

DESIGN AND CONSTRUCTION OF A LIQUID-LIQUID
EXTRACTOR UTILIZING ULTRASONIC ENERGY

by
elem
Hughey A. Woodle, Jr.
!!!

Thesis submitted to the Graduate Faculty of the
Virginia Polytechnic Institute
in candidacy for the degree of
MASTER OF SCIENCE
in
CHEMICAL ENGINEERING

APPROVED:

APPROVED:

Director of Graduate Studies

Head of Department

Dean of Engineering

Supervisor or Major Professor

June, 1955

Blacksburg, Virginia

TABLE OF CONTENTS

	Page
I. INTRODUCTION	1
II. LITERATURE REVIEW	3
Liquid-liquid Extraction	4
Liquid-liquid Extraction	4
Applications of Liquid-liquid Extraction	6
Mass Transfer in Liquid-liquid Extraction	9
Diffusion Process	9
Two-film Theory	9
Validity of Two-film Theory	10
Film Conditions	11
Interphase Transfer	11
Rate of Transfer	12
Effect of Flow Rate on Transfer	13
Over-all Mass Transfer Coefficient	14
Distribution Equilibria	14
Theoretical Transfer Unit	15
Extraction Efficiency, Height of a Transfer Unit	17
Height Equivalent to a Theoretical Stage	18

	Page
Liquid-liquid Extraction Systems of Operation	20
Single Contact	20
Multiple Contact, Cocurrent	21
Multiple Contact, Continuous Countercurrent	21
Systems of Operation by Solvent Classification	24
Calculation Methods	26
Design of Extraction Equipment	27
Equipment Classification	28
Mixer-settler	28
Spray Column	31
Packed Column	31
Perforated and Sieve Plate Columns	31
Baffle Columns	35
Pulse Columns	35
Centrifugal Extractor	38
Ultrasonics	40
Ultrasonic Generators	40
Piezoelectric Generators	41
Piezoelectric Materials	43

	Page
Effects of Ultrasonic Waves	48
Cavitation	48
Conditions Favorable for Cavitation	49
Mechanical Effects of Cavitation	49
Thermal Effects of Cavitation	50
Chemical Effects of Cavitation	50
Electrical Effects of Cavitation	51
Applications of Ultrasonic Waves	52
Ultrasonic Testing	52
Ultrasonic Cleaning	53
Ultrasonic Emulsification	53
Ultrasonic Communication	54
Miscellaneous Applications	54
Application of Ultrasonics to Liquid-liquid Extraction	55
Liquid-liquid Extraction	55
Liquid Mixing	56
Extraction Contactor	56
Ultrasonic Dispersion of Liquid Droplets	57
Ultrasonic Stirring Effects	57
Ultrasonic Thermal Effects	57

	Page
Previous Applications	58
Postulation	59
III. EXPERIMENTAL	60
Purpose of Investigation	60
Plan of Investigation	61
Materials	62
Equipment	62
Selection of Special Equipment Pieces	62
Equipment Sub-Assemblies	64
Design and Construction Procedure	91
Reactor Design	91
Operational Procedure	105
Safety Measures	105
Preparation for Start-up	106
Electrical Power Switches	107
Filling Feed and Solvent Tanks	107
Operation of Constant Temperature Bath	108
Operation of Cooling Oil System	108
Feed and Solvent Circulation	108
Regulation of Air Pressure	109
Extract and Raffinate Tanks	109

	Page
Generator Warm-up	110
Feed and Solvent Flow Rate Control	110
Interfacial Control	111
Sample Collection	111
Power to Transducer	111
Temperature Measurements	112
Electrical Power Measurements	112
General Operation	112
Level of Feed and Solvent Tanks	112
Shut-down Procedure	113
Emptying Extract and Raffinate Tanks	113
Shut-down and Checking	114
Extractor Evaluation	114
System Employed for Evaluation	114
Make-up and Handling of Feed and Solvent	114
Solvent to Feed Ratio	116
Flow Rates	117
Number of Tests Made	117
Insonation Intensity	117
Sample Collection	117
Analysis of Samples	119

	Page
Calibration of Flow Meters	120
Calibration of Thermocouples	120
Sample Calculations	131
Calibration of Flow Meters	131
Calibration of Stage Efficiency	137
IV. DISCUSSION	138
Discussion of Design and Construction	138
Constant Temperature Circulating Lines for Feed and Solvent	138
Electrical Wiring	139
Discussion of the Reactor	139
Design	139
Construction	139
Liquid Mixing in the Reactor	140
Discussion of Extractor Operation	144
Collection of Samples	144
Electrical Measurements	144
Discussion of Extraction Results	145
Variance of Stage Efficiency with Flow Rates and Insonation	145
Content of Extract Samples	145
Recommendations	147
Limitations	149

	Page
V. CONCLUSIONS	151
VI. SUMMARY	152
VII. BIBLIOGRAPHY	154
VIII. ACKNOWLEDGMENTS	162
IX. VITA	165

TABLES

		Page
Table	I. Applications of Various Non-mechanical Separation Methods	5
Table	II. Major Commercial Applications of Liquid-liquid Extraction Processes . . .	8
Table	III. Classification of Extraction Equipment	29
Table	IV. Comparative Chart of Physical Properties of Piezoelectric Crystals	44
Table	V. Construction Materials and Items of Apparatus Employed in the Design and Construction of a Liquid-Liquid Extractor	63
Table	VI. Operational Data and Results for Extractor Evaluation Test	118
Table	VII. Refractive Index Data for Acetone-Water-1,1,2-Trichloroethane Mixtures at 25 °C	121
Table	VIII. Calibration of "TRI-FLAT" Flow Meter, Serial X2-1344/2, for 1,1,2-Trichloroethane at 25 °C	124
Table	IX. Calibration of "TRI-FLAT" Flow Meter, Serial X2-1344/4, for 1,1,2-Trichloroethane Rich Extract Phase at 25 °C	126
Table	X. Calibration of "TRI-FLAT" Flow Meters, Serials X2-1344/1 and 3, for Feed and Water Rich Raffinate Phase at 25 °C	128
Table	XI. Calibration Data for Extract Flow Meter	136

FIGURES

	Page
Figure 1. Distribution of Acetone Between Water and 1,1,2-Trichloroethane at 25 °C	16
Figure 2. Illustrated Difference Between H.T.U. and H.E.T.S.	19
Figure 3. Single Contact Extraction	22
Figure 4. Co-current Multiple Contact Extraction	23
Figure 5. Continuous Countercurrent Extraction	25
Figure 6. Two-stage Mixer-settler Extractor	30
Figure 7. Simplified Spray Column Extractor	32
Figure 8. Packed Column Extractor	33
Figure 9. Perforated Plate Column Extractor	34
Figure 10. Baffle Column Extractor	36
Figure 11. Pulse Column Extractor	37
Figure 12. Luwesta (Centrifugal) Extractor	39
Figure 13. Interaction Between Force and Field in Piezoelectric Crystals	42
Figure 14. Various Available Shapes of Barium Titanate Crystals	46
Figure 15. Spectrum of the Sounds that Can Be Produced by Means of Piezoelectric Generators	47
Figure 16. HYPERSONIC Generator	66

	Page
Figure 17. HYPERSONIC Generator, with Tuning Drawer Removed	67
Figure 18. Unmounted Transducer	68
Figure 19. Transducer Mounted in Metal Housing	69
Figure 20. Electrical Conditions for Full Excitation of Bowl-shaped Barium Titanate Ceramic Transducer	70
Figure 21. Modified Reactor Design for Ultrasonic Extraction	72
Figure 22. Glass Reactor of Modified Design	73
Figure 23. Drilling Plan for Thermocouple Panel for Ultrasonic Extraction	74
Figure 24. Drilling Plan for Electric Panel for Ultrasonic Extraction	75
Figure 25. Drilling Plan for Hydraulic Panel for Ultrasonic Extraction	76
Figure 26. Control Panel Frame for Ultrasonic Extraction	77
Figure 27. Tank Racks for Ultrasonic Extraction	79
Figure 28. Completed Control Panel and Adjacent Equipment Sub-Assemblies	81
Figure 29. Schematic Flow Diagram for Ultrasonic Extraction	82
Figure 30. Tubing Sketch of Feed and Solvent Tanks for Ultrasonic Extraction	83
Figure 31. Tubing Sketch of Extract and Raffinate Tanks for Ultrasonic Extraction	84
Figure 32. Tubing Sketch of Reactor and Settling Column for Ultrasonic Extraction	85

	Page
Figure 33. Sight Gage for Ultrasonic Extraction	86
Figure 34. Settling Column for Ultrasonic Extraction	87
Figure 35. Mounting of Sight Gages for Feed and Solvent Tanks	88
Figure 36. Installed Settling Column	89
Figure 37. Sketch of Thermocouple Well Construction for Ultrasonic Extraction	90
Figure 38. Arrangement of Rubicon Potentiometer in Relation to Control Panel	92
Figure 39. Panel Wiring Diagram for Ultrasonic Extraction	93
Figure 40. Arrangement of Constant Temperature Bath, Cooling Coils, and Circulating Pumps	94
Figure 41. Air Pressure Control Panel	95
Figure 42. Reactor Design for Ultrasonic Extraction	97
Figure 43. Plastic Reactor of Original Design	99
Figure 44. Example of Bond Between Nickel Sheet and Glass Edge of Reactor	102
Figure 45. Metal Reactor Employed in Evaluation Test	104
Figure 46. Liquid Equilibria of System Acetone- Water-1,1,2-Trichloroethane	115
Figure 47. Acetone Concentration of 1,1,2-Trichloroethane Phase for System Acetone-Water- 1,1,2-Trichloroethane	122

	Page
Figure 48. Acetone Concentration of Aqueous Phase for System Acetone-Water-1,1,2-Trichloroethane	123
Figure 49. Flow Meter Calibration for Solvent	125
Figure 50. Flow Meter Calibration for Extract	127
Figure 51. Flow Meter Calibration for Feed and Raffinate	129
Figure 52. Thermocouple Calibration	130
Figure 53. Ball Float Characteristic Curve X-1006	135
Figure 54. Reactor in Operation without Insonation	141
Figure 55. Reactor in Operation with Insonation	143

I. INTRODUCTION

Extraction is the separation of a mixture into its components by the action of a solvent that is selective for one of its components. This unit operation, usually carried out as an isothermal process, is employed when it is economically infeasible to perform separation of a mixture by such methods as distillation and evaporation, or when the physical properties of the system (such as sensitivity of components to thermal increases or components having relatively the same volatilities) would not allow the employment of distillation or evaporation as separation methods. Extraction may be divided into two main types, i. e. liquid-solid and liquid-liquid.

Liquid-liquid extraction is the treatment of a liquid (carrier solvent) containing a solute with a solvent that is more selective for the solute than the original. Liquid-liquid extraction involves: (1) mixing of the solvent and feed mixture, (2) separation of the resulting phases, and (3) removal and recovery of solvent from each phase, usually accomplished by distillation. One criteria for selection of solvents for a system is that they should be immiscible, or at the most only partially miscible, with each other to allow separation of the mixture into two phases after contacting and settling of the three components takes place.

Rate of transfer of a solute through an interface is directly proportional to: (1) conductance, (2) area of the interface, and (3) concentration gradient across the interface. In mass transfer, conductance is inversely related to the resistance offered by an interface. Total mass transfer through an interface between two given liquids may be increased by reducing the interfacial resistance or by increasing the total interfacial area between the two liquids. It has been demonstrated in numerous cases that ultrasonic waves produce selective interfacial microagitation, gross stirring, and thermal effects in multi-phase liquid systems.

The purpose of this investigation was to design and construct a laboratory scale liquid-liquid extraction unit incorporating an ultrasonic field with equipment specifications as: (1) insonation frequency of 400 kilocycles per second, (2) insonation intensities corresponding to plate currents of 0 to 200 milliamperes, and (3) flow rate of solvent and feed through reactor ranging approximately from one-half to ten pounds per minute, in varying solvent-to-feed ratios.

II. LITERATURE REVIEW

The ultimate use of the equipment and knowledge gained by this investigation will be to facilitate the study of the effect of ultrasonic energy on mass transfer in multiple-component, two-phase liquid systems. Three important physical operations that are used or recognized by chemical engineers are to be combined in this study to give a better understanding of the interrelation of the three. These operations are liquid-liquid extraction, mass transfer, and generation and application of ultrasonic waves and energy.

For the purpose of discussion, the literature review for this investigation has been divided into three main sections: (1) unit operation of liquid-liquid extraction and its inherent mass transfer, (2) ultrasonics, and (3) application of ultrasonics to liquid-liquid extraction.

Liquid-liquid Extraction

Separation of the components of a liquid solution by the unit operation of liquid-liquid extraction has gained considerable importance during recent years in the chemical and oil industry in general. Study of this particular operation will be divided into four general categories, each with related topics: (1) over-all field and varied uses, (2) mass transfer occurring in liquid-liquid extraction, (3) different extraction systems of operation, and (4) extraction process equipment.

Liquid-liquid Extraction. A common problem facing most chemical engineers in today's complex chemical industry is the separation of the components of a liquid solution. Extraction is one of several methods used in this type of separation. Others include evaporation, fractional crystallization, and distillation. Liquid-liquid extraction is usually considered as a means of performing the desired separation when⁽⁹⁾; (1) boiling points of liquid mixture components are within a few degrees of each other, (2) one or more compounds are sensitive to heat, and (3) relatively small amounts of desired product in solution would necessitate large heat requirements for other methods of separation. Table I indicates the

TABLE I
Applications of Various Non-mechanical
Separation Methods

Method	Dissolved Solids		Liquid Solutions			Heat-Sensitive Materials
	Single	Two or More	Volatile Fraction Minor	Volatile Fraction Major	Close-Boiling Fractions	
Crystallization	Na ₂ SO ₄ · 10 H ₂ O	KCl from NaCl	-----	-----	-----	Quinine from alkaloids
Evaporation	Sugar, NaCl	NaCl from NaOH	Concentration of spent H ₂ SO ₄ , butanol from dibutyl phthalate	Flashing of propane from propane-fat extract	-----	Evaporated milk freeze drying; fruit juice concentration
Adsorption	Antibiotics	Decolorization of sugar	-----	Drying of benzene, gasoline, etc., with silica gel and alumina gel	Recovery of aromatics from hydrocarbons	Antibiotics
Ion exchange	-----	Separation of heavy metals, water softening	-----	-----	-----	-----
Thermal diffusion	-----	-----	-----	-----	Tall oil, separation of high-octane gasoline components	-----
Ordinary distillation	-----	-----	Stabilization of gasoline, recovery of organic solvents, stripping	Gasoline rerunning	Heavy water	Molecular distillation, vitamins
Azeotropic or extractive distillation	-----	-----	Absolute alcohol using bichloroethylene or ether	-----	Toluene from hydroformate, butadiene recovery	-----
Liquid-liquid extraction	Phenol from water using benzene	NaCl from NaOH using NH ₃ , chromate from vanadate salts using ketones	Aniline from water using nitrobenzene	Acetic acid from water	Lube oil refining	Antibiotics and vitamins

Von Berg, R. L. and H. F. Wiegandt: *Liquid-liquid Extraction*, Chem. Engr., 59, 6-189 (1952).

appropriate employment of the various unit operations which involve phase changes or mass transfer between phases⁽⁸⁵⁾.

