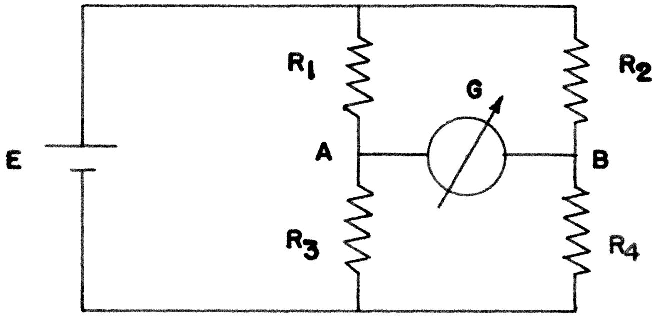


FIGURE 2. BRIDGE CIRCUIT USED TO MEASURE CHANGES IN CAPACITANCE



LEGEND:

A,B = NULL POINTS

$R_{1,2,3}$ = BALANCING RESISTORS

$R_4$ = MEASURING RESISTANCE

E = D-C VOLTAGE SOURCE

G = GALVANOMETER

FIGURE 3. NULL BRIDGE RESISTANCE MEASURING CIRCUIT

across $R_2$ equals that across $R_4$. If no current flows through G, the currents through $R_1$ and $R_2$ must be equal, as must be those through $R_3$ and $R_4$. This condition can exist only when

$$R_1/R_2 = R_3/R_4$$

From this simple relationship, if any three of the resistances are known at balance, then the fourth can be calculated.

The alternating-current bridge circuit, Figure 4, page 20, is preferred over the direct current bridge when it is necessary to amplify the output. In any A-C bridge the output increases with the departure from balance; unlike D-C bridge circuits, direction of unbalance is not clearly indicated by polarity of output current or potential. However, the phase of the output potential of an A-C bridge circuit is changed 180° by passing through the balance point, so that by using phase-sensitive rectifiers both magnitude and direction of unbalance may be indicated.

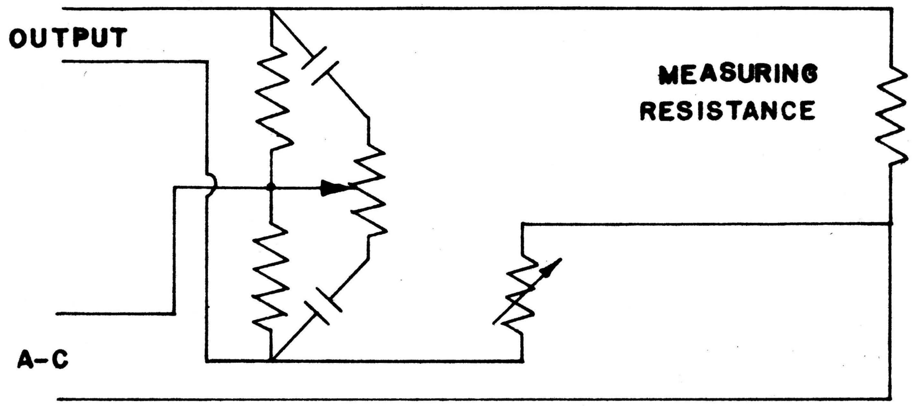


FIGURE 4. A-C BRIDGE CIRCUIT FOR MEASURING RESISTANCE

# III. EXPERIMENTAL

This section contains the purpose and plan of investigation, materials and apparatus used, method of procedure, data, and results obtained in this investigation.

## Purpose of Investigation

The purpose of this investigation is to evaluate a differential pressure, an electrical capacitance, and an electrical resistance measuring element to measure the location of the aqueous-organic interface in a liquid-liquid extractor.

## Plan of Experimentation

The following general plan was followed during this investigation.

Literature Review. A review of the literature was conducted in order to determine the operational characteristics of a pulsed-packed extractor, the physical properties of the system, and various methods of measuring liquid level and their adaptability to control.

Equipment. Three types of liquid-level measuring units were selected and assembled. These were a differential pressure, electrical capacitance, and an electrical resistance unit.

Data and Results. The three units were tested for stability and sensitivity and the results compared. From the data obtained the more reliable unit was selected.

## Materials

The following is a list of materials used in this investigation.

Air. Compressed, 20 pounds per square inch, gage. Obtained from air supply lines in the Chemical Engineering Department's Unit Operations Laboratory, Virginia Polytechnic Institute, Blacksburg, Virginia. Used to operate the recorder-controller and differential pressure unit.

Nitric Acid. Reagent grade. Obtained from Allied Chemical Corp., Richmond, Virginia. Used in the aqueous phase of the electrical resistance system.

Tri-n-Butyl Phosphate. Industrial grade. Obtained from Commercial Solvent Corporation, Cincinnati, Ohio. Used as the organic phase for the electrical resistance and capacitance systems.

Water. Single distilled. Obtained from the distillation unit in the ion exchange laboratory of the Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Virginia. Used as the aqueous phase in each system.

Xylene. Meets ASTM D-95-46. Cat. No. X-4, Lot No. 702075. Obtained from Fisher Scientific Co., Silver Spring, Maryland. Used as the organic phase in the differential pressure system.

## Apparatus

The following is a list of the apparatus used in this investigation.

Air Manifold. Constructed from 1/2 inch standard steel pipe with five gate valves and five pressure gages. Used to distribute supply air to the differential pressure system.

Beakers. 2000, 3000, and 4000 ml. Obtained from Fisher Scientific Co., Silver Spring, Maryland. Used to hold organic and aqueous phases for each system.

**Capacitance Measuring Unit**. Fielden Telstor, Serial No. 33610, Range 25-2500 micromicrofarads, output at full scale 1.1 milliamperes or 250 millivolts. Manufactured by Fielden Instrument Division of Robertshaw Fulton Controls Co., Philadelphia, Pennsylvania. Used to detect and indicate changes in capacitance.

**Conductivity Recorder**. Resistance Dynalog, multi-record conductivity recorder, Model 9160, Serial No. H24066, Range 0-100, linear in conductivity (0-100 micro mho with a cell constant of 0.01). Manufactured by the Foxboro Co., Foxboro, Massachusetts. Used to record conductance (reciprocal resistance) and as the source of the 1000 cycle current for conductivity measurements.

**Differential Pressure Instrument**. Range: 0-25 inches of water. Manufactured by Foxboro Co., Foxboro, Massachusetts. Used as part of differential pressure unit.

**Differential Pressure Instrument**. Range: 0.2-3.5 inches of water differential, Model 175D. Manufactured by Moore Products, Richmond, Virginia. Used as part of the differential pressure unit.

**Needle Valve**. One-quarter inch. Manufactured by Foxboro Co., Foxboro, Massachusetts. Used to regulate the air supply to the pressure probes.