Liquid-liquid extraction must include the following sequence of steps⁽⁸⁵⁾: (1) production of an intimate contact of feed mixture with solvent, (2) separation into two liquid phases, and (3) recovery of solute from extract phase and solvent from both phases. Depending on the particular type of equipment employed, steps (1) and (2) may be carried out simultaneously in the same extractor or separately in different pieces of equipment. Step (3) is carried out individually, usually by distillation.

Liquid-liquid extraction as a unit operation has increased in popularity in recent years in many different fields and processes, different reasons being responsible in each case. Yearly reviews on unit operations, including liquid-liquid extraction, are presented in "Industrial and Engineering Chemistry."

Applications of Liquid-liquid Extraction. The petroleum industry has the largest number of operations involving liquid-liquid extraction, on purely a volume basis⁽⁸⁵⁾. Important applications of this unit operation, on a much smaller volume basis than employed in the petroleum industry, are in the fields of pharmaceuticals, vitamins, and antibiotics.

In general, extraction is thought of as being applicable to the separation of molecules of different chemical types, while in contrast distillation is carried out to accomplish the separation of molecules of different size or molecular weight⁽²⁴⁾.

Extraction methods have proven valuable for removal of trace amounts of impurities and colored bodies, for recovery and purification of wide variety of organic compounds and pharmaceuticals, and in treating liquors whose solid content create difficulty in heat transfer in the desired separation by vaporization methods.

The major commercial applications of liquid-liquid extraction processes are listed⁽⁴⁶⁾ in Table II. Varied examples of this increasingly popular unit operation are: (1) extraction of chlorophyll from alfalfa⁽³⁶⁾, (2) separation of chromium and vanadium salts⁽⁸⁷⁾, and (3) hydrocarbon separation by use of liquid carbon dioxide⁽²⁷⁾.

TABLE II

Major Commercial Applications of
Liquid-liquid Extraction Processes

- I. Petroleum Refining
 - A. Separating high purity products
 - Toluene
 - Butadiene
 - Olefins
 - B. Separating one or more groups of compounds from cuts of wide boiling range
 - 1. Treatment of kerosene, Edeleanu (93)
 - 2. Lubricating oils treated with selected solvents
 - Edeleanu (liquid SO₂)
 - Chlorex [bis(2-chloroethyl)ether]
 - Duo-Sol
 - Furfural
 - Nitrobenzene
 - Phenol
 - C. Dewaxing, deasphalting, and decarbonizing operations
- II. Extraction of Acetic Acid
 - A. Wood distillation
 - B. Recovery of acetic acid from dilute solutions from cellulose acetate, etc.
- III. Phenol Recovery
 - A. Raschig process, primary phenol production
 - B. Gas works liquids, recovery and by-product phenols
 - C. Recovery of phenol from a wide variety of solutions
- IV. Chemical Processes with Liquid-Liquid Contacting
 - A. Nitration { Here the value of countercurrent operations may be
 - B. Sulfonation { questioned because of the danger of over-reaction
- V. Vitamins and Antibiotics
- VI. Vegetable Oil Refining
- VII. Fischer-Tropsch Synthesis of Liquid Fuels
 - A. Separation of water-soluble by-products
 - B. Separation of oil-soluble oxygenated by-products

Morello, V. S. and N. Poffenberger: Commercial Extraction Equipment, Ind. Eng. Chem., 42, 1021 (1950).

Mass Transfer in Liquid-liquid Extraction

Mass transfer is the movement of one or more components between phases of a system⁽⁶⁾. Examples of mass transfer are the absorption of a volatile component from a vapor phase, the extraction of a component from a solid or liquid in solution by a solvent, and separation of volatile components of a liquid by distillation. Solute transfer across a liquid-liquid interface is a particular case of a general problem, that of reactions occurring at or across phase boundaries⁽³⁹⁾.

Diffusion Process. Moving immiscible liquids are brought into contact in extraction operations for the purpose of causing diffusion of a solute from one liquid to the other across the phase boundary⁽⁷⁵⁾. In this diffusion process the solute passes through various portions of the fluid that are in viscous or turbulent flow.

Two-film Theory. Experimental work conducted on kinetics of mass transfer across liquid-liquid boundaries has been less than that carried on for gas-liquid and solid-liquid systems. The two-film theory developed by Lewis and Whitman⁽⁴¹⁾ for gas absorption has been generally extended to liquid-liquid systems.

Assumptions made in the case of the two-film theory may be summarized⁽⁵⁾ as:

1. "On either side of the interface, between the two moving immiscible liquids in contact, there exists a relatively stationary fluid film.

2. The disturbed main body of each fluid is in comparatively violent motion and solute concentration gradients therein are rapidly equalized due to mixing by fluid motion.

3. Transfer of solute across the interface does not take place by diffusion. Equilibrium is set up here and the transfer of solute involved is the result of the prevailing equilibrium conditions. This reaction is so rapid that it is not a controlling factor in the process.

4. Passage of the solute through the stationary films can occur only by diffusion and concentration gradients are set up in consequence through each stationary film.

5. The only controlling factor in the process is the rate of solute diffusion across the interfacial stationary films."

Validity of Two-film Theory. The two-film theory as described, while generally applied to liquid-liquid systems, is not always accepted as the proper approach to the problem. Investigations have shown that under conditions of high turbulence, eddy and not molecular diffusion may be the mechanism

of transport to the interface in liquid-liquid systems⁽³⁹⁾. It has also been found⁽⁴⁰⁾ that transfer of many organic compounds across liquid-liquid interfaces sets up interfacial turbulence even in unstirred systems, for example, acetone transferring between ethyl acetate and water. It is the opinion of Lewis and Pratt^(39,40) that unless turbulence is known to be absent at the interface, the two-film concept should not be applied, for even if empirical correlations can be obtained with one system in a particular extractor the data cannot be transferred to other systems of other equipment.

Film Conditions. In a study of mobile and rigid films⁽³⁹⁾ it has been suggested that while in general mobile films have no effect on transfer rates, rigid films reduce transfer possibly by damping interfacial tension.

Interphase Transfer. The over-all rate of reaction at phase boundaries may be thought of as being controlled by the following factors⁽³⁴⁾:

1. Mass transfer on the continuous-phase side of the interface.
2. Chemical reaction at the interface or mass transfer through it.
3. Mass transfer on the dispersed-phase side of the interface.

This classification leads to three possible types of reactions⁽³⁹⁾;

1. Transport-controlled, where the rate of reaction is much faster than the rate of mass transport to and from the boundary.

2. Reaction-controlled, where the rate of reaction is much slower than the transport rate.

3. Complex, where the two rates are of the same order and both factors are therefore important.

Rate of Transfer. It has been proposed⁽⁸³⁾ that rate of transfer of a solute between two immiscible liquids is controlled by laws of diffusion. This would indicate that the rate of diffusion is proportional to the concentration gradient and surface area across which the diffusion is taking place. Potential for the diffusion is determined by the concentration of the diffusing material in the bulk of the first fluid, and the solute concentration which would be in equilibrium with the bulk of the second liquid phase. If the two concentrations are equal, no diffusion or mass transfer will take place⁽⁶⁰⁾.

Rate of solute transfer from diluent to solvent layer is directly proportional to the area of contact and degree of agitation⁽²³⁾. Prediction of mass transfer behavior for agitated heterogeneous systems may be made by a method devised

by Vermeulen, Williams, and Langlois⁽⁸⁴⁾. Their method of determining interfacial area and mean drop diameter by photo-electric cell measurements offers a practical index to the effectiveness of particular methods of mixing immiscible liquids.

Rate of transfer may also be expressed as:

$$\frac{N}{\theta} = K A \Delta C_{1m} \quad (1)$$

where:

$$\frac{N}{\theta} = \begin{array}{l} \text{lb moles solute transferred per unit time,} \\ \text{lb moles/hr} \end{array}$$

$$K = \begin{array}{l} \text{over-all mass transfer coefficient,} \\ \text{lb moles/sq ft - hr - unit conc diff} \end{array}$$

$$A = \text{total interfacial area of contact, sq ft}$$

$$\Delta C_{1m} = \begin{array}{l} \text{logarithmic mean concentration difference} \\ \text{at either end of extractor based on} \\ \text{inorganic phase.} \end{array}$$

Effect of Flow Rate on Transfer. For low flow rates, transport to the interface takes place by molecular diffusion⁽³⁹⁾. With an increase in turbulence, an increased transfer rate will be evident as the diffusional layer thicknesses will be decreased. Disruption of the diffusional layer by increased turbulence will cause transfer to the interface by eddy and not molecular diffusion, with a slight increase in flow rate having a large effect on the transfer rate.

Over-all Mass Transfer Coefficient. The over-all coefficient is considered as the reciprocal of the total resistance to mass transfer from the body of one phase to the other⁽⁶⁰⁾. Over-all resistance is assumed to be composed of two resistances in series, resistance to the two films at the interface being considered appreciable. In terms of individual film transfer coefficients, the over-all coefficient has been shown to be:

$$1/K_w = 1/k_w + H/k_s \quad (2)$$

where:

K_w = over-all coefficient based on water phase,
lb mols/hr - sq ft - lb mol/cu ft

k_w = individual coefficient of water phase,
lb mols/hr - sq ft - lb mol/cu ft

k_s = individual coefficient of solvent phase,
lb mols/hr - sq ft - lb mol/cu ft

H = distribution coefficient, dimensionless.

Distribution Equilibrium. The laws of diffusion and distribution equilibria govern the transfer of a solute from the carrier solvent to the extracting solvent in an extraction operation.

If to a liquid system of two phases there is added an amount of a third liquid or solute that is soluble in both

phases, this added substance will be distributed between the two phases in a definite ratio at a given temperature ⁽²⁹⁾.

In equation form, this distribution law may be stated as:

$$C_1/C_2 = H \quad (3)$$

where:

C_1 = solute concentration in first liquid phase,
mol fraction

C_2 = solute concentration in second liquid phase,
mol fraction

H = distribution coefficient, dimensionless.

For ideal situations, this law gives a linear relationship. For most actual extraction operations, however, the linear relationship holds only for very dilute solutions ⁽²⁵⁾.

The relation of solute distribution between the carrier solvent and proposed extracting solvent is an important factor in choice of the solvent most advantageous for any particular extraction operation. Figure 1 illustrates the distribution of a solute between two liquid phases.

Theoretical Transfer Unit. In order to evaluate continuous extraction equipment, there is need of some standard to which the equipment may be compared. To get away from the ideal stage of single contact, Colburn ^(17,18) proposed the idea of transfer units. This transfer unit is the concentration

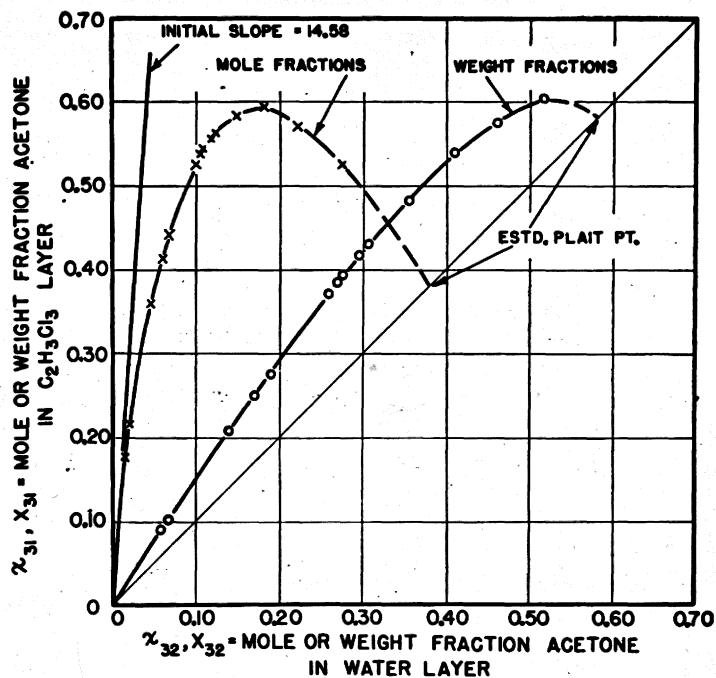


Figure 1. Distribution of Acetone Between Water
and 1,1,2-Trichloroethane at 25 °C

Treybal, R. E., L. D. Weber, and J. F. Daley: The System
Acetone-Water-1,1,2-Trichloroethane, *Ind. Eng. Chem.*, **38**,
817 (1946).

change experienced per unit of concentration causing the change.

In equation form, the transfer unit may be expressed as⁽³²⁾:

$$N = \int_{y_2}^{y_1} \frac{dy}{y^x - y} \quad (4)$$

where:

N = number of mass transfer units, dimensionless

y = concentration of solute in one phase, mol fraction

y^x = concentration of solute in extract phase that is in equilibrium with the concentration of solute in raffinate phase at the same point, mol fraction

y_1 = concentration of solute in the extract phase at the feed end of the column, mol fraction

y_2 = concentration of solute in the extract phase at the solvent end of the column, mol fraction.

Extraction Efficiency, Height of a Transfer Unit. For extraction operations in general, the stage efficiency may be thought of as the number of theoretical stages required for the extraction divided by the number of actual stages required to perform the desired extraction. For use in continuous extractors, Colburn^(17,18) has suggested that a unit length of a column be defined as the continuous countercurrent

equivalent to a theoretical transfer stage. A measure of the column efficiency is the actual height of a particular column divided by the number of transfer units. This may be expressed as:

$$\text{H.T.U.} = \frac{H}{N} \quad (5)$$

where:

H.T.U. = height of a transfer unit, ft

H = height of column, ft

N = number of theoretical transfer units,
dimensionless.

From this, the H.T.U. value for a column can be used as means of evaluating the column efficiency.

Height Equivalent to a Theoretical Stage. Stagewise columns may be compared by means of the height equivalent to a theoretical stage⁽²⁶⁾, or H.E.T.S. The H.E.T.S. may be defined as the total actual height of the column divided by the number of theoretical stages. H.E.T.S. is analogous to H.T.U. but is used for stagewise extraction, while H.T.U. is used for continuous counter-current extraction. Figure 2 illustrates the difference between these two terms, H.T.U. and H.E.T.S.