Platform Jack. Eight inch square platform, cross-membered extension jack. Obtained from Fisher Scientific Co., Silver Spring, Maryland. Used as an adjustable support for the xylene-water system used with the differential pressure unit.

Polyethylene Tubing. Size: 1/4 and 3/8 inch. 44P-1/4 and 66P-3/8. Manufactured by Imperial Brass Co., Cleveland, Ohio. Used as air lines and syphon hoses.

Pressure Gages. 0-30 pounds per square inch, gage. Quantity: 7. Manufactured by Taylor Instruments. Used on air manifold and differential pressure instruments.

Probe. Capacitance, Concentric cylindrical plates, eight inches in length. Constructed from teflon coated 1/4 inch stainless steel rod and 3/4 inch galvanized steel pipe. Used as the measuring element in the capacitance unit.

Probe. Pressure. Quantity: 2, one ten inches and the other 7.5 inches in length. Constructed from 1/4 inch stainless steel tubing and mounted two inches apart in a stainless steel plate eight inches in diameter and 1/4 inch thick. Used as the bubbling probes for the differential pressure unit.

Probe. Resistance. Quantity: 2, nine inches in length. Constructed from 1/4 inch stainless steel tubing and mounted 1/2 inch apart in 6 x 6 x 1/2 inch plywood board. Used as the measuring element for the resistance unit.

Recorder-Controller. Serial No. 586774. Manufactured by the Brown Instruments Division of Minneapolis Honeywell, Philadelphia, Pennsylvania. Used to record the output of the capacitance unit.

Recorder-Controller. Model 5310, Serial No. 255748. Manufactured by the Foxboro Company, Foxboro, Massachusetts. Used to record the output from the differential pressure unit.

Stop-cock. Glass. Obtained from Fisher Scientific Co., Silver Spring, Maryland. Used with syphon hoses.

## Method of Procedure

The methods of procedure used in evaluating the stability and sensitivity of the differential pressure, electrical capacitance, and electrical resistance unit are explained in the following paragraphs.

Differential Pressure Unit. The differential pressure unit was assembled as shown in Figure 5, page 28. The beaker, E, was three-fourths filled with 50 per cent distilled water and 50 per cent xylene. Valves V-1 and V-4 were opened to supply air at 20 pounds per square inch, gage, to the instruments. The bubble rate was set at six to eight bubbles per ten seconds. The position of the interface was changed in increments of 1/2-inch between the bubble probe ends, starting with both probes in the organic phase to both probes in the aqueous phase and back to both probes in the organic phase by raising and lowering the platform jack, F. The output at each increment was recorded on the recorder, D, for a series of five tests conducted at one day intervals. During the interval between the daily test, the interface was left at a median position between the probe ends for stability tests and the output of the unit recorded before and after each daily test.

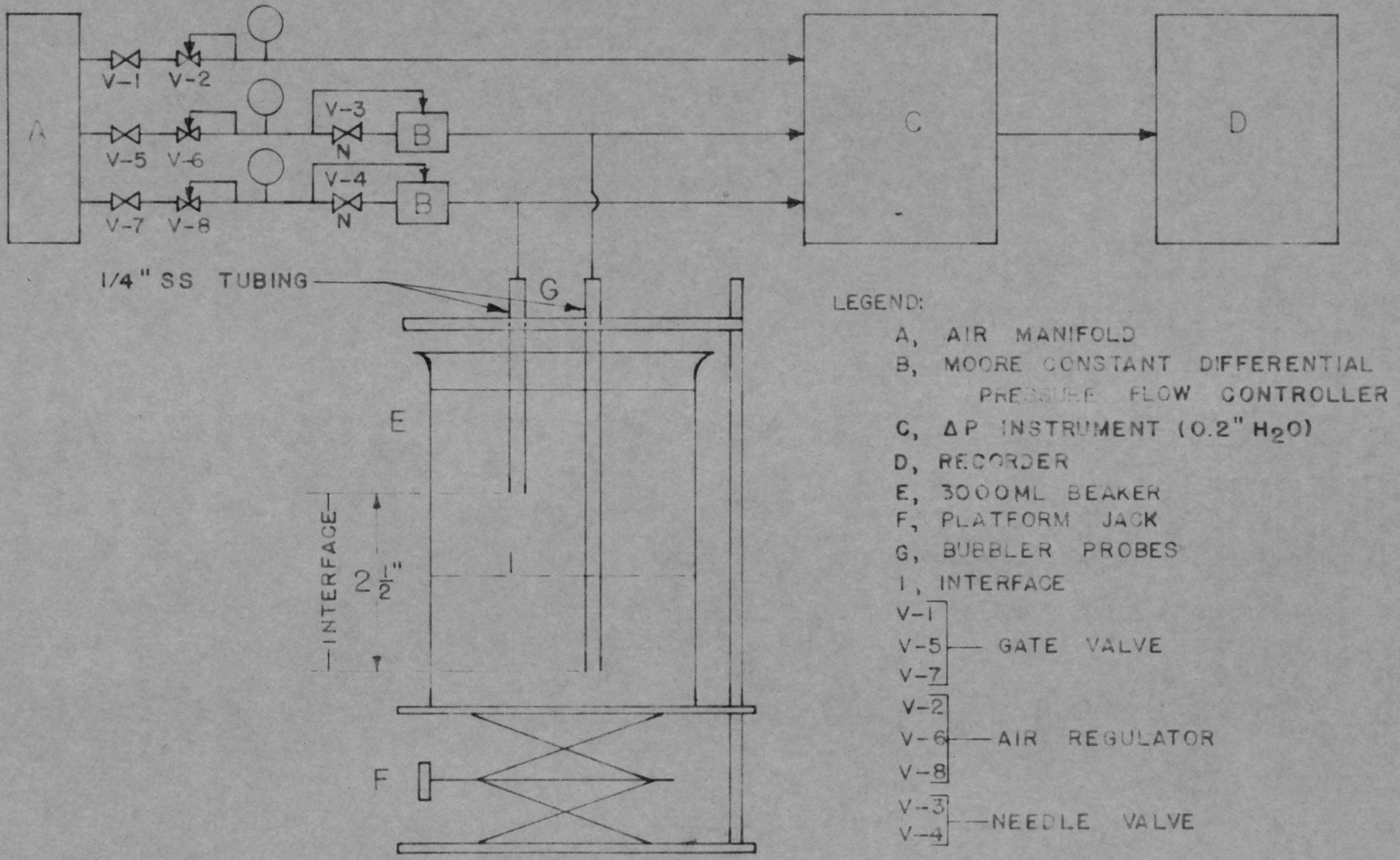


FIGURE 5. BLOCK DIAGRAM OF DIFFERENTIAL PRESSURE UNIT

Electrical Capacitance Unit. The electrical capacitance unit was assembled as shown in Figure 6, page 30. The beaker, F, was filled with 50 per cent distilled water and 50 per cent tri-n-butyl phosphate. The capacitance measuring instrument was turned on and allowed to warm up for one hour. The capacitance measuring instrument was then calibrated according to the procedure outlined in the manufacturer's bulletin(7). The position of the interface was varied in 1/2 inch increments by adding one phase and syphoning off the other. A complete cycle was made for each test; i.e., the probe depths were varied from complete submergence in the organic phase to complete submergence in the aqueous phase and back to complete submergence in the organic phase. Two tests were conducted each day for five days with the capacitance measuring unit being recalibrated each day between tests. The interface was left at a median position for 24 hours for stability tests and the output recorded before and after each day's tests.

Electrical Resistance Unit. The electrical resistance unit was assembled as indicated in Figure 7, page 31. The beaker, E, was filled with distilled water and approximately four milliliters of concentrated nitric acid was added to the water. Half of the resulting dilute acid solution was withdrawn and the beaker refilled with tri-n-butyl phosphate.

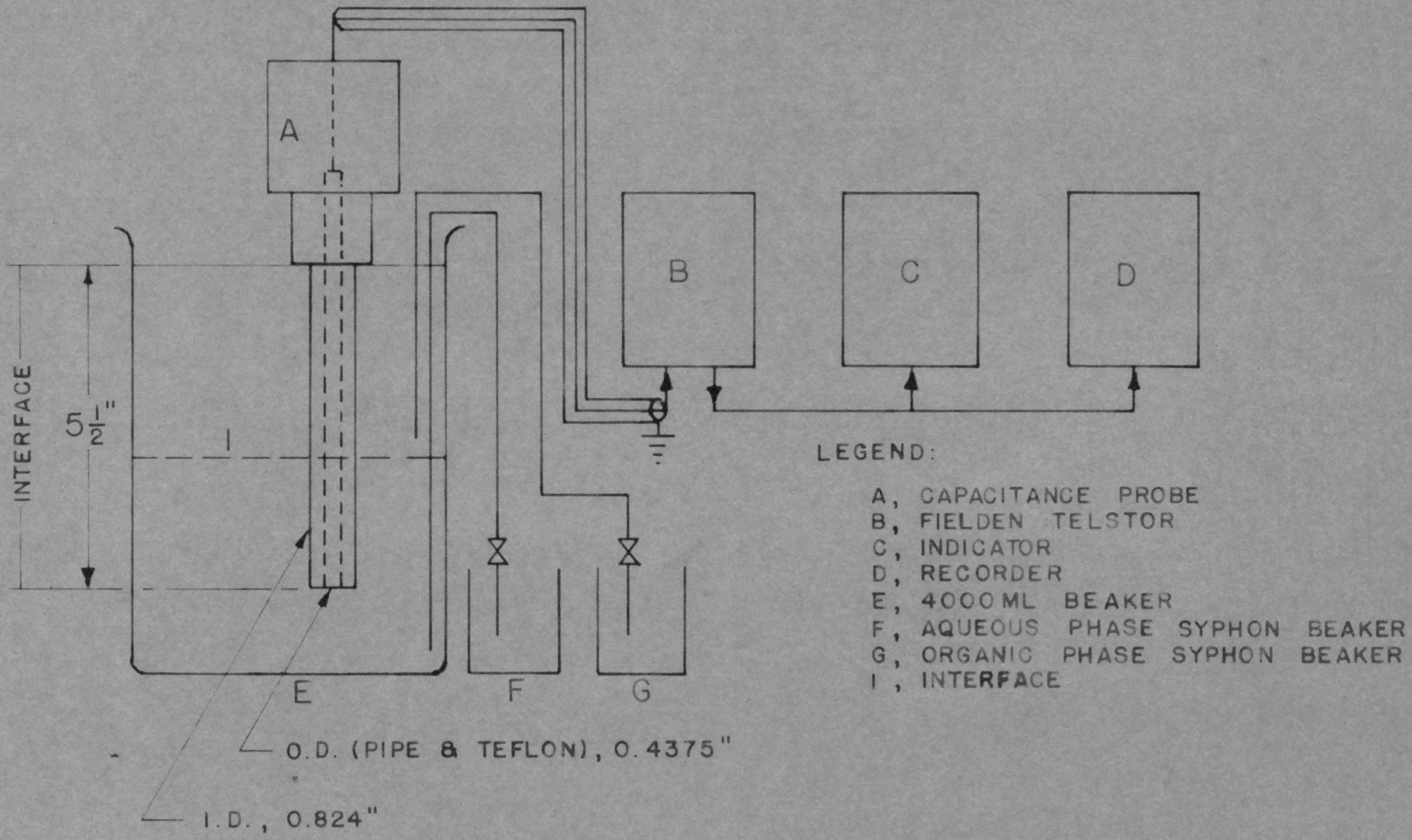
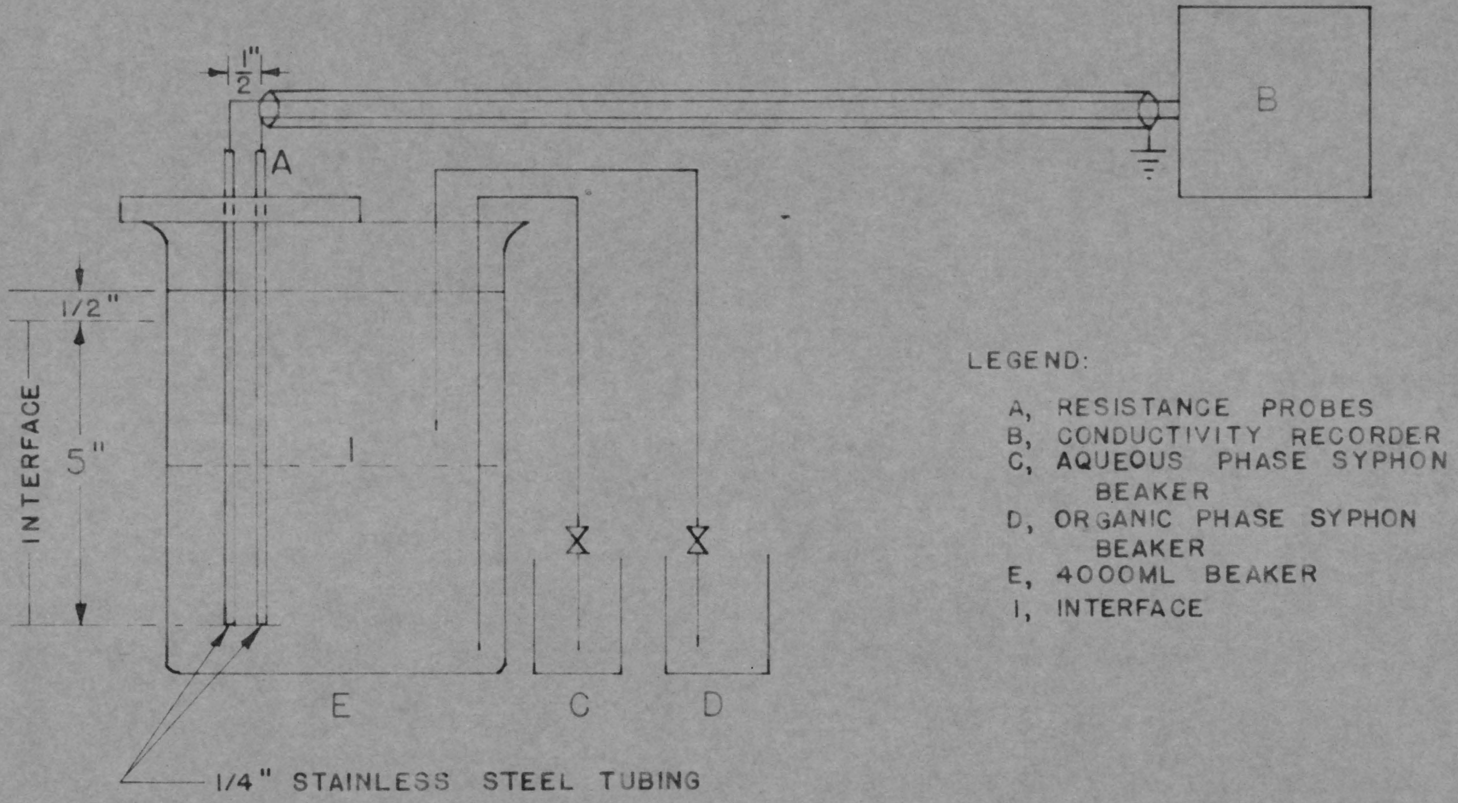