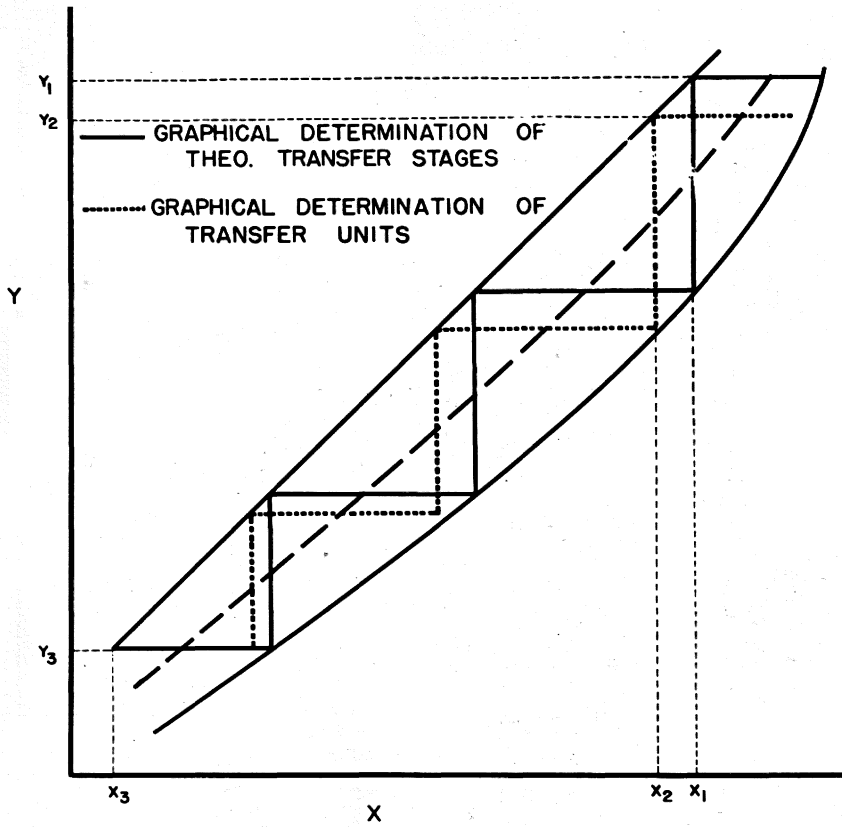


Figure 2. Illustrated Difference Between H.T.U. and H.E.T.S.

Liquid-liquid Extraction Systems of Operation

The operation of extraction may be divided into four basic systems with regard to contact between feed and solvent. These four systems are⁽⁹⁾: (1) single contact, (2) simple multiple contact, (3) countercurrent multiple contact, and (4) continuous countercurrent contact with reflux. A further breakdown of these four systems has been proposed by Elgin⁽²³⁾ as:

I. Single Contact

II. Multiple Contact

A. Cocurrent

1. Multistage

2. Differential

B. Continuous Countercurrent

1. With Reflux

2. Without Reflux

Single Contact. This is the simplest type of extraction, solvent and feed being brought together for a single batch extraction. Final extract and raffinate from a single contact operation are essentially in equilibrium with each other, the amount of solute which may be extracted with a given amount of solvent being limited⁽⁶²⁾. This type, typical flow arrangement

being shown in Figure 3, is seldom employed for large scale industrial work.

Multiple Contact, Cocurrent. Multistage extraction of this classification indicates feed solution being treated with successive portions of fresh solvent, each portion performing an ideal contact. The mixture formed in each stage is separated, with raffinate going to the next stage to be contacted with fresh solvent⁽⁹⁾. In differential cocurrent contact, solvent is added in differential portions, with extract being removed as fast as it is formed. Illustration of flow arrangement for this type extraction is made in Figure 4.

Multiple Contact, Continuous Countercurrent. True countercurrent extraction would require the two liquid phases in contact with each other to pass continuously in opposite directions. Reflux may be used in extraction operations in a manner similar to that of distillation⁽⁶²⁾. Reflux is provided by removing solvent from the extract leaving the column so as to form a heavy extract phase saturated with solvent, part being returned to the column, and the remainder being withdrawn as extract product. This procedure gives an extract much richer than an extract of composition in equilibrium with the feed.

Two major points of interest concerning the use of reflux in extraction cannot be overestimated from the standpoint of

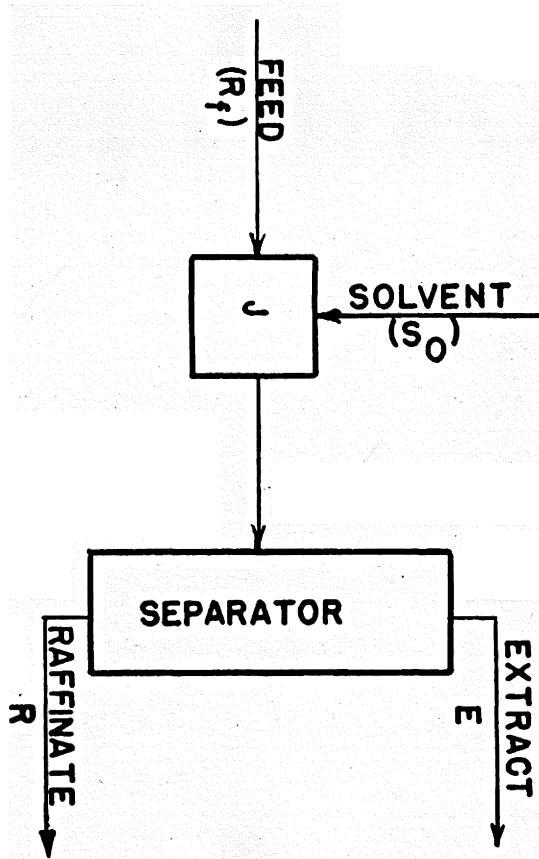


Figure 3. Single Contact Extraction

Bull, F. W. and G. J. Coli: Graphical Methods As Applied to Extraction Problems, Va. Poly. Inst., Blacksburg, Va., Engr. Exp. Sta. Bull., No 72 (1949).

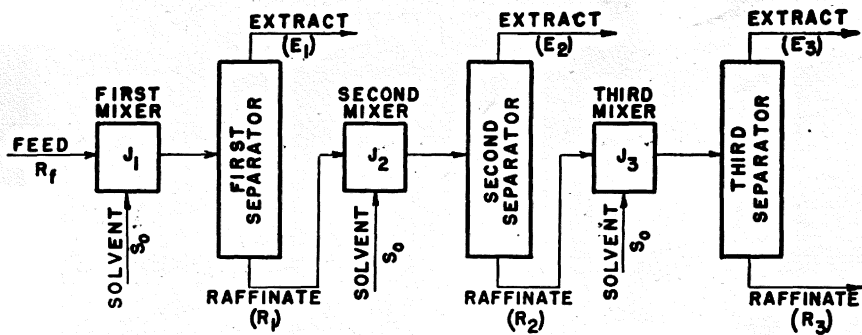


Figure 4. Co-current Multiple Contact Extraction

Bull, F. W. and G. J. Coli: Graphical Methods As Applied to Extraction Problems, Va. Poly. Inst., Blacksburg, Va., Engr. Exp. Sta. Bull., No 72, (1949).

practical applicability⁽⁸²⁾: (1) through the use of proper reflux it is possible, with a reasonable number of stages, to make separations which would otherwise be impossible even by the use of an infinite number of stages, and (2) in order to separate a constituent from a mixture, the solvent used does not necessarily have to be selective toward that constituent, if the right type reflux is employed. The fact must not be overlooked that reflux advantages may be had only at the expense of more solvent or energy per unit charging stock and less throughput of product. An example of flow arrangement for continuous countercurrent extraction is shown in Figure 5.

Systems of Operation by Solvent Classification. Another convenient method of classifying extraction systems depends upon the nature of the solvent and physical arrangement of the apparatus employed⁽⁷⁶⁾, each division having both stagewise and continuous contact, depending upon desired conditions of operation.

1. Single solvent systems, including all those which consist of, or which may be reduced to the equivalent of three components, the two to be separated and the solvent.

2. Mixed solvents, arrangements employing a solvent solution consisting of at least two components.

3. Double solvents (fractional extraction), arrangements where mixture to be separated is distributed between two immiscible solvents, the system containing at least four components.

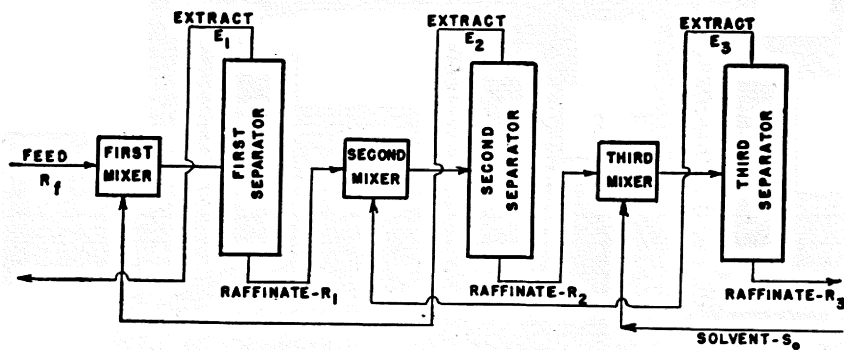


Figure 5. Continuous Countercurrent Extraction

Bull, F. W. and G. J. Coli: Graphical Methods as Applied to Extraction Problems, Va. Poly. Inst., Blacksburg, Va., Engr. Expt. Sta. Bull., No 72 (1949).

Most liquid-liquid extraction operations are of the first classification, or single solvent systems. In special cases where it is desired to bring about modification of single solvent effects such as improvement of selectivity and lowering of solvent freezing point, a mixed solvent or solvent solution is employed as the extracting medium⁽⁷⁷⁾.

Calculation Methods. Algebraic methods of calculating the number of theoretical number of stages required to achieve a certain degree of extraction have been proposed⁽³⁴⁾. However, because of difficulty in application of the algebraic methods, graphical methods using the principle of these ideas have come into prominence.

The method of Ponchon and Saveritt has been modified for utilization in extraction⁽⁴³⁾, making use of the solvent content-concentration diagram for a ternary system. This particular method is covered by Brown⁽⁶⁾, Perry⁽²⁵⁾, and Bull and Coli⁽⁹⁾.

Material balances carried out on the triangular phase diagram gave a graphical solution to countercurrent stage-wise extraction operations. Proper employment of this type phase diagram, giving necessary relations between composition and quantity of extract layer leaving any stage and those of raffinate layer leaving the same stage, is covered by Hunter and Nash⁽³⁴⁾, Perry⁽²⁵⁾, and Bull and Coli⁽⁹⁾.

Saal and Van Dyck⁽⁵⁸⁾ emphasize the analogies between distillation and extraction in their main forms and also mention the possibility of applying to extraction a graphical method of computation similar to that developed by McCabe and Thiele. Explanation and application of this method is covered by Brown⁽⁶⁾ and Bull and Coli⁽⁹⁾.

For the exact procedure of the various graphical methods of calculation, reference is made to the authors cited, particularly to Perry⁽²⁵⁾, who gives a very good discussion and bibliography for the various methods.

Design of Extraction Equipment

Operations involving physical separation are essentially ones in which power is expended to make a separation⁽²⁾. Extraction is one of several operations in which the power required is expended to a reasonable degree of over-all efficiency, power requirements in extraction being largely the heat required for recovery of solvent⁽⁴⁶⁾. As operating costs of the actual extractor itself may be considered small in comparison to total cost of operating the entire system, including power, a design of extractor that will give more ease in operating and less maintenance trouble is usually the one chosen.

In a continuous liquid-liquid extraction operation, the function of the contactor is to provide a large interfacial area for mass transfer between the two liquid phases⁽²⁰⁾. Production of this interface in a manner best suited for the particular system in operation determines many of the special features found in the different extractor designs.

Commercial extraction equipment has not been generally standardized, developing over a period of years as found best suitable for particular situations, mixing and contacting of the two phases first and then separation of the phases being the basic duty of equipment in the over-all operation.

Equipment Classification. A major classification of extraction equipment may be based on the method used for separating the two phases, gravity or centrifugal force, most commercial extractors being of the gravity type⁽⁴⁶⁾. A general classification of types of extraction equipment is given in Table III. Illustrations of typical extractor types discussed are shown in Figures 6 through 12.

Mixer-settler. The mixer-settler type of extractor, using a series of vessels, is the oldest extraction device for both batch and continuous operation⁽⁸⁶⁾. This type extractor consists of a mixing chamber followed by a settling chamber for each stage of extractor. The mixer, only a centrifugal pump

TABLE III

Classification of Extraction Equipment

I. Two Phases Separated by Gravity

A. Contact made through one phase as a film

1. Extended surfaces
2. Packed towers
Large packing grids

B. Contact made with one phase dispersed as droplets

- | | |
|--|---|
| <p>B₁. No additional power used to maintain dispersion or redispersion</p> <ol style="list-style-type: none">1. Spray tower2. Baffle tower3. Perforated plate4. Bubble cap5. Packed tower | <p>B₂. Extra power used to redisperse or maintain dispersion</p> <ol style="list-style-type: none">1. Mixer and settler2. Baffle tower with movable members |
|--|---|

II. Two Phases Separated by Centrifugal Force

Morello, V. S. and N. Poffenberger: Commercial Extraction Equipment, Ind. Eng. Chem., 42, 1021 (1950).

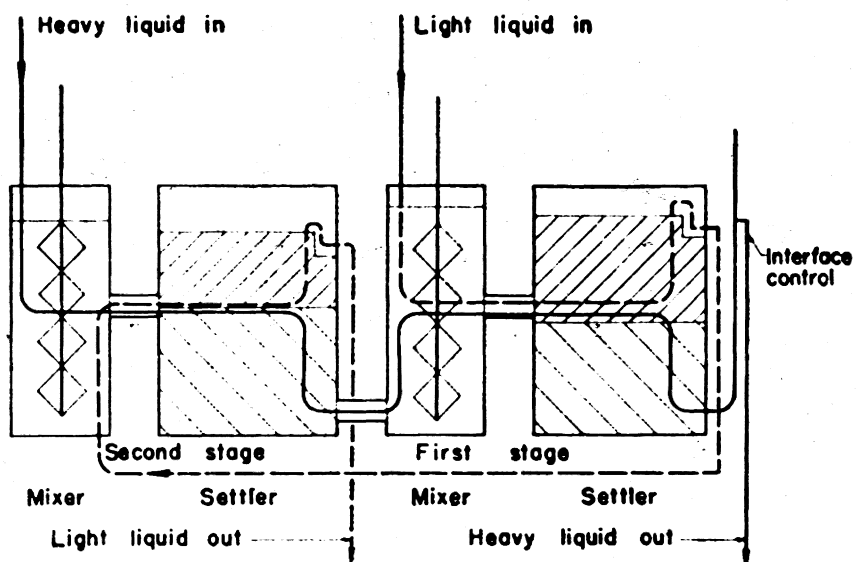


Figure 6. Two-stage Mixer-settler Extractor

Morello, V. S. and H. Poffenberger: Commercial Extraction Equipment, Ind. Eng. Chem., 42, 1021 (1950).

at times, is usually a chamber having a propeller or turbine agitator and baffles. Several variations of this type extractor have developed over a period of years, two recent developments being one reported by National Research Corporation⁽⁴⁸⁾ and a "pump-mix" settler developed by Coplan and Davidson⁽²⁰⁾.

Spray Column. A spray column is a vertical cylinder through which one liquid flows as the continuous phase, with the second liquid dispersed in the first⁽⁸⁵⁾. Density relation of the two liquids determines direction of flow. Spray columns are the simplest type of extraction equipment in construction and operation, having low cost, high capacity, and ease in cleaning. This column is not used to any large extent in commercial extraction operations; however, one of the few commercial applications is that employed in the Columbia alkali process⁽⁸⁰⁾ for extracting sodium chloride and sodium chlorate to make "rayon grade" caustic soda from electrolytic soda.

Packed Column. This extractor is very similar to the spray column; a solid material such as Raschig rings or Berl saddles is employed as packing to help increase dispersion. Packed towers are used extensively in many chemical and petroleum operations⁽⁸⁵⁾.

Perforated and Sieve Plate Columns. The principle of redistributing the dispersed phase into droplets a number of

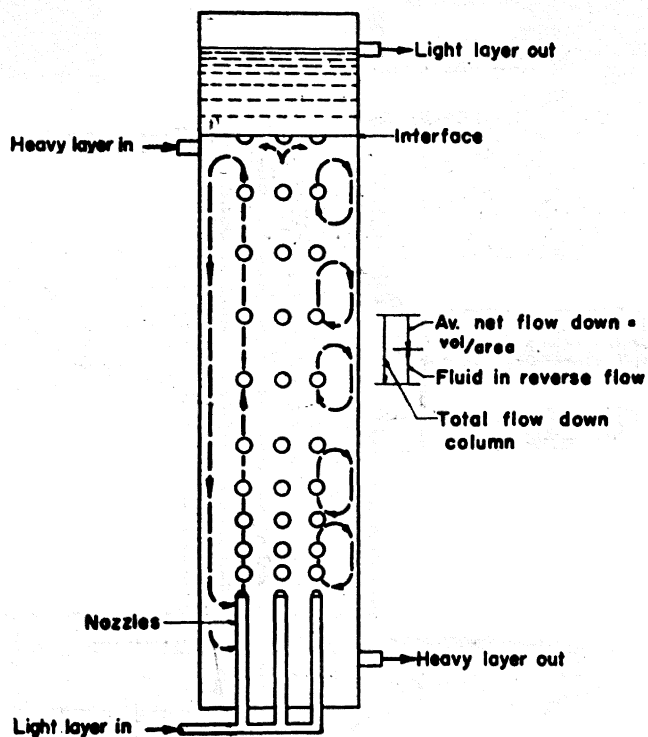


Figure 7. Simplified Spray Column Extractor

Morello, V. S. and N. Poffenberger: Commercial Extraction Equipment, Ind. Eng. Chem., 42, 1021 (1950).

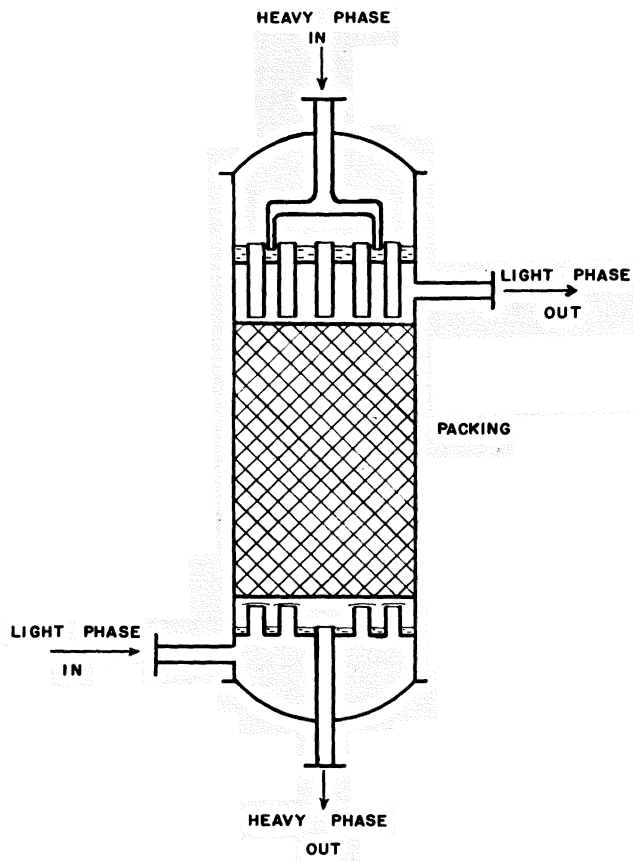


Figure 8. Packed Column Extractor

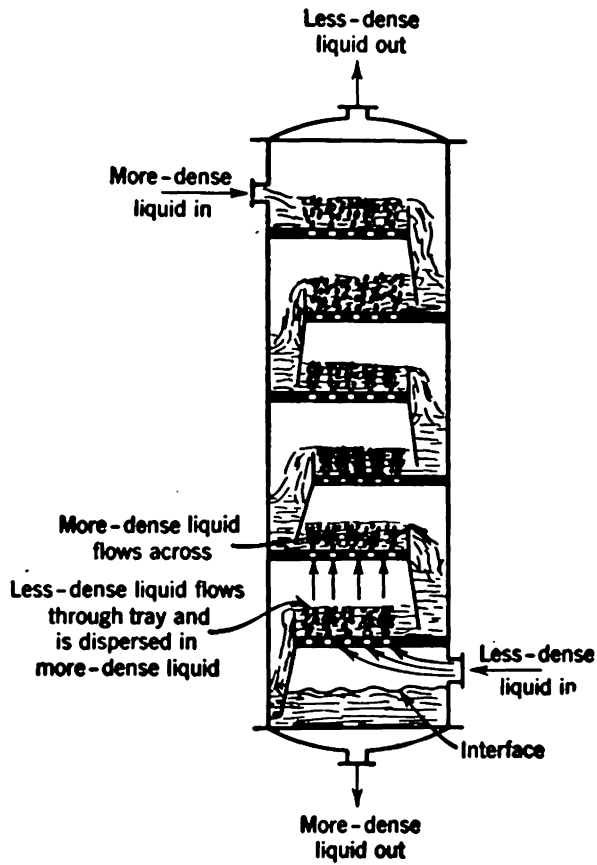


Figure 9. Perforated Plate Column Extractor

Brown, G. G., et al.: "Unit Operations," p. 301.
John Wiley and Sons, Inc., New York, N. Y.,
1950.

times by passage through plates having a large number of small holes is utilized in this type of extractor. There are many various designs of this type column, tray spacings varying from six inches to two feet⁽⁸⁵⁾. Operation of this type column depends upon the dispersed phase collecting under or above each plate until sufficient pressure is developed to cause the liquid to move through the holes in a jet and form droplets that proceed to the next plate to perform the cycle again. Perforated plate columns, especially those with perforations in vertical plates, have proved very satisfactory where they have been installed, being very popular in the oil industry.

Baffle Columns. The basic idea of this type extractor is a tower fitted with horizontal baffles over which the dispersed liquid phase flows, forming a large surface for contact with the continuous phase⁽⁸⁵⁾. Liquid flows across one baffle and then cascades to the one below. This type column is one of the most satisfactory types of commercial extractors⁽⁴⁶⁾, being of simple construction and with no small holes to plug or corrode. Smooth flow of liquid makes it possible for this type column to handle liquids that emulsify easily and would present problems in other type extractors.

Pulse Columns. In recent years an idea of giving increased turbulence and interface in either a packed or perforated plate

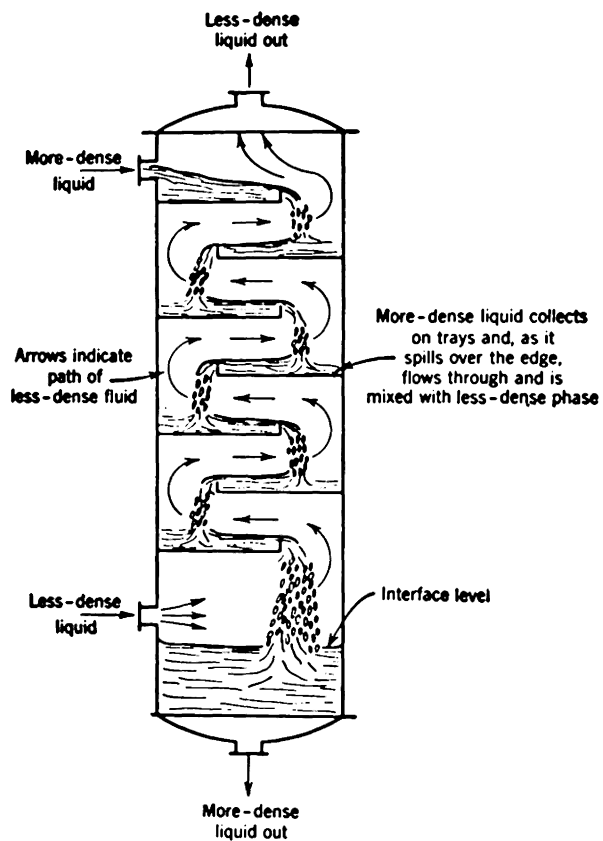


Figure 10. Baffle Column Extractor

Brown, G. G., et al.: "Unit Operations," p. 298.
John Wiley and Sons, Inc., New York, N. Y.,
1950.

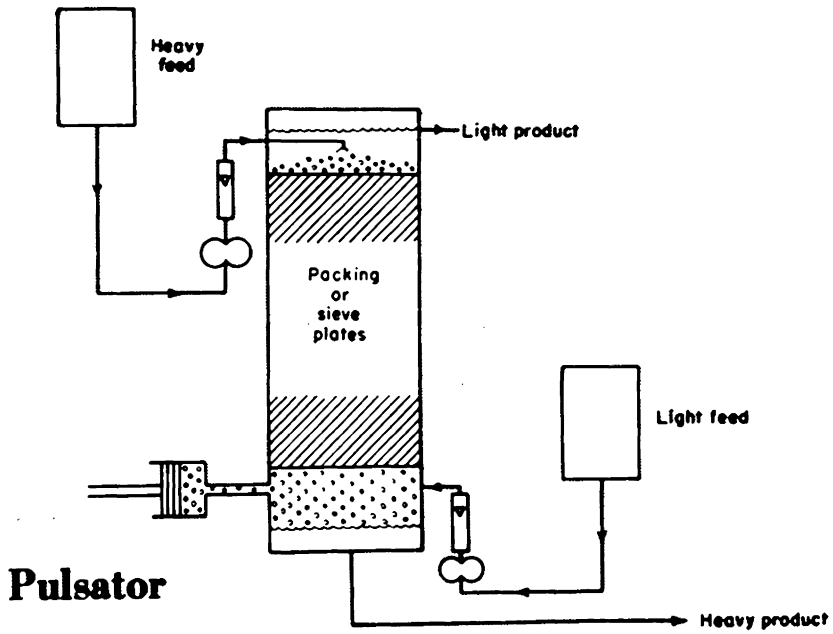
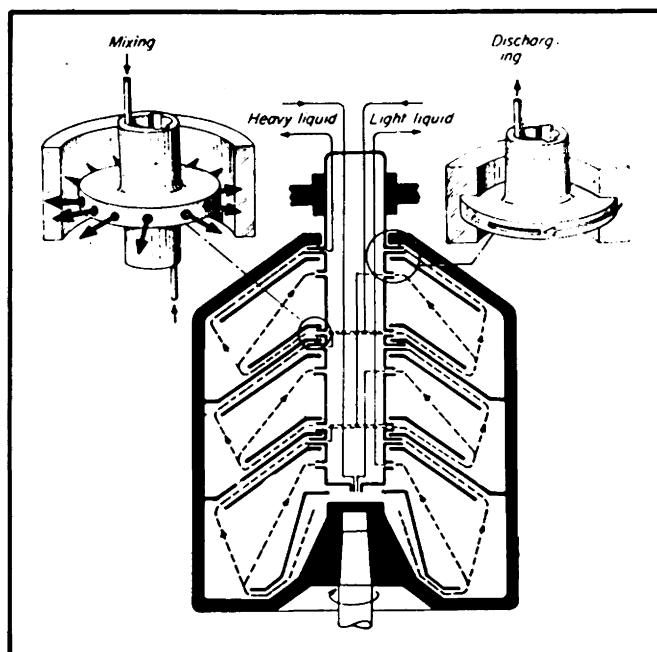


Figure 11. Pulse Column Extractor

Wiegandt, H. F. and H. L. Von Berg: Key to Better Extraction, Chem. Engr., 61, 7-183 (1954).

column has led to development of pulse columns. In such a column, an up-and-down pulsing motion is superimposed on the net counter-current flow of the liquid phases through a series of stationary perforated plates or packing material⁽⁶¹⁾. Experimental pulse columns have demonstrated that they are equal in extraction performed to ordinary columns three to five times as high. Emulsification problems are thought to be a possible limit to maximum use of this type column.

Centrifugal Extractor. In recent years a special type of extractor utilizing centrifugal force has come into use for special purposes. This type extractor is expensive, requiring careful design and construction to withstand severe mechanical stresses. This extractor, having a high throughput to inventory ratio⁽⁴⁶⁾, is very useful in handling materials which are easily decomposed, or which undergo spontaneous degradation in the process liquors. Application of this type extractor commercially has been mainly in the field of antibiotics and vitamins.



LUWESTA extractor combines three stages in one machine.

Figure 12. Luvesta (Centrifugal) Extractor

Von Berg, R. L. and H. F. Wiegandt: **Liquid-liquid Extraction**, *Chem. Engr.*, **59**, 6--189 (1952).

Ultrasonics

"Ultrasonics," sometimes confused with "supersonics," is the term employed in the field of acoustics that refers to sound waves of a frequency that is above that normally detected by human beings, approximately 15,000 to 20,000 cycles per second⁽⁵⁵⁾. ("Supersonics" is now generally thought of as related to the speed of airplanes or missiles traveling at a speed greater than that of sound in air.)

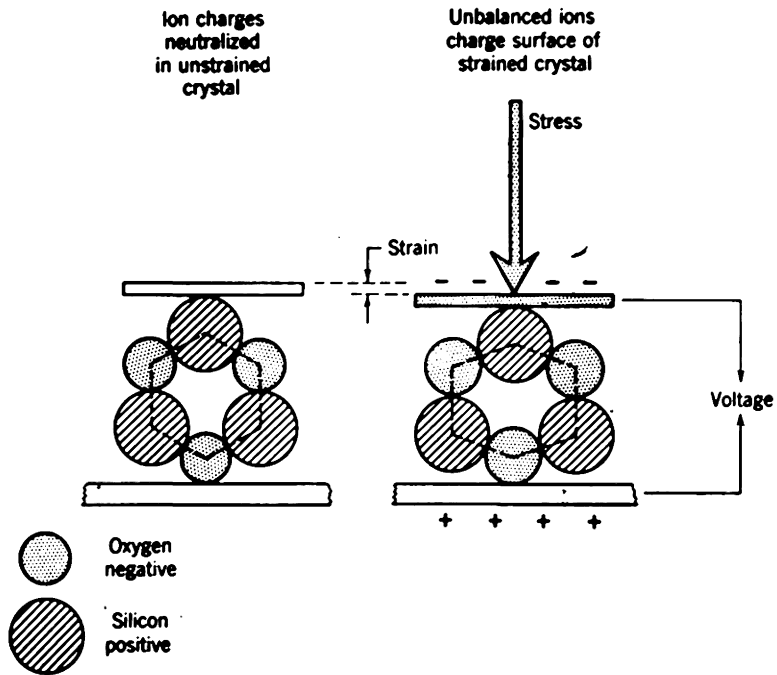
Loomis and Wood⁽⁴²⁾, in 1927, carried out a series of investigations of the applications of ultrasonics that had a great impact on the thinking and efforts of other scientists in this field of investigation. Their investigations were more qualitative than quantitative, in that observable effects of ultrasonic waves such as cavitation, emulsification, formation of fogs, etc., were reported. Much varied work has been accomplished since the time of this study to further investigate the properties, effects, and possible uses of ultrasonic waves^(4,30,44,64,72).