FIGURE 6. BLOCK DIAGRAM OF ELECTRICAL CAPACITANCE UNIT



LEGEND:

A, RESISTANCE PROBES
B, CONDUCTIVITY RECORDER
C, AQUEOUS PHASE SYPHON BEAKER
D, ORGANIC PHASE SYPHON BEAKER
E, 4000ML BEAKER
I, INTERFACE

FIGURE 7. BLOCK DIAGRAM OF ELECTRICAL RESISTANCE UNIT

The conductivity recorder, B, was turned on and allowed to warm up for one hour. The position of the interface was varied in 1/2 inch increments from 1/2 inch organic and five inches of aqueous phase to all organic and back to 1/2 inch organic and five inches of aqueous phase, by adding one phase and syphoning off the other. The interface was left in a median position for 24 hours for stability tests and the output of the unit recorded before and after each day's tests.

## Data and Results

The data and results obtained from the differential pressure, electrical capacitance, and electrical resistance units tested are presented in this section.

Performance of Differential Pressure Unit. The data obtained from tests to determine the sensitivity of the differential pressure unit to a change in the position of the interface are presented in Table II, page 33. The data for the stability of the unit are presented in Table III, page 34. Figure 8, page 35, represents the best curve for the data contained in Table II.

## TABLE II

## Sensitivity of the Differential Pressure Unit

| Probe Depth<br>Organic<br>in. | <br>Aqueous<br>in. | Test No. | Output<br>Per Cent of Span[a]<br>1 | <br><br>2 | <br><br>3 | <br><br>4 | <br><br>5 |
|---|---|---|---|---|---|---|---|
| 2.5 | 0.0 | | 16.5 | 12.5 | 12.5 | 12.5 | 12.5 |
| 2.0 | 0.5 | | 36.0 | 34.5 | 33.0 | 33.0 | 33.0 |
| 1.5 | 1.0 | | 40.5 | 37.5 | 37.0 | 36.5 | 37.0 |
| 1.0 | 1.5 | | 44.0 | 42.0 | 40.5 | 41.0 | 41.5 |
| 0.5 | 2.0 | | 48.0 | 46.5 | 45.0 | 46.0 | 46.0 |
| | | | | | | | |
| 0.0 | 2.5 | | 43.0 | 35.0 | 39.0 | 36.0 | 27.0 |
| 0.5 | 2.0 | | 48.0 | 46.5 | 46.0 | 46.0 | 46.0 |
| 1.0 | 1.5 | | 44.0 | 42.0 | 42.0 | 41.5 | 41.0 |
| 1.5 | 1.0 | | 40.5 | 37.5 | 37.0 | 37.0 | 37.5 |
| 2.0 | 0.5 | | 36.0 | 34.5 | 33.0 | 32.5 | 32.5 |
| 2.5 | 0.0 | | 14.0 | 12.5 | 12.5 | 12.5 | 12.5 |

[a] Span of a calibrated 3-15 psig pneumatic recorder.

## TABLE III

## Drift of the Differential Pressure Unit at Constant Interface

| Date | Probe Depth<br>Organic<br>in. | <br>Aqueous<br>in. | Output<br>Per Cent of Span | Drift<br>Equivalent Inches of Interface |
|---|---|---|---|---|
| 4/9/62 | 1.5 | 1.0 | 41.0 | |
| 4/10/62 | | | 40.5 | 0.43 |
| 4/10/62 | | | 37.5 | |
| 4/11/62 | | | 39.5 | 0.32 |
| 4/11/62 | | | 37.5 | |
| 4/12/62 | | | 38.0 | 0.15 |
| 4/12/62 | | | 37.5 | |
| 4/13/62 | | | 37.5 | 0.06 |
| 4/13/62 | | | 36.5 | |
| 4/14/62 | | | 37.0 | 0.00 |
| | 1.5 | 1.0 | | 0.23, Average |