Ultrasonic Generators. Over the period of years during which ultrasonic wave studies have been conducted, several types of wave generators have been developed. Four principal types of generators used in ultrasonic work are: (1) piezo-electric, (2) magnetostrictive, (3) electromagnetic, and

(4) gas current. Of these four types of generators, the most widely accepted and used for the treatment of liquid systems is the piezoelectric. As the generator used in this investigation is of the piezoelectric type, further discussions of the last three types will not be undertaken.

Piezoelectric Generators. Piezoelectric crystals were employed in the ultrasonic generators initially constructed for experimental studies and are still prominent in the application of ultrasonic waves to various systems of different states of matter. The word piezoelectric comes from the Greek language, meaning "pressure electricity." Jacques and Pierre Curie⁽¹¹⁾ found in 1880 that this effect of "pressure electricity" holds for several crystals, including tourmaline, quartz, and Rochelle salt. A more quantitative definition of piezoelectricity is⁽¹²⁾ "the electric polarization produced by mechanical strain in crystals belonging to certain classes, the polarization being proportional to the strain and changing sign with it."

If pressure is applied to a crystal belonging to a crystal system without center of symmetry, certain crystal faces become electrically charged; conversely, if a potential difference is applied to certain faces of the crystal, it contracts or expands according to the direction of the applied voltage. Figure 13 illustrates the piezoelectric charging effect of a quartz crystal.



**Figure 13. Interaction Between Force and Field
in Piezoelectric Crystals**

**Huster, T. F. and R. H. Bolt: "Sonic," p. 88.
John Wiley and Sons, Inc., New York, N. Y.,
1955. 1 ed.**

Excitation of piezoelectric crystals is caused by application of a high frequency electrical field to the crystal surface. Maximum vibration of the crystal surface is obtained when the frequency of the applied electric field is the same as the natural mechanical frequency of the crystal. Piezoelectric crystals used for ultrasonic studies are connected by suitable electrodes to a high frequency generator and either immersed in an oil bath or mounted so that the crystal surface is in contact with oil or other liquid transmitting medium. In some cases, the transmitting medium may be the liquid that is to be treated.

Piezoelectric Materials. Piezoelectric materials for ultrasonic generators are available in a comparatively wide variety⁽¹⁰⁾. Properties of the various materials differ widely, and determine to a large extent the application of each type material. Quartz crystals are very well suited for frequency control; quartz and EDT (ethylene diamine tartrate) are used for crystal filtering; ADP (ammonium dihydrogen phosphate) has been used in applications of Sonar⁽¹⁰⁾.

A piezoelectric material that has proved to be well suited for ultrasonic applications in liquid mediums is the ceramic barium titanate ($BaTiO_3$)⁽¹⁰⁾. The comparison chart, Table IV, indicates some of the desirable properties of barium titanate in relation to other piezoelectric materials. For a given

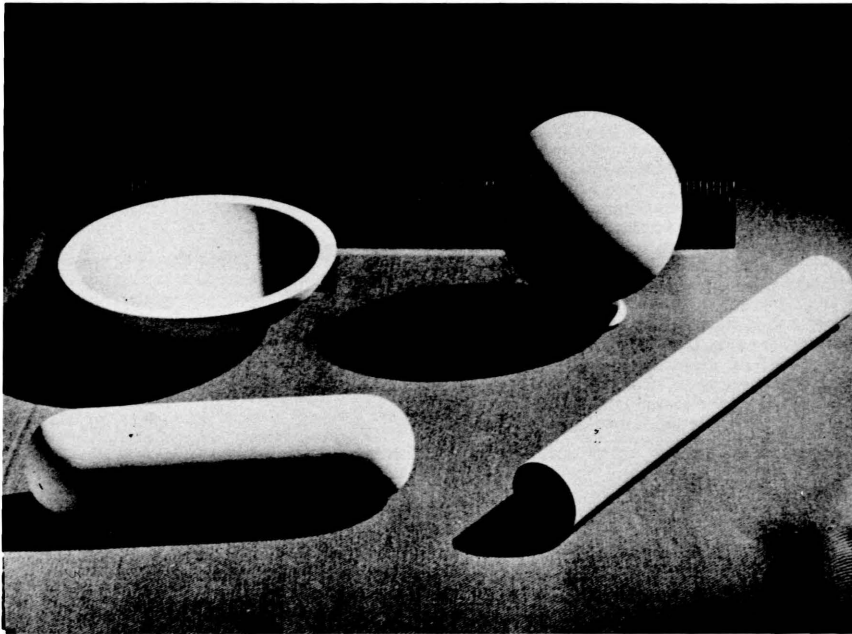
TABLE IV

Comparative Chart of Physical Properties
of Piezoelectric Crystals

Expander Modes	Motion Out Voltage in d 10^{-12} m/V	Dielectric Constant K	Voltage Out Force in g $V \cdot m$ Newton	Elastic Modulus Y 10^9 Newton m^2	Voltage Out Motion in $Y \cdot g$ 10^6 V/m	Coupling Coefficient $k = \sqrt{dg Y}$	Frequency Constant short- circuited kc.mm
Rochelle Salt (34°C)							
X cut lateral	165	200	.093	19.3	2.9	.54	2100
Y cut lateral	27	9.2	.33	10.7	3.6	.31	1180
ADP							
Lateral	24	15.3	.177	19.3	3.4	.29	1630
Quartz							
Lateral and Parallel)	2.3	4.5	.058	80	4.6	.103	2700
Lithium Sulfate (LH)							
Parallel	16.0	10.3	.175	46	8.0	.36	2360
DKT							
Lateral	11.0	6.5	.192	25	4.8	.23	1770
EDT							
YX cut Lateral	14.3	8.2	.156	26	4.0	.22	2050
Tourmaline							
Parallel	1.93	6.6	.033	160	5.3	.10	3600
Barium Titanate, Prepolarized							
Parallel	190	1700	.0125	87	1.10	.46	2000 - 2500
Lateral	78	1700	.0052	87	.45	.19	2000

Butterworth, J. W.: Kilowatts of Ultrasonic Power for Industry, "Ultrasonics--two Symposia." Chem. Engr. Prog. Sym. Series, No 1, 47, 71 (1951).

applied voltage, the obtainable physical motion of a barium titanate crystal is approximately 80 times greater than that of quartz. High potentials, in the order of thousands of volts for quartz, are not required for the use of barium titanate. Raw materials for production of barium titanate crystals are abundant in this country, and this material may be cast, pressed, or extruded in various shapes and sizes according to demand. Several different shapes of available barium titanate crystals are shown in Figure 14. A very useful barium titanate element is that formed in the shape of a focusing bowl. Higher intensities are obtainable from focusing elements, in relation to other type elements, as is evident in Figure 15.



**Figure 14. Various Available Shapes of Barium
Titanate Crystals**

Stokes, C. A. and J. E. Vivian: Applications of Sonic Energy in the Process Industries, "Ultrasonics--two Symposia." Chem. Engr. Prog. Sym. Series, No 1, 47, 13 (1951).

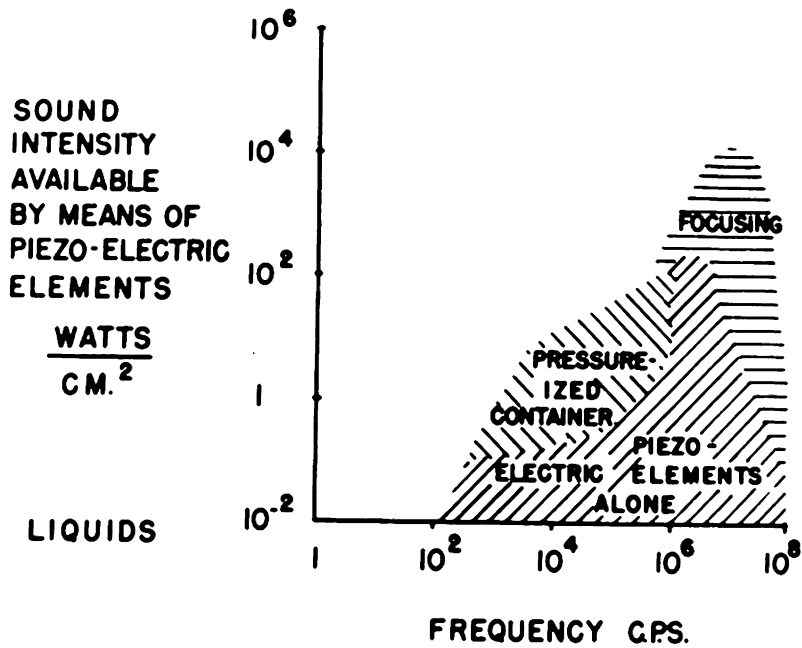


Figure 15. Spectrum of the Sounds that Can Be Produced
by Means of Piezoelectric Generators

Sansel, R. W.: Physical and Economic Limitations in the Application of Sonic and Ultrasonic Energy to Industrial Processes, "Ultrasonics--two Symposia." Chem. Engr. Prog. Sym. Series, No 1, 47, 79 (1951).

Effects of Ultrasonic Waves

In the application of ultrasonic waves to the treatment of liquid systems, cavitation is responsible for many of the resulting effects. For this discussion, the effects of cavitation in a liquid are divided into four parts: (1) mechanical, (2) thermal, (3) chemical, and (4) electrical.

Cavitation. "Cavitation" refers to the formation and violent collapse of small bubbles or cavities in a liquid due to pressure changes, in a "plus and minus" relation, to which the liquid may be subjected⁽⁸⁹⁾. A study and understanding of cavitation is important in such varied fields as medicine, hydraulics, water transportation, water signal transmission or communication, and industrial materials processing. The importance of this phenomenon of cavitation in liquids is emphasized by the impressive array of works compiled as a cavitation bibliography⁽⁴⁹⁾ by the David Taylor Model Basin in Washington, D. C.

Magnitude of the pressure required to produce this rupturing effect in a liquid depends upon conditions surrounding and condition of the liquid system in question. Cavities, gas or vapor filled ranging in size from submicroscopic to large enough to be seen by the naked eye, may be formed in a liquid by chemical, mechanical, or thermal action. Cavitation can occur in a liquid system when the total pressure at a point

in the liquid body is reduced to the vapor pressure of the liquid or below, and may be produced in a liquid by the application of high intensity ultrasonic waves. A liquid undergoing ultrasonic treatment is subjected to cyclic compression and tension, rupture of a liquid by this manner depending greatly on the magnitude of the tension, or negative pressure, during the rarefaction phase of the ultrasonic wave in the liquid.

Conditions Favorable for Cavitation. Cavitation will not occur in every liquid system insonated by ultrasonic waves. Intensity of the wave, physical condition of the liquid, and surrounding conditions combine to determine when and where cavitation will occur. A general intensity level of approximately 0.3 watt per square centimeter is suggested by Samsel⁽⁵⁹⁾ as that required for cavitation in a liquid system under atmospheric pressure. Physical condition of the actual liquid insonated is directly related to the occurrence of cavitation. Presence of dissolved gases or nuclei in liquids is necessary⁽⁹¹⁾ for cavitation to take place under normal operating conditions. An outside pressure on the liquid of approximately two atmospheres is suggested as the most favorable for cavitation^(13,68).

Mechanical Effects of Cavitation. Terrific point-wise hammering and pounding forces result from the high pressures produced on collapse of cavities in a liquid. The over-all

resultant force is one of very violent agitation. Pressures on the order of 765 tons per square inch may be created by a cavity collapsing at 1/100 its original size⁽⁵⁰⁾. Agitation caused by this terrific shearing and tearing action plays an important role in the effects of ultrasonic waves.

Thermal Effects of Cavitation. It is known that a liquid undergoing ultrasonic insonation over a period of time exhibits a temperature rise^(3,42,64). Although the rise of the entire body of liquid may be only a few degrees, it has been estimated that point-wise temperatures at collapse of cavities may rise as much as several hundred degrees⁽⁸⁸⁾.

Intense sound treatment in liquid and solid systems is always accompanied by considerable thermal effects which are very greatly increased in the presence of extended phase boundaries, as they occur in micro-heterogeneous systems⁽⁶⁴⁾.

Chemical Effects of Cavitation. Weissler⁽⁸⁸⁾ reports detailed investigations of some chemical effects of intense ultrasonic waves in liquids as follows: (1) liberation of iodine from an aqueous solution of potassium iodide and carbon tetrachloride, (2) delay in formation of colloidal sulfur, and (3) depolymerization of polystyrene in toluene and hydroxyethyl cellulose in water. In the three cases studied, it was found that ultrasonic waves cause chemical reactions

in liquids only in the presence of dissolved gases, indicating that cavitation is responsible for these effects. Weissler defines "sonochemistry"⁽⁸⁹⁾ as "the production of chemical changes by sound waves."

A comprehensive listing of reported effects produced in ultrasonic fields that appear to be correlated with chemical action is presented by Thompson⁽⁷²⁾. Such effects include: (1) accelerated reaction rates, (2) electrolytic effects, (3) oxidation, (4) luminescence, (5) polymerization, (6) decomposition, (7) inversion of sucrose, (8) increased crystallization, and (9) the determination of molecular properties.

Electrical Effects. Electrical effects of cavitation have been investigated, but due to the lack of conclusive information, no definite theories in regard to this effect have been advanced. One investigator⁽⁵³⁾ did find considerable variances in potential in a region of cavitation in a liquid, while none were found in the same liquid not undergoing cavitation. Another investigator⁽²⁸⁾ advanced the idea that electrical forces created during cavitation are responsible for observed chemical reactions.

Applications of Ultrasonic Waves

Chemical engineers have always been intrigued by new and different forms of energy and possible applications of that form of energy to operations or processes. As another new form of energy with possible uses, ultrasonic waves have become a new tool⁽⁷¹⁾ available to chemical engineers in their endeavor to ever improve unit operations and unit processes. The application of ultrasonic waves in a technical manner may be divided into two major fields⁽⁴⁵⁾: (1) low-power applications, ultrasonic waves being used as an instrument for inspection, testing or measuring and (2) high-power applications, where the energy carried in the ultrasonic wave is utilized in bringing about a physical or chemical change.

Ultrasonic Testing. Non-destructive testing of materials by use of ultrasonic waves is one of the first applications to be employed commercially on a large scale. Ultrasonic instruments are used for inspection and discovery of minute flaws in many types of materials, including steel, aluminum, magnesium, brass, plastics, and glass. There are three general types of tests that can be performed by use of ultrasonic waves⁽¹⁶⁾: (1) step-by-step plant inspection during fabrication, (2) fatigue testing to periodically check parts that may develop faults due to

continued use, and (3) repair testing, to locate areas to be removed, rewelded, etc. The advantages of ultrasonic testing are brought out in the realization that small flaws through 30 feet of steel may be detected⁽⁷¹⁾, and that fatigue testing may be done on equipment shafts and axles while actually in place on equipment pieces.

Thickness gaging for such materials as steel or aluminum is carried out by ultrasonics using a pulsed ultrasonic tester⁽¹⁵⁾, giving good results on thicknesses of one-half inch and up. A resonance method is used in testing thickness of parts between 0.005 and 0.5-inch thick.

Ultrasonic Cleaning. Perhaps one of the most useful applications of ultrasonically induced cavitation is in the cleaning of small precision metal parts that have been contaminated by dust or process oils. Remington Rand and Schick⁽⁴⁵⁾ employ this idea to great advantage in the economical, fast, and sure cleaning of electric razor heads. Ultrasonic cleaning of very small and accurately machined parts for delicate instruments has been developed by Bendix Aviation Corporation⁽⁴⁵⁾.

Ultrasonic Emulsification. The possibility of using ultrasonic energy as a means of obtaining emulsions of two immiscible liquids has been investigated quite thoroughly by Bondy and Sollner^(65,66,67,68) and by others⁽¹⁴⁾. These investigators

found that this emulsifying effect depends on ultrasonically induced cavitation. Interest in this method of forming emulsions is high in the pharmaceutical industry for special purposes and for making emulsions of very small quantities of liquids⁽¹⁴⁾.

Troger⁽⁴⁵⁾ reported that large volumes of liquids may be successfully processed by using the Pohlmann Ultrasonic Whistle. One thousand liters of a 20 per cent paraffin emulsion were obtained by recirculation. Without the use of ultrasonics, only a five per cent emulsion was obtained.

Ultrasonic Communication. Sound signalling was one of the first technical applications of ultrasonics. One of the first investigators interested in this field was M. L. F. Richardson⁽⁵⁶⁾, who was influenced by the sinking of the "Titanic." His work was centered around patented methods for detecting the presence of icebergs in fog by use of ultrasonic waves. Underwater submarine signalling using ultrasonic waves has created interest during war time⁽⁷¹⁾.

Miscellaneous Applications. As a new type of energy, the possible use of ultrasonics in many and varied fields has been studied by numerous persons. Not all of these studied applications are used on a commercial basis, for various reasons. Some of these miscellaneous applications are: (1) improvement of leather conditioning⁽⁴⁵⁾, (2) flotation ore dressing⁽⁴⁵⁾,

(3) water degassing^(33,45), (4) degassing of molten glass and metals^(33,45), (5) tinning of aluminum for soldering^(33,45), (6) descaling metal strips⁽⁴⁵⁾, (7) acceleration of electrochemical reactions^(30,81), (8) dust precipitation^(21,22), (9) carbon black agglomeration⁽⁷⁰⁾, and (10) processing of milk⁽⁷⁰⁾.

Application of Ultrasonics to Liquid-liquid Extraction

Problems in which rate processes are controlled by diffusion of material at an interface are everyday occurrences for chemical engineers. These diffusion rates, within reasonable limits, are related to velocities at such interfaces. Because of the high local velocities associated with high frequency, high intensity sound energy, it is thought that an increased rate of mass transfer may be obtained at the interfacial boundary between two phases⁽⁵⁰⁾.

Liquid-liquid Extraction. Liquid-liquid extraction processes include the following sequence of steps: (1) production of an intimate contact of feed mixture with solvent, (2) separation into two liquid phases, and (3) recovery of solute from extract phase and solvent from both phases⁽⁸⁵⁾. Steps (2) and (3), although affecting the over-all economic efficiency of an extraction operation, do not affect the actual mass transfer

of solute in the system in the manner of step (1). The accomplishment of a better contact between feed and solvent has been the prime consideration of the many and varied new equipment designs created. The number of designs proposed, not all commercially successful, is very large, as evidenced by the patent literature⁽⁷⁸⁾.

Liquid Mixing. Mixing in a liquid system is brought about by turbulence and momentum transfer⁽⁵⁷⁾. Turbulence involves fluid motion of a rotary eddy current type and takes place in all directions. Turbulence is brought about by the interaction of momentum, fluid properties and surfaces, and it is believed that mixing is accomplished by transfer of momentum to and from small masses, thus causing them to move in various directions.

Turbulence produces a dispersion of one liquid phase in the other and provides a large surface area for mass transfer at a phase boundary between the two phases. It also aids mass transfer to the phase boundary in a single phase because of the movement and circulation within each phase relative to this surface⁽⁸⁵⁾.

Extraction Contactor. In a continuous liquid-liquid extraction operation, the function of the contactor is to provide a large interfacial area for mass transfer between the two liquid phases⁽²⁰⁾. As has been previously discussed, increased

turbulence in the contactor will increase this interfacial area available for mass transfer.

Ultrasonic Dispersion of Liquid Droplets. Ultrasonic dispersion of droplets of one liquid phase into a second, immiscible with the first, has been demonstrated many times^(14,53). This dispersion effect has been utilized in production of emulsions, but could also be utilized in providing an increase in interfacial area between the two phases present in a liquid-liquid extraction system. This dispersing action results from cavitation, which is favored at the interface between two liquid phases⁽⁶⁵⁾.

Ultrasonic Stirring Effects. A second manner of increasing mass transfer in a liquid-liquid extraction system would be that of keeping a solute concentration gradient across the two phase interface that is always as large as possible, giving a greater driving force for transfer of the solute. Gross stirring effects^(65,73) of ultrasonic waves would reduce the concentration gradient within a single liquid phase, increasing the gradient across a two phase interface, by keeping fresh solute containing feed liquid of one phase and fresh solvent, with relatively small amount of solute, at opposite sides of the interface.

Ultrasonic Thermal Effects. A third possible effect of ultrasonic waves on mass transfer in a two phase liquid system

is the resulting temperature effect. It has been reported that point-wise temperatures resulting from collapse of cavities is on the order of several hundred degrees⁽³⁸⁾, and that two phase liquid systems having an interface have a more marked thermal effect resulting from ultrasonic treatment than to single phase systems⁽⁶⁴⁾. In some cases⁽⁷⁾, solubility of a solute in a solvent increases with an increase in temperature. As this temperature rise is an inherent characteristic of liquids insonated by ultrasonic waves, its possible effect is not to be neglected in the application of ultrasonic energy to mass transfer in liquid-liquid extraction. For certain systems, this effect might be advantageous, and for others it might be detrimental.

Previous Applications. To the present knowledge of the writer, only two investigations applying ultrasonic energy to liquid-liquid extraction have actually been conducted. The first is the 400 kilocycle insonation of the system carbon tetrachloride-acetone-water⁽⁴⁷⁾ and the second is the application of ultrasonics to a spray-tower extraction of phenanthrene from methanol by gasoline, increasing the extraction by 76 per cent in a small apparatus⁽³¹⁾. Brief mention of application of ultrasonics to extraction operations has been made in the

literature^(8,28), but evidence of further work from these authors is not known.

Postulation. Known properties of ultrasonic waves and effects of these waves that have been observed would lead one to believe that this form of energy can be utilized to increase mass transfer in a liquid-liquid extraction operation.

By considering together the known properties and effects of ultrasonic waves and different methods employed to increase mass transfer of a solute in a liquid-liquid system, it is postulated that ultrasonic energy may affect mass transfer in a liquid-liquid extraction system in a positive direction by at least three mechanisms, simultaneously occurring but separate in nature⁽⁷⁴⁾: (1) increasing conductance of interface by decreasing interfacial resistance to mass transfer, (2) increasing interfacial area between two liquid phases by reducing size and increasing number of particles of dispersed phase, and (3) increasing concentration gradient across the interface by decreasing concentration gradient within a single phase.

III. EXPERIMENTAL

The experimental section of this thesis is divided into the following sections: (1) purpose of investigation, (2) plan of investigation, (3) materials and equipment used, (4) construction and operational procedure, and (5) equipment evaluation.

Purpose of Investigation

The purpose of this investigation was to design and construct a laboratory scale liquid-liquid extraction unit incorporating an ultrasonic field with equipment specifications as:

- (1) insonation frequency of 400 kilocycles per second,
- (2) insonation intensities corresponding to plate currents of 0 to 200 milliamperes, and
- (3) flow rate of solvent and feed through reactor ranging approximately from one-half to ten pounds per minute, in varying solvent-to-feed ratios.

Plan of Investigation

The plan for the accomplishment of work entailed in this investigation was divided into four main sections: (1) literature review, (2) design of equipment, (3) construction, and (4) testing of equipment with the system acetone-water-1,1,2-trichloroethane.

Literature review for this investigation was conducted with the idea of studying the published results of research workers dealing with the two major concepts present in the general field of investigation: (1) mass transfer in the unit operation of liquid-liquid extraction, and (2) the manner in which ultrasonic energy affects mass transfer.

Three important factors considered in the original equipment design were over-all flow plan of liquids, instrumentation, and reactor design. Each of these was investigated before construction began.

The project and investigation were complete when the unit was operating in a manner considered proper by design and construction personnel, based on knowledge of ultrasonics and liquid-liquid extraction. Testing was done with the system acetone-water-1,1,2-trichloroethane.

Materials

Construction materials and items of apparatus employed in the design and construction of the liquid-liquid extractor are given in Table V.

Equipment

This section deals with equipment assemblies and sub-assemblies and contains information concerning selection, construction, and assembly of the individual items of the extractor system.

Selection of Special Equipment Pieces. The primary problems arising in the selection of individual instruments and controls were: (1) the accurate measurement of flow rates and temperatures, and (2) a method of obtaining steady rate of flow for feed and solvent. The HYPERSONIC Generator, Model BU-204, and focused barium titanate ceramic transducer were selected as the ultrasonic equipment for this investigation.

Flow Meters. The extractor of this thesis was designed for use with liquid systems of varying specific gravities. This required flow meters that were calibrated with a correction for specific gravity. No

TABLE V

Construction Materials and Items of Apparatus Employed in
the Design and Construction of a Liquid-Liquid Extractor