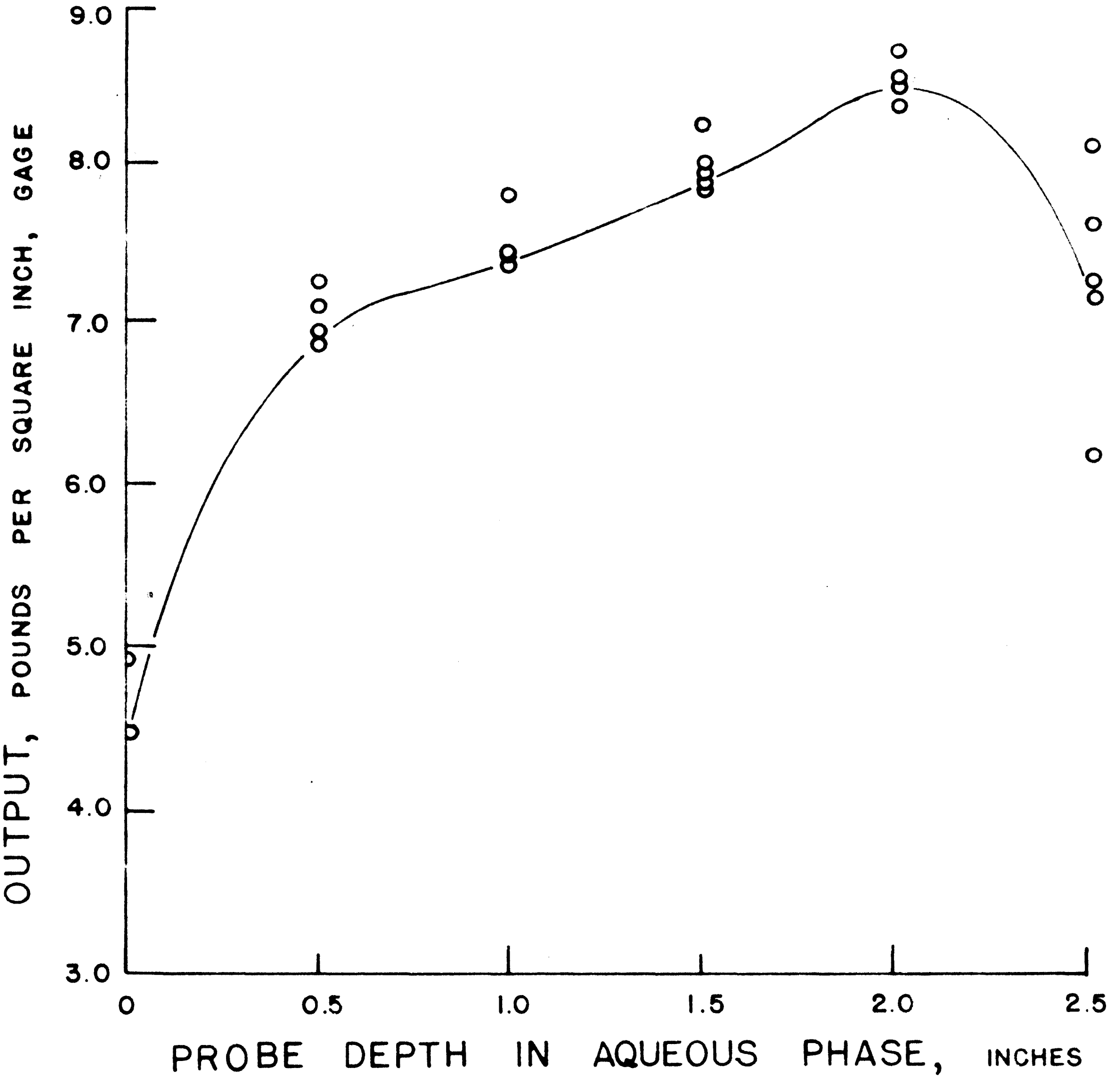


FIGURE 8. CALIBRATION CURVE FOR THE DIFFERENTIAL PRESSURE PROBE

<u>Performance of Electrical Capacitance Unit</u>. The data obtained from tests to determine the sensitivity of the electrical capacitance unit are presented in Table IV, page 37. The data for the stability of the unit are presented in Table V, page 38. Figure 9, page 39, represents the best curve for the data contained in Table IV.

<u>Performance of Electrical Resistance Unit</u>. The data obtained from tests to determine the sensitivity of the electrical resistance unit are presented in Table VI, page 40. The data for the stability of the unit are presented in Table VII, page 41. Figure 10, page 42, represents the best curve for the data contained in Table VI.

## <u>Sample Calculations</u>

The sample calculations employed in converting the data for the differential pressure and electrical resistance units from per cent of span to pounds per square inch, gage, and micromhos, respectively, are presented below.

# TABLE IV

## Sensitivity of the Electrical Capacitance Unit

| Probe Depth | | Output | | | | |
|---|---|---|---|---|---|---|
| Organic<br>in. | Aqueous<br>in. | ma | | | | |
| | Test No. | 1 | 2 | 3 | 4 | 5 |
| 5.5 | 0.0 | | 1.04 | 1.05 | * | * |
| 5.0 | 0.5 | | 1.02 | 1.05 | * | * |
| 4.5 | 1.0 | | 1.00 | 1.03 | * | * |
| 4.0 | 1.5 | | 0.92 | 0.95 | 1.09 | * |
| 3.5 | 2.0 | | 0.80 | 0.84 | 0.98 | 1.09 |
| 3.0 | 2.5 | | 0.70 | 0.76 | 0.86 | 1.02 |
| 2.5 | 3.0 | | 0.59 | 0.65 | 0.75 | 0.91 |
| 2.0 | 3.5 | | 0.48 | 0.53 | 0.62 | 0.78 |
| 1.5 | 4.0 | | 0.40 | 0.41 | 0.52 | 0.69 |
| 1.0 | 4.5 | | 0.32 | 0.28 | 0.40 | 0.58 |
| 0.5 | 5.0 | | 0.21 | 0.18 | 0.28 | 0.44 |
| 0.0 | 5.5 | | 0.12 | # | 0.17 | 0.35 |
| Unit Recalibrated | | | | | | |
| 0.0 | 5.5 | 0.10 | 0.10 | 0.10 | 0.11 | 0.10 |
| 0.5 | 5.0 | 0.30 | 0.22 | 0.22 | 0.25 | 0.17 |
| 1.0 | 4.5 | 0.40 | 0.32 | 0.32 | 0.37 | 0.28 |
| 1.5 | 4.0 | 0.50 | 0.43 | 0.44 | 0.54 | 0.42 |
| 2.0 | 3.5 | 0.60 | 0.56 | 0.57 | 0.64 | 0.52 |
| 2.5 | 3.0 | 0.69 | 0.65 | 0.68 | 0.75 | 0.63 |
| 3.0 | 2.5 | 0.78 | 0.75 | 0.78 | 0.86 | 0.78 |
| 3.5 | 2.0 | 0.89 | 0.87 | 0.88 | 1.00 | 0.90 |
| 4.0 | 1.5 | 0.96 | 0.96 | 1.00 | * | 1.02 |
| 4.5 | 1.0 | 1.04 | 1.04 | 1.08 | * | 1.10 |
| 5.0 | 0.5 | 1.06 | 1.05 | 1.09 | * | * |
| 5.5 | 0.0 | 1.08 | 1.07 | 1.10 | * | * |
| 5.0 | 0.5 | 1.06 | 1.09 | 1.09 | 1.09 | 1.09 |
| 4.5 | 1.0 | 1.04 | 1.08 | 1.08 | 1.08 | 1.08 |
| 4.0 | 1.5 | 0.93 | 0.99 | 1.00 | 0.99 | 0.98 |
| 3.5 | 2.0 | 0.83 | 0.89 | 0.88 | 0.89 | 0.88 |
| 3.0 | 2.5 | 0.72 | 0.79 | 0.78 | 0.79 | 0.76 |
| 2.5 | 3.0 | 0.61 | 0.70 | 0.66 | 0.68 | 0.62 |
| 2.0 | 3.5 | 0.50 | 0.61 | 0.54 | 0.57 | 0.50 |
| 1.5 | 4.0 | 0.41 | 0.48 | 0.42 | 0.46 | 0.38 |
| 1.0 | 4.5 | 0.32 | 0.34 | 0.32 | 0.35 | 0.28 |
| 0.5 | 5.0 | 0.23 | 0.23 | 0.20 | 0.23 | 0.17 |
| 0.0 | 5.5 | 0.13 | 0.14 | # | 0.13 | # |

* - output beyond upper range of indicator.