Number required	Unit	Name	Description	Supplier
<u>Frames for Control Panel and Tanks</u>				
190	ft	angle iron	1-1/4 x 1-1/4 x 3/16-in.	Steel Service Roanoke, Virginia
6	sq ft	steel	1/8-in. sheet	Steel Service Roanoke, Virginia
35	ft	strap	3/4-in. aluminum	Applied Mechanics Dept. Virginia Polytechnic Institute Blacksburg, Virginia
8	ea	bolt and nut	5/16 x 3-in., machine	Graves-Humphries Hardware Co. Roanoke, Virginia
55	ea	bolt and nut	1/4 x 3/4-in., machine	Graves-Humphries Hardware Co. Roanoke, Virginia
5	ea	bolt and nut	1/4 x 3-in., machine	Graves-Humphries Hardware Co. Roanoke, Virginia
60	ea	washer	1/4-in. nom., flat	Graves-Humphries Hardware Co. Roanoke, Virginia
32	ea	bolt and nut	No 10, oval head, 1-1/2-in., machine	Lucas Motor Co. Blacksburg, Virginia
32	ea	washer	No 8, countersunk finishing	Lucas Motor Co. Blacksburg, Virginia
10	ea	lag screw	1/4 x 2-in.	Tidewater Supply Co. Roanoke, Virginia
64	sq ft	plywood	5 ply, 1/2-in., one side good	Economy Lumber Co. Christiansburg, Va.
96	ft	board	2 x 4-in., pine	Economy Lumber Co. Christiansburg, Va.
42	ft	board	1 x 8-in., pine	Economy Lumber Co. Christiansburg, Va.
1	lb	nails	16 d, common	Economy Lumber Co. Christiansburg, Va.
2	qt	paint	Martin-Sonour, satin gloss, Bayberry Green	Roanoke Hardware Co. Roanoke, Virginia
1	qt	varnish	Pratt & Lambert, clear gloss	Economy Lumber Co. Christiansburg, Va.
4	pr	hinge	1-1/2-in. brass, cabinet	Blacksburg Hardware Blacksburg, Va.
2	ea	catch	friction catch, cabinet	Blacksburg Hardware Blacksburg, Va.
2	ea	handle	3-in. chrome, cabinet pull	Blacksburg Hardware Blacksburg, Va.
<u>Electrical Wiring System</u>				
300	ft	wire	No 16 solid, thermo- plastic, hook-up	Newark Electric Co. Chicago, Illinois
30	ft	wire	No 14 copper, 2 wire "Romex" cable	Electrical Department Virginia Polytechnic Institute Blacksburg, Va.
50	ea	lug	spade term., general wiring type	Allied Radio Corp. Chicago, Illinois
1	roll	tape	60 ft., No 33 Scotch black insulated	Allied Radio Corp. Chicago, Illinois
1	ea	term. strip	6 term. Fanning strip	Allied Radio Corp. Chicago, Illinois
1	ea	term. strip	2 term. Fanning strip	Allied Radio Corp. Chicago, Illinois
1	ea	terminal	6 term. barrier term.	Allied Radio Corp. Chicago, Illinois
1	ea	terminal	2 term. barrier term.	Allied Radio Corp. Chicago, Illinois
1	ea	switch box	No 24221 Trumbull switch box, 30 A, 250 volt, 2 pole	General Electric Co. Roanoke, Virginia
1	ea	panel box	No 29A12 Trumbull fuse panel, 13 circuit	General Electric Co. Roanoke, Virginia
3	ea	socket	ceramic light socket	Electrical Department Virginia Polytechnic Institute Blacksburg, Virginia
3	ea	switch	toggle, dead center, D.P.D.T., 15A-125V	Cosby Electric Co. Richmond, Virginia
1	ea	voltmeter	A. C. voltmeter, 150 volt rating, type RA-35	Westinghouse Electric Co. Roanoke, Virginia
1	ea	ammeter	A. C. ammeter, 10 amp rating, type RA-35	Westinghouse Electric Co. Roanoke, Virginia
1	ea	milliammeter	R. F. milliammeter, 500 ma rating, type RT-35	Westinghouse Electric Co. Roanoke, Virginia
12	ea	light	pilot light and bracket, No UT 1377	General Electric Co. Roanoke, Virginia
12	ea	switch	Pass & Seymour, No 1391, residence type, brown	General Electric Co. Roanoke, Virginia
12	ea	cover plate	Pass & Seymour, double opening, Cirrom-X, No 97021	General Electric Co. Roanoke, Virginia
1	ea	clock	Westlock Manor, elect. wall clock, No. 1080	Westinghouse Electric Co. Roanoke, Virginia
1	ea	flasher	bimetallic, socket type, 60 watts	Blacksburg Hardware Blacksburg, Virginia
2	ea	light bulb	100 watt, 110 volt	Electrical Department Virginia Polytechnic Institute Blacksburg, Virginia
1	ea	light bulb	7-1/2 watt, 110 volt, red	Blacksburg Hardware Blacksburg, Virginia
<u>Thermocouple System</u>				
30	ft	thermocouple wire	copper-constantan, Brown Duplex, 20 gage, No 9B1C2	Minneapolis-Honeywell Regulator Co. Philadelphia, Pa.
1	ea	switch	ten point, thermocouple rotary switch	Leads & Northrup Co. Philadelphia, Pa.
1	ea	vacuum bottle	thermos brand, No 2280	American Thermos Bottle Company Norwich, Conn.
1	ea	potentiometer	Rubicon high precision, type "D"	Rubicon Company Philadelphia, Pa.
1	ea	standard cell	cadmium, Dpoley	Fisher Scientific Co. New York, N. Y.
2	ea	battery	1-1/2 volt dry cell	Western Auto Co. Blacksburg, Va.
7	ea	tee	5/16-in., "SWAGELOK", all tube tee, aluminum	Shelby Jones Co. Havertown, Pa.
7	ea	stopper	No 00, rubber	Chemical Engineering Dept. Virginia Polytechnic Institute Blacksburg, Va.
3	ea	plug	insulated banana plug	Allied Radio Corp. Chicago, Illinois
3	ea	jack	insulated banana jack	Allied Radio Corp. Chicago, Illinois
<u>Tanks, Tubing, and Fittings for Flow Lines</u>				
300	ft	tubing	5/16-in. O. D., O.035-in. wall, aluminum	Whitehead Metal Products Company New York, New York
8	ea	tank	12-in. x 24-in., stainless steel, 500 psi.	A. C. Tank Company Burlington, Wisc.
30	ea	valve	HOKK, No D371, 1/4-in. NPT, aluminum	Shelby Jones Co. Havertown, Pa.
60	ea	connector	SWAGELOK, No A500-1-4 male connector, 5/16-in. T x 1/4-in. P, aluminum	Shelby Jones Co. Havertown, Pa.
14	ea	plug	SWAGELOK, No A500-11 plug, 5/16-in. T, aluminum	Shelby Jones Co. Havertown, Pa.
60	ea	elbow	SWAGELOK, No A500-8-4 female elbow, 5/16-in. T x 1/4-in. P, aluminum	Shelby Jones Co. Havertown, Pa.
45	ea	tee	SWAGELOK, No A500-3 all tube tee, 5/16-in. T,	Shelby Jones Co. Havertown, Pa.
4	ea	cap	SWAGELOK, No A500-10 cap, 5/16-in. T, aluminum	Shelby Jones Co. Havertown, Pa.
5	ea	union	SWAGELOK, No A500-6 union, 5/16-in. T, aluminum	Shelby Jones Co.
<u>Sight Gages</u>				
8	ea	pipe	3/8-in. dia. x 18-in. long, pyrex flanged glass pipe	Sentinel Glass Co. Hatboro, Pa.
16	ea	flange	3/8-in. flange, aluminum	Sentinel Glass Co. Hatboro, Pa.
16	ea	insert	3/8-in. insert, asbestos	Sentinel Glass Co. Hatboro, Pa.
64	ea	bolt and nut	1/4 x 3/4-in. machine	Sentinel Glass Co. Hatboro, Pa.
16	ea	gasket	3/8-in. gasket, asbestos	Sentinel Glass Co. Hatboro, Pa.
2	ft	angle iron	1-1/4 x 1-1/4 x 3/16-in.	Steel Service Roanoke, Virginia
2	ft	strap iron	3/4-in.	Steel Service Roanoke, Virginia
1	sq ft	alum plate	1/4-in.	Applied Mechanics Dept. Virginia Polytechnic Institute Blacksburg, Virginia
32	ea	bolt and nut	1/8 x 1-in. machine	Blacksburg Hardware Blacksburg, Virginia
<u>Reactors</u>				
12	ea	funnel	Bunsen filtering, pyrex, 100 mm. dia.	Fisher Scientific Co. New York, N. Y.
2	ft	tubing	5/16-in. tygon tubing	Fisher Scientific Co.
1	sq ft	tin	22 gage, sheet	Chemical Engineering Dept. Virginia Polytechnic Institute Blacksburg, Virginia
2	sq ft	nickel	0.001-in., sheet	A. D. Mackay Company New York, N. Y.
1	roll	solder	wire solder	Blacksburg Hardware Blacksburg, Virginia
2	oz	silver paste	No 6316 silver paste	E. I. duPont de Nemours & Co., Inc. Electrochemicals Dept. Perth Amboy, N. J.
<u>Settling Column</u>				
1	ea	pipe	2-in. dia. x 12-in. long, pyrex flanged glass pipe	Sentinel Glass Co. Hatboro, Pa.
2	ea	flange	2-in. aluminum flange	Sentinel Glass Co. Hatboro, Pa.
1	sq ft	alum. plate	1/4-in.	Applied Mechanics Dept. Virginia Polytechnic Institute Blacksburg, Virginia
3	ft	angle iron	1 x 1 x 1/8-in.	Steel Service Roanoke, Virginia
2	sq ft	plywood	5 ply, 1/2-in., one side good	Economy Lumber Co. Christiansburg, Va.
6	ea	bolt and nut	No 10, oval head, 1-1/2-in., machine	Lucas Motor Co. Blacksburg, Virginia
6	ea	washer	No 8, countersunk finishing	Lucas Motor Co. Blacksburg, Virginia
<u>Pumps and Flow Meters</u>				
3	ea	pump	lab. pump, 7 gal/min, Cat No 13-874-93	Fisher Scientific Co. New York, N. Y.
2	ea	pump	lab. pump, 4.5 gal/min, Cat No 13-874-92	Fisher Scientific Co. New York, N. Y.
1	ea	flow meter	size 2, Fig 735-Z, TRI-FLAT, serial X2-4192/1 ss float, 2FI/4-20-5/70 tube	Fischer & Porter Co. Hatboro, Pa.
4	ea	flow meter	size 3, Fig 735-Z, TRI-FLAT, 3FG/8-20-5 tube, serials X2-1344/1-4, glass & ss floats	Fischer & Porter Co. Hatboro, Pa.
<u>Air Pressure Line</u>				
10	ft	pipe	1/2-in., black iron	Noland Company, Inc. Roanoke, Virginia
45	ft	pipe	1/4-in., black iron	Noland Company, Inc. Roanoke, Virginia
1	ea	union	1/2-in., black iron	Noland Company, Inc. Roanoke, Virginia
3	ea	elbow	1/2-in., black iron	Noland Company, Inc. Roanoke, Virginia
1	ea	tee	1/2-in., black iron	Noland Company, Inc. Roanoke, Virginia
1	ea	nipple	1/2-in., close, black iron	Noland Company, Inc. Roanoke, Virginia
16	ea	elbow	1/4-in., black iron	Noland Company, Inc. Roanoke, Virginia
3	ea	union	1/4-in., black iron	Noland Company, Inc. Roanoke, Virginia
5	ea	tee	1/4-in., black iron	Noland Company, Inc. Roanoke, Virginia
1	ea	plug	1/4-in., cast iron	Noland Company, Inc. Roanoke, Virginia
2	ea	valve	1/4-in., globe	Noland Company, Inc. Roanoke, Virginia
1	ea	tank	4-in. dia., 12-in. long, rolled steel, welded from 4-in. pipe and 1/4-in. plate	Chemical Engineering Dept. Virginia Polytechnic Institute Blacksburg, Virginia
1	ea	regulator	1/4-in. Mason Heilan air regulator, No 71	Noland Company, Inc. Roanoke, Virginia
2	ea	gage	0-100 psig, Mason Heilan, No 3716-4	Noland Company, Inc. Roanoke, Virginia
3	ft	angle iron	3/4 x 3/4 x 1/8-in.	Steel Service Roanoke, Virginia
<u>Constant Temperature Bath</u>				
1	ea	bath	Fisher unitized, with jar, base, heater, stirrer, thermostat, thermometer	Fisher Scientific Co. New York, N. Y.
10	ft	tubing	1/4-in., copper	Chemical Engineering Dept. Virginia Polytechnic Institute Blacksburg, Virginia
2	ea	adapter	1/4-in. tubing to 3/8-in. pipe	W. B. Clements Co. Roanoke, Virginia
2	ea	reducer	reducing coupling, 1/2-in. to 3/8-in.	Noland Company, Inc. Roanoke, Virginia
30	ft	pipe	1/2-in., galvanized	Noland Company, Inc. Roanoke, Virginia
2	ea	nipple	3/3-in., 3-in. long, galvanized	Noland Company, Inc. Roanoke, Virginia
9	ea	elbow	1/2-in., galvanized	Noland Company, Inc. Roanoke, Virginia
3	ea	tee	1/2-in., galvanized	Noland Company, Inc. Roanoke, Virginia
2	ea	union	1/2-in., galvanized	Noland Company, Inc. Roanoke, Virginia
4	ea	valve	1/2-in., globe	Noland Company, Inc. Roanoke, Virginia
<u>Ultrasonic Equipment</u>				
1	ea	generator	Brush HYPHASONIC, Model BU-304, Serial 535071, with tuning driers A & B, range of 100 to 1000 kilocycles per second	Brush Electronics Co. Cleveland, Ohio
1	ea	transducer	Brush HYPHASONIC, Model BU-305A, 400 KC, serial 513082	Brush Electronics Co. Cleveland, Ohio

constant volume meters having capacities within the range of operation of this extractor system, approximately 100 to 900 grams per minute, were available. "TRI-FLAT" flow meters, Fischer and Porter Company, were selected. Factory calibration of these meters was accurate within 1-1/2 per cent of maximum flow. Calibration accuracy may be increased by employing correction factors for float design and fluid viscosity.

Thermocouple System. Matched wire for copper-constantan thermocouples was employed. A Rubicon potentiometer was installed for measuring potential, and provisions were made for the future installation of a multiple-point, indicating potentiometer.

Constant Flow Apparatus. A positive pressure was required for feed and solvent streams entering the reactor, to overcome back pressure caused by ultrasonic cavitation. Air pressure in the range of ten pounds per square inch, gage, controlled by a pressure regulator, was selected as the motivating force for smooth and positive flow from feed and solvent tanks to the reactor.

Equipment Sub-Assemblies. In cases where standard engineering and scientific items of apparatus were employed or constructed, engineering drawings and pictures are shown to identify the

particular sub-assembly for the reader. Only items of unique interest to this investigation are discussed in detail.

Ultrasonic Generator. HYPERSONIC generator, model BU-204, was used in this investigation. This generator, produced by Brush Development Company, Cleveland, Ohio, has a nominal output of 250 watts over a frequency range of 100 to 1000 kilocycles. Circuit is a self-excited Hartley oscillator. Important physical features of this generator are shown in Figures 16 and 17.

Barium Titanate Transducing Crystal. The transducer employed in this investigation was a focused, bowl-shaped, barium titanate ceramic element of approximately 400 kilocycles frequency. The transducer was mounted in a metal housing to facilitate electrical connections and safe handling. Figure 18 shows the unmounted transducer; a view of the transducer mounted in the housing is given in Figure 19. Illustration of the principle of operation of this particular type of piezoelectric transducer is given in Figure 20.

Reactor. A special reactor was constructed for this investigation. The reactor was made from a pyrex glass funnel and was fitted with an acoustical window of 0.001-inch nickel sheet on the bottom. Design for this reactor

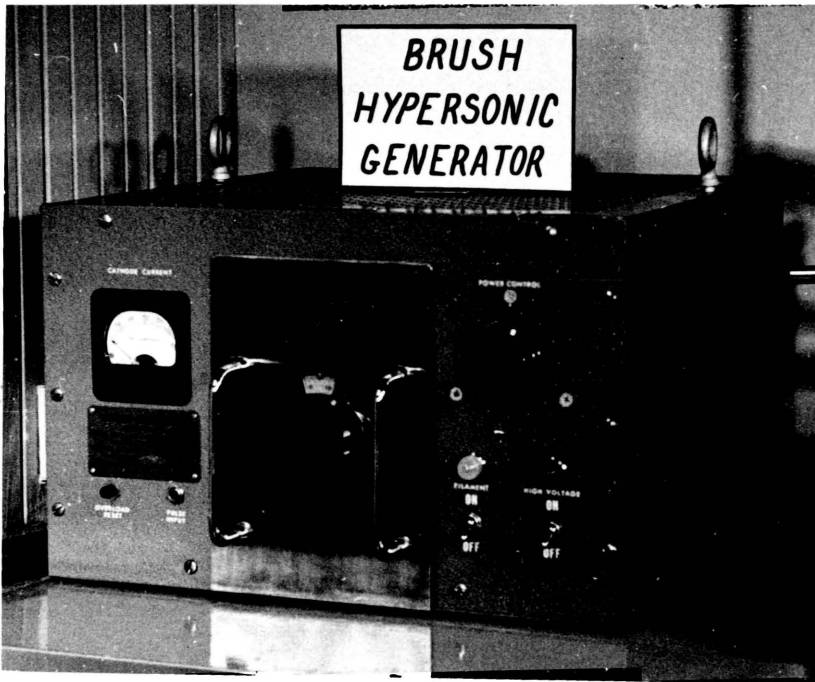


Figure 16. HYPERSONIC Generator



Figure 17. HYPERSONIC Generator, with Tuning
Drawer Removed

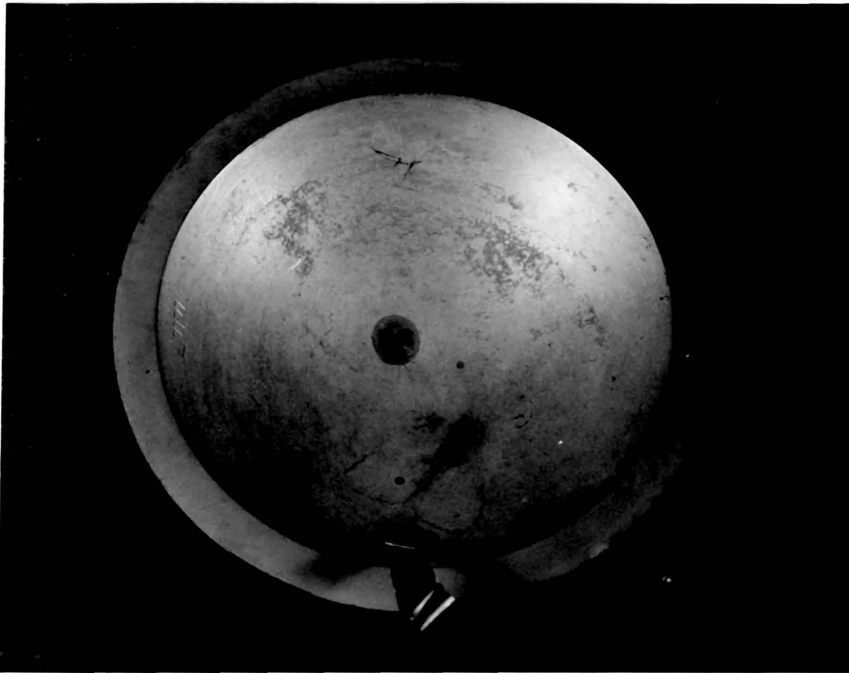
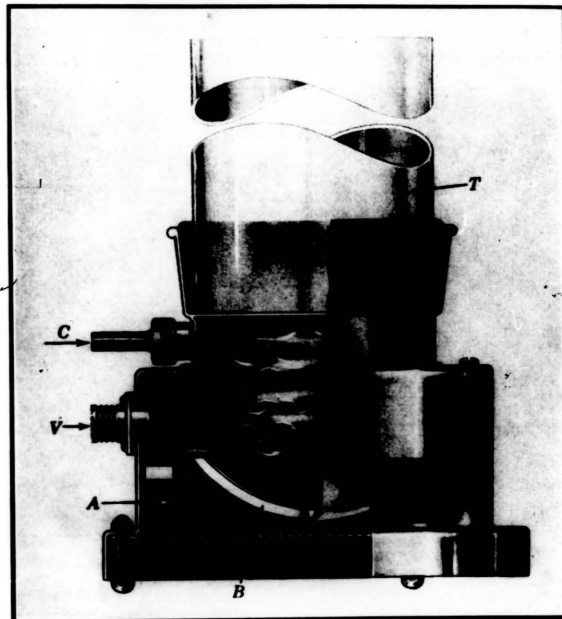


Figure 18. Unmounted Transducer



Commercial focusing transducer bowl. *A*, air-filled box; *B*, ceramic bowl; *C*, inlet for cooling liquid; *V*, connector for driving voltage; *T*, treatment chamber. Courtesy of Brush Development Company, Cleveland, Ohio.