# - output beyond lower range of indicator.

## TABLE V

## Drift of the Electrical Capacitance Unit at Constant Interface

| Date | Probe Depth |  | Output | Drift |
|---|---|---|---|---|
|  | Organic in. | Aqueous in. | ma | Equivalent Inches of Interface |
| 4/9/62 | 3.5 | 2.0 | 0.81 |  |
| 4/10/62 |  |  | 0.81 | -0.38 |
| 4/10/62 |  |  | 0.89 |  |
| 4/11/62 |  |  | 0.82 | 0.00 |
| 4/11/62 |  |  | 0.90 |  |
| 4/12/62 |  |  | 0.96 | 0.05 |
| 4/12/62 |  |  | 0.91 |  |
| 4/13/62 |  |  | 1.09 | 1.25 |
| 4/13/62 |  |  | 0.88 |  |
| 4/14/62 |  |  | 0.75 | -0.62 |
|  | 3.5 | 2.0 |  | 0.00 ± 0.46, Average |

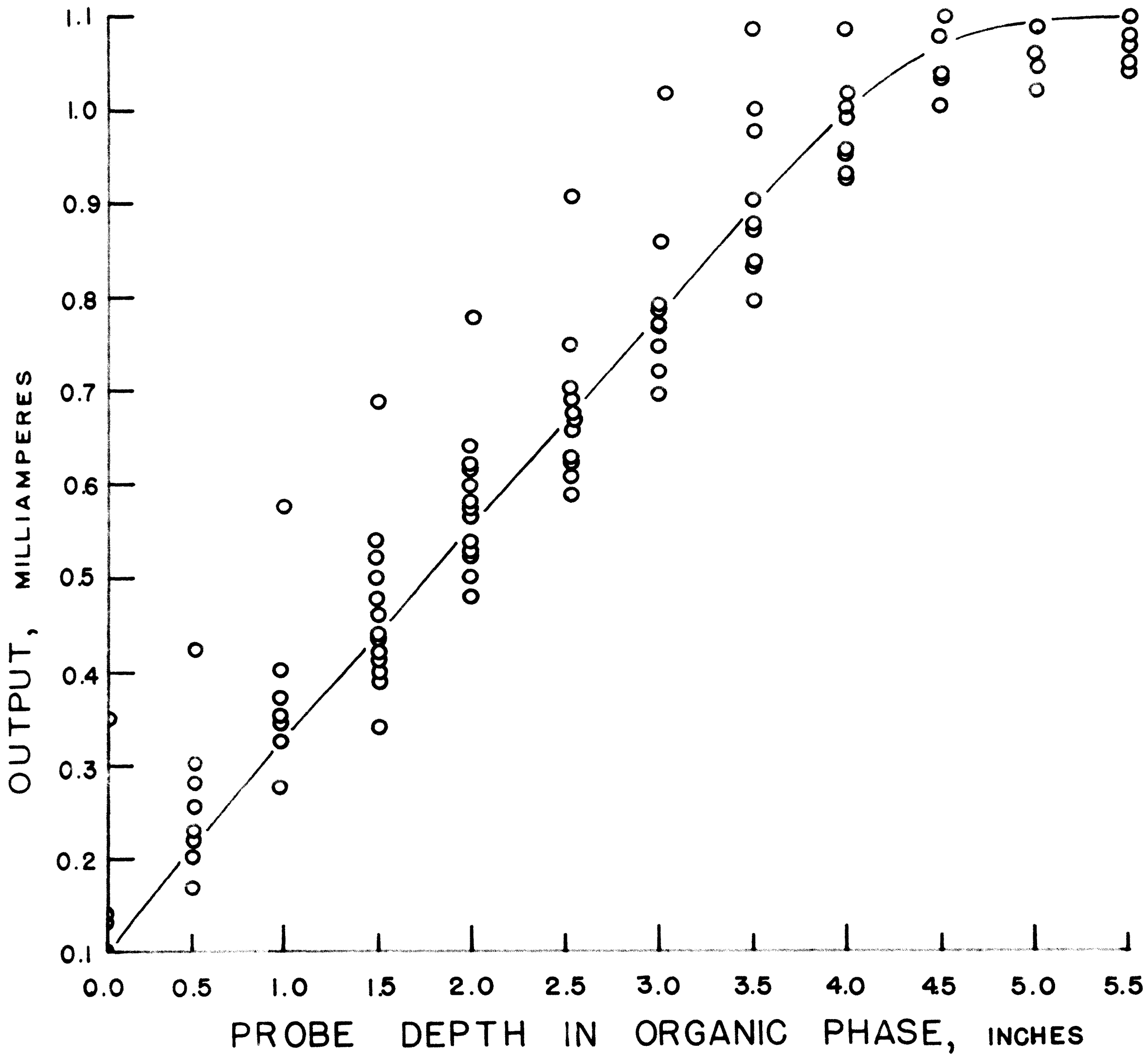


FIGURE 9. CALIBRATION CURVE FOR CAPACITANCE PROBE

## TABLE VI

## Sensitivity of the Electrical Resistance Unit

| Probe Depth | | | Output | | | | |
|---|---|---|---|---|---|---|---|
| Organic<br>in. | Aqueous<br>in. | | Per Cent of Span[a] | | | | |
| | | Test No. | 1 | 2 | 3 | 4 | 5 |
| 5.0 | 0.0 | | 1 | 1 | 1 | 1 | 1 |
| 4.5 | 0.5 | | 10 | 9 | 9 | 8 | 10 |
| 4.0 | 1.0 | | 14 | 17 | 17 | 15 | 12 |
| 3.5 | 1.5 | | 19 | 24 | 25 | 23 | 19 |
| 3.0 | 2.0 | | 24 | 30 | 30 | 32 | 27 |
| 2.5 | 2.5 | | 27 | 40 | 43 | 38 | 36 |
| 2.0 | 3.0 | | 28 | 52 | 48 | 48 | 42 |
| 1.5 | 3.5 | | * | 58 | 49 | 50 | 52 |
| 1.0 | 4.0 | | * | 67 | 62 | 62 | 59 |
| 0.5 | 4.5 | | * | 69 | 74 | 68 | 72 |
| 0.0 | 5.0 | | 87 | 79 | 84 | 74 | 78 |
| 0.5 | 4.5 | | 78 | 76 | 77 | 70 | 74 |
| 1.0 | 4.0 | | 67 | 67 | 68 | 65 | 65 |
| 1.5 | 3.5 | | 59 | 58 | 58 | 52 | 56 |
| 2.0 | 3.0 | | 52 | 48 | 50 | 48 | 45 |
| 2.5 | 2.5 | | 45 | 42 | 43 | 43 | 40 |
| 3.0 | 2.0 | | 35 | 31 | 35 | 35 | 35 |
| 3.5 | 1.5 | | 28 | 26 | 29 | 27 | 26 |
| 4.0 | 1.0 | | 19 | 16 | 20 | 18 | 18 |
| 4.5 | 0.5 | | 11 | 11 | 14 | 10 | 11 |
| 5.0 | 0.0 | | 1 | 1 | 1 | 1 | 1 |

NOTE: An 0.5 inch layer of organic phase was kept at all times.

[a] Span of 0-400 micromho conductivity recorder.

* Conductivity recorder became erratic.

## TABLE VII

## Drift of the Electrical Resistance Unit at Constant Interface

| Date | Probe Depth | | Output | Drift |
|---|---|---|---|---|
| | Organic<br>in. | Aqueous<br>in. | Per Cent of Span | Equivalent Inches of Interface |
| 4/12/62<br>4/13/62 | 2.5 | 2.5 | 43<br>43 | 0.20 |
| 4/13/62<br>4/14/62 | | | 40<br>43 | 0.20 |
| 4/14/62<br>4/15/62 | | | 42<br>43 | 0.20 |
| 4/15/62<br>4/16/62 | | | 35<br>39 | -0.10 |
| | 2.5 | 2.5 | | 0.00 ± 0.175, Average |

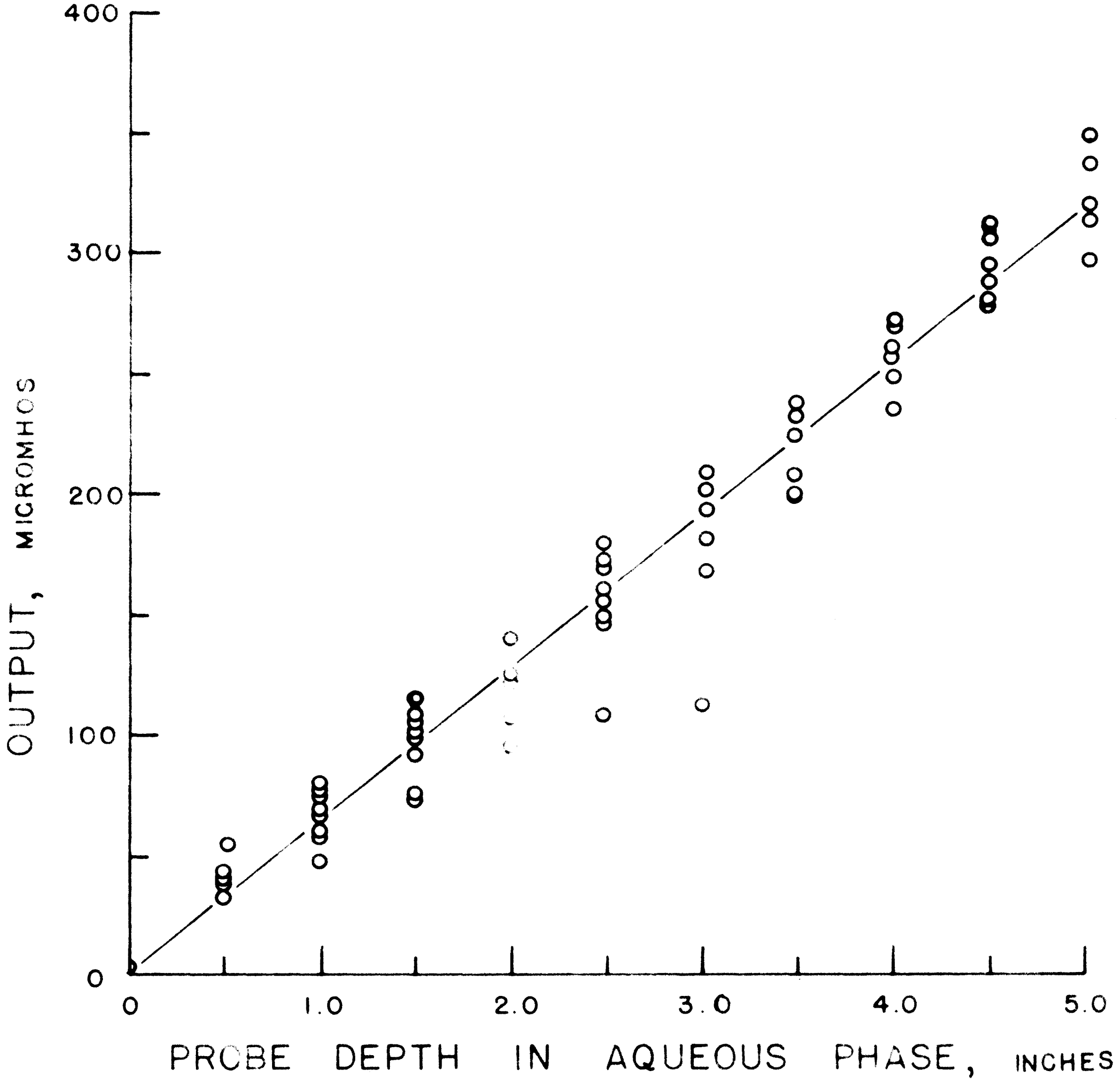


FIGURE 10. CALIBRATION CURVE FOR RESISTANCE PROBE

Conversion of Differential Pressure Unit Data. The data recorded as per cent of span for the differential pressure unit was converted by use of the following formula:

$$P = 3 + 12D$$

where:

$P$ = output, pounds per square inch, gage

$D$ = output, fraction of span.

$$P = 3 + 12(0.42)$$

$$P = 8.04 \text{ psig}$$

Conversion of Electrical Resistance Unit Data. The data recorded as per cent of span for the electrical resistance was converted to micromhos by use of the following formula:

$$C = 4D$$

where:

$C$ = output, micromhos

$D$ = output, per cent of span.

$$C = (4)(78)$$

$$C = 312\ \mu\text{mhos}$$

Conversion of Stability Data. The stability data for each unit was converted to equivalent inches of either the organic or aqueous phase by using the values obtained from the respective calibration curve of each unit.

## IV. DISCUSSION

The following paragraphs discuss the procedure used, results obtained, recommendations for future study, and the limitations imposed on this investigation.

### Discussion of Procedure

The procedure used in evaluating the performance of each unit did not include a study of the effect of temperature on the drift of the units. The response time or the time required for the output of a unit to attain a steady value after a change in the position of the interface was observed but no data recorded. Only static tests were used to evaluate each unit, where the final application of each unit would be to a system whose interface is not only changing but agitated as well. The complete system was not used with any unit; i.e., the differential pressure unit used a system of xylene-water; the electrical capacitance unit used a system of tri-n-butyl phosphate-water; the electrical resistance unit used a system of tri-n-butyl phosphate-water-nitric acid.

## Discussion of Results

The results obtained in this investigation indicate that of the three units tested the differential pressure unit was the most stable and had the best reproducibility of gain. Although the electrical resistance unit possessed the least zero drift, a slight agitation of the interface caused the unit to become erratic.

This investigation evaluated three specific units and not three specific methods. The differential pressure unit as assembled was not sensitive enough to give an output signal from the recorder-controller strong enough to operate a control valve for the system tri-n-butyl phosphate and water.

The response time for the differential pressure and electrical capacitance units was on the order of a few seconds with the electrical capacitance unit responding slightly more rapid than the differential pressure unit. The response time for the electrical resistance unit was approximately one minute with the amount of organic phase increasing and approximately five minutes with the organic phase decreasing.

*Results of Tests of Differential Pressure Unit.* The differential pressure unit has a non-linear calibration curve. The output of the unit increased from 4.50 pounds per square inch, gage, to 6.96 pounds per square inch, gage, when both probes were in the organic phase and the longer probe moved 1/2 inch into the aqueous phase. The output was approximately linear from the above position to a maximum value of 8.52 pounds per square inch, gage. As the position of the interface moved above both probe ends the output decreased to 7.20 pounds per square inch, gage. The output of the unit was expected to be linear with the maximum value occurring with both probes extending into the aqueous phase. A possible explanation for the non-linearity of the output is that the surface tension at the interface caused an additional pressure drop to occur at the interface. By extrapolation of the linear portion and the two end points of the calibration curve the magnitude of this surface phenomena expressed in equivalent output of the unit is approximately two pounds per square inch. This means that as the longer bubbler probe was extended from the organic to the aqueous phase two pounds per square inch was added to the output and when the shorter bubbler probe followed into the aqueous phase two pounds per square inch was subtracted from the output of the unit.

The presence of a maximum in the calibration curve will result in the unit being unstable with the output at or near the maximum.

Results of Tests of Electrical Capacitance Unit. The results of the tests performed with the electrical capacitance unit indicated that this unit was the least desirable of the three units tested. It was necessary to recalibrate the instrument each day. The output of the unit varied as much as 0.35 milliamperes for the same position of the interface. This is equivalent to a 1.5 inch change in the position of the interface.

Results of Tests of Electrical Resistance Unit. The results of the tests performed with the electrical resistance unit indicated a linear response to changes in the position of the interface of 32.0 micromhos per 0.5 inch of change. The average deviation of the output at a constant interface was determined to be equivalent to ± 0.175 inches.

## Recommendations

The recommendations for future study of measuring the position of the organic-aqueous interface in a liquid-liquid extractor using differential pressure, electrical capacitance and electrical resistance are discussed in the following paragraphs.

Changes in the Differential Pressure Unit. The difference in the lengths of the bubble probes in this investigation was 2.5 inches. This difference was proportional to an output of less than half of the working range of the unit. It is recommended that the difference in probe lengths be increased to five inches in order that the full working range of the unit will be utilized.

Changes in the Electrical Capacitance Unit. The electrical capacitance unit used in this investigation lacked sufficient stability to be applied to a control system. It is recommended that a crystal oscillator be used with a null bridge capacitance circuit. The measuring probe should be divided into two equal sections with each section forming a leg of the null bridge. The location of the interface would be between the two sections of the measuring element.

Electrical Resistance Unit. The electrical resistance unit used was observed to have a response time several hundred times longer than the other units. It is recommended that the instrument used with the measuring element be changed and the unit re-evaluated on the basis of sensitivity, stability and response.

Future Study. It is recommended that the above changes in each unit be made and static tests performed under constant temperature conditions. The final evaluation of each unit should be based on the static tests as well as tests performed with the units mounted on an operating liquid-liquid extractor.

## Limitations

The limitations of the investigation of the evaluation of a differential pressure, an electrical capacitance, and an electrical resistance unit for measuring the position of the organic-aqueous interface in a liquid-liquid extractor are presented here.

A system of xylene-distilled water was used with the differential pressure unit.

A system of tri-n-butyl phosphate-distilled water was used with the electrical capacitance unit.

A system of tri-n-butyl phosphate-distilled water-nitric acid was used with the electrical resistance unit.

Each system was contained in a beaker in order to perform static tests.

A series of five tests were performed on each unit over a period of five days to determine the unit's sensitivity by varying the position of the interface in one-half inch intervals.

The units were left operating at a constant interface between each daily test in order to determine the unit's stability.

## V. CONCLUSIONS

The evaluation of the measurement of the position of the interface in the system: xylene-water with a differential pressure unit; tri-n-butyl phosphate-water with an electrical capacitance unit; tri-n-butyl phosphate-water-nitric acid with an electrical resistance unit led to the following conclusions.

1. The change in output for a one-half inch change in the position of the interface for the differential pressure unit was 0.54 pounds per square inch per one-half inch.

2. The output of the differential pressure unit varied 0.23 inches of interface position at a constant interface.

3. The change in output for a one-half inch change in the position of the interface for the electrical resistance unit was 0.115 milliamperes per one-half inch.

4. The output of the electrical capacitance unit varied ± 0.46 inches of interface position at a constant interface.

5. The change in output for a one-half inch change in the position of the interface for the electrical resistance unit was 32.0 micromhos per one-half inch.

6. The output of the electrical resistance unit varied ± 0.175 inches of interface position at a constant interface.

# VI. SUMMARY

The purpose of this investigation was to evaluate a differential pressure, an electrical capacitance, and an electrical resistance measuring element to measure the location of the aqueous-organic interface in a liquid-liquid extractor.

Each element was assembled with a measuring instrument and recorder into a unit. The sensitivity of the differential pressure unit was determined by suspending the measuring element in a system of xylene-distilled water and varying the interface position in increments of one-half inch. The stability of the unit was determined by leaving the interface at a constant position for periods of 24 hours. The electrical capacitance and resistance units used a system of tri-n-butyl phosphate-distilled water and tri-n-butyl phosphate-distilled water-nitric acid, respectively, and were tested for sensitivity and stability in the same manner described for the differential pressure unit.

From the experimental results it was determined that the sensitivity of the differential pressure, electrical capacitance, and electrical resistance unit was 0.54 pounds per square inch per one-half inch change in interface position, 0.115 milliamperes per one-half inch change in interface position, and 32.0 micromhos per one-half inch change in interface position, respectively. The drift of the differential pressure, electrical capacitance and electrical resistance unit at a constant interface was 0.23 inches, ± 0.46 inches, and ± 0.175 inches, respectively.

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