Figure 19. Transducer Mounted in Metal Housing

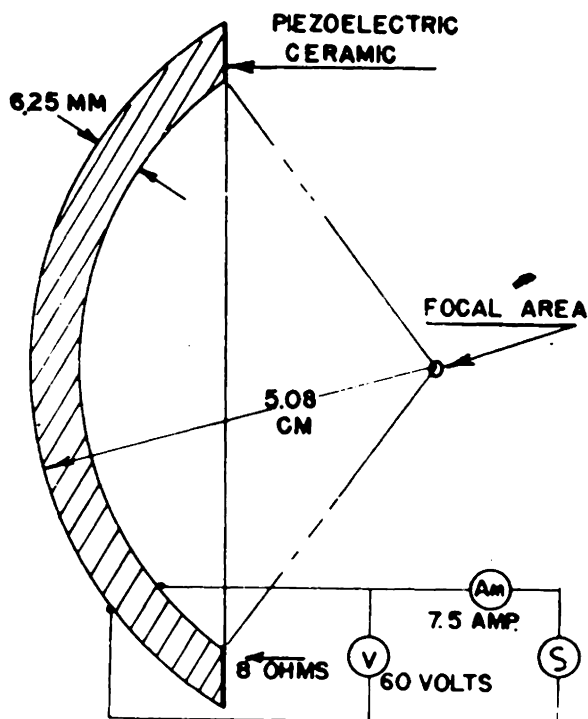


Figure 20. Electrical Conditions for Full Excitation of Bowl-shaped Barium Titanate Ceramic Transducer

Butterworth, J. W.: Kilowatts of Ultrasonic Power for Industry, "Ultrasonics—two symposia." Chem. Eng. Pro. Syn. Series, No 1, 47, pp. 69-75 (1951).

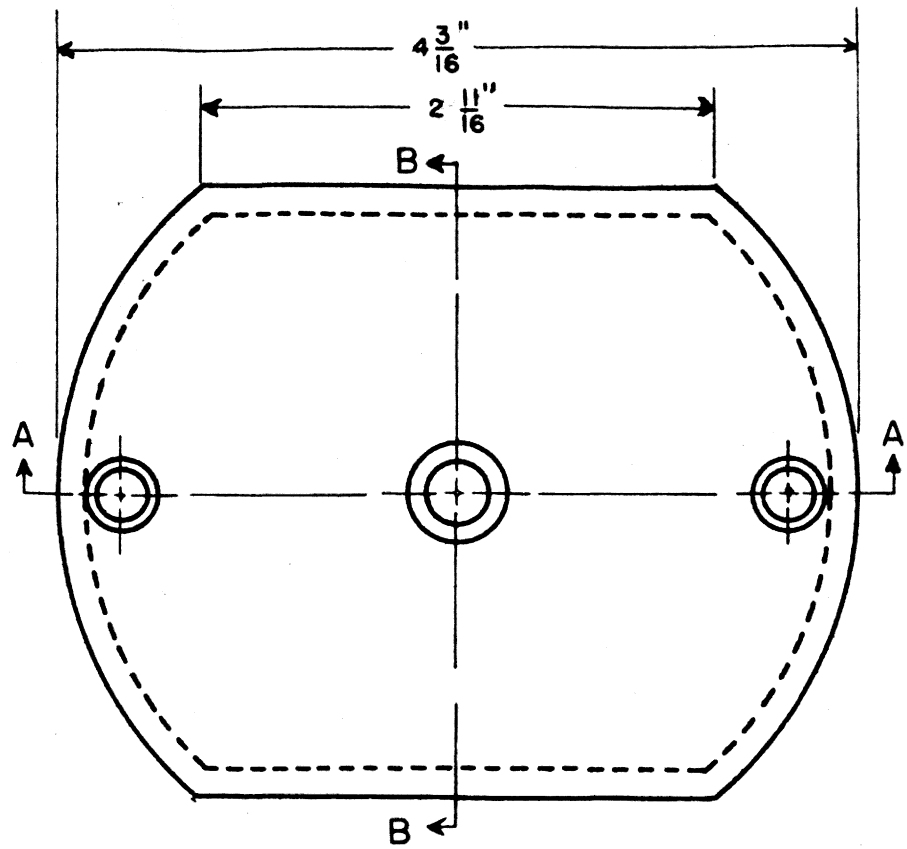
is shown in Figure 21. A picture of the constructed reactor is shown in Figure 22.

Control Panel, Frame and Tank Racks. Plans for the different sections of the major control panel are given in Figures 23, 24, and 25. Plans for the panel frame are given in Figure 26, and for the tank racks in Figure 27. Figure 28 shows the major control panel, extract and raffinate tanks, and area for reactor and settling column after they were incorporated into one unit.

Tubing and Fittings. A schematic flow diagram for ultrasonic extraction is shown in Figure 29. The sketch for feed and solvent tank tubing is given in Figure 30, for extract and raffinate tanks in Figure 31, and for reactor and settling column in Figure 32.

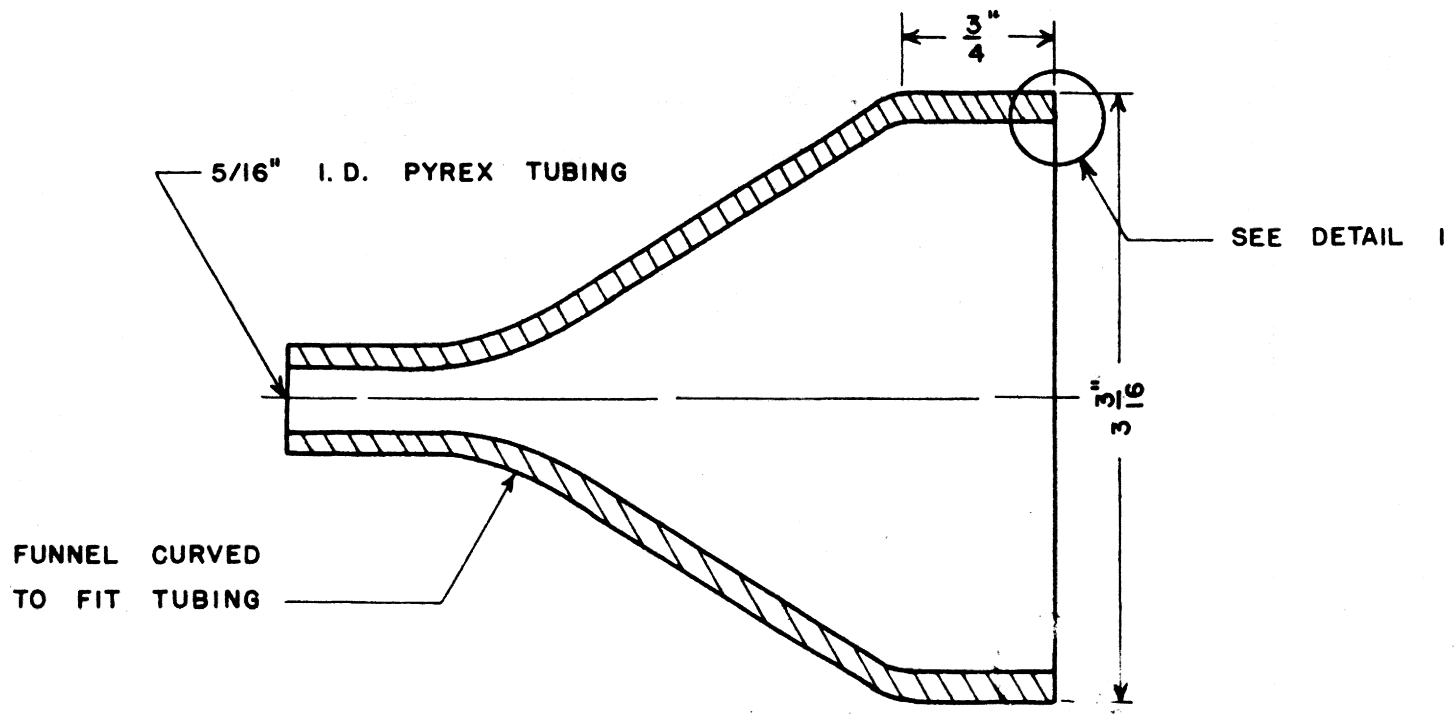
Sight Gages and Settling Column. Figure 33 shows details of sight gage construction, and Figure 34 gives details of settling column construction. Figure 35 shows the mounting used for feed and solvent tank sight gages. The installed settling column is shown in Figure 36.

Thermocouple and Electrical Wiring System. The thermocouple well containing a copper-constantan thermocouple is shown in Figure 37. Arrangement of the Rubicon Potentiometer in relationship to the control panel is illustrated

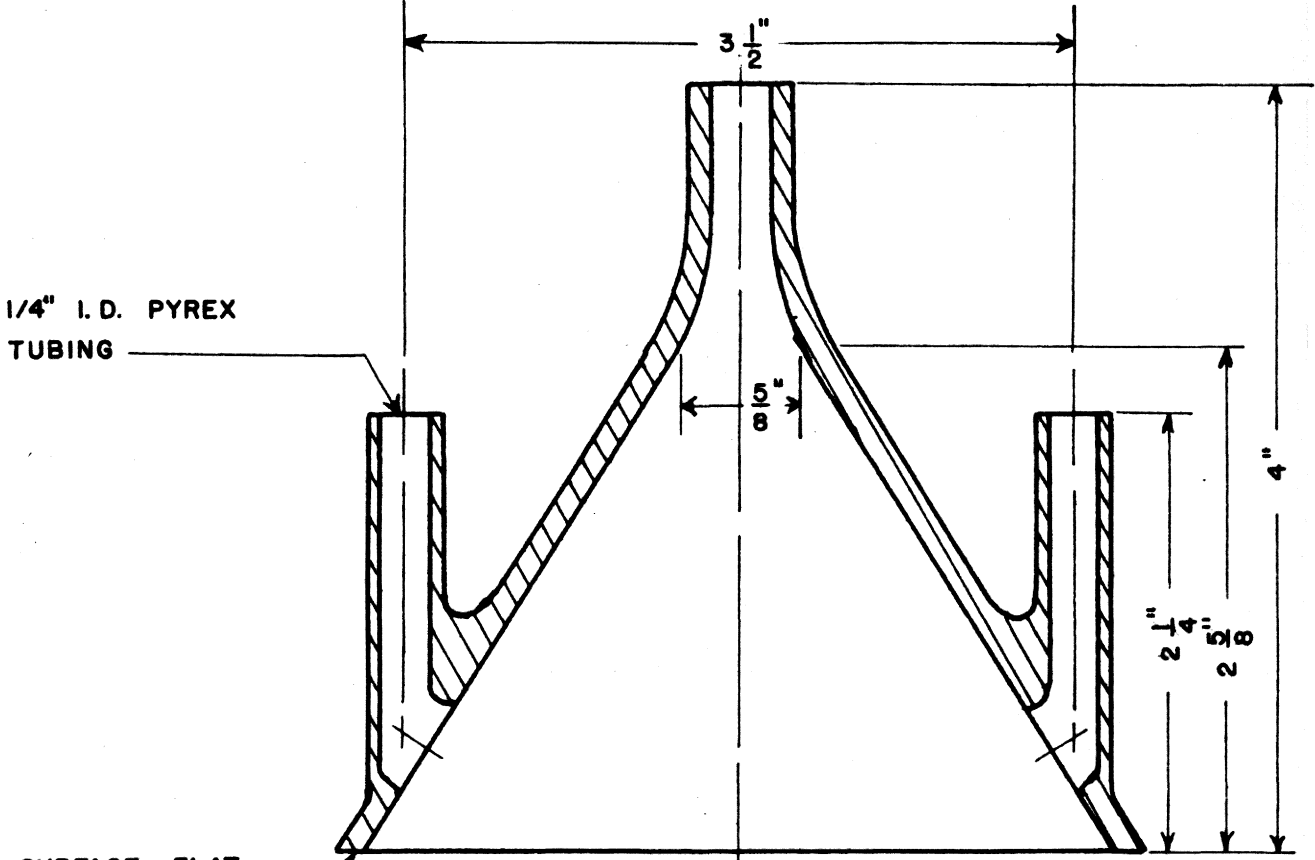


PLAN VIEW

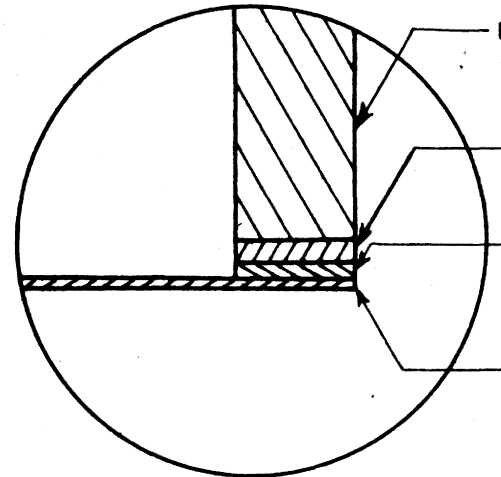
NOTE 1: REACTOR TO BE CONSTRUCTED FROM 60° PYREX FUNNEL.



SECTION B-B



SECTION A-A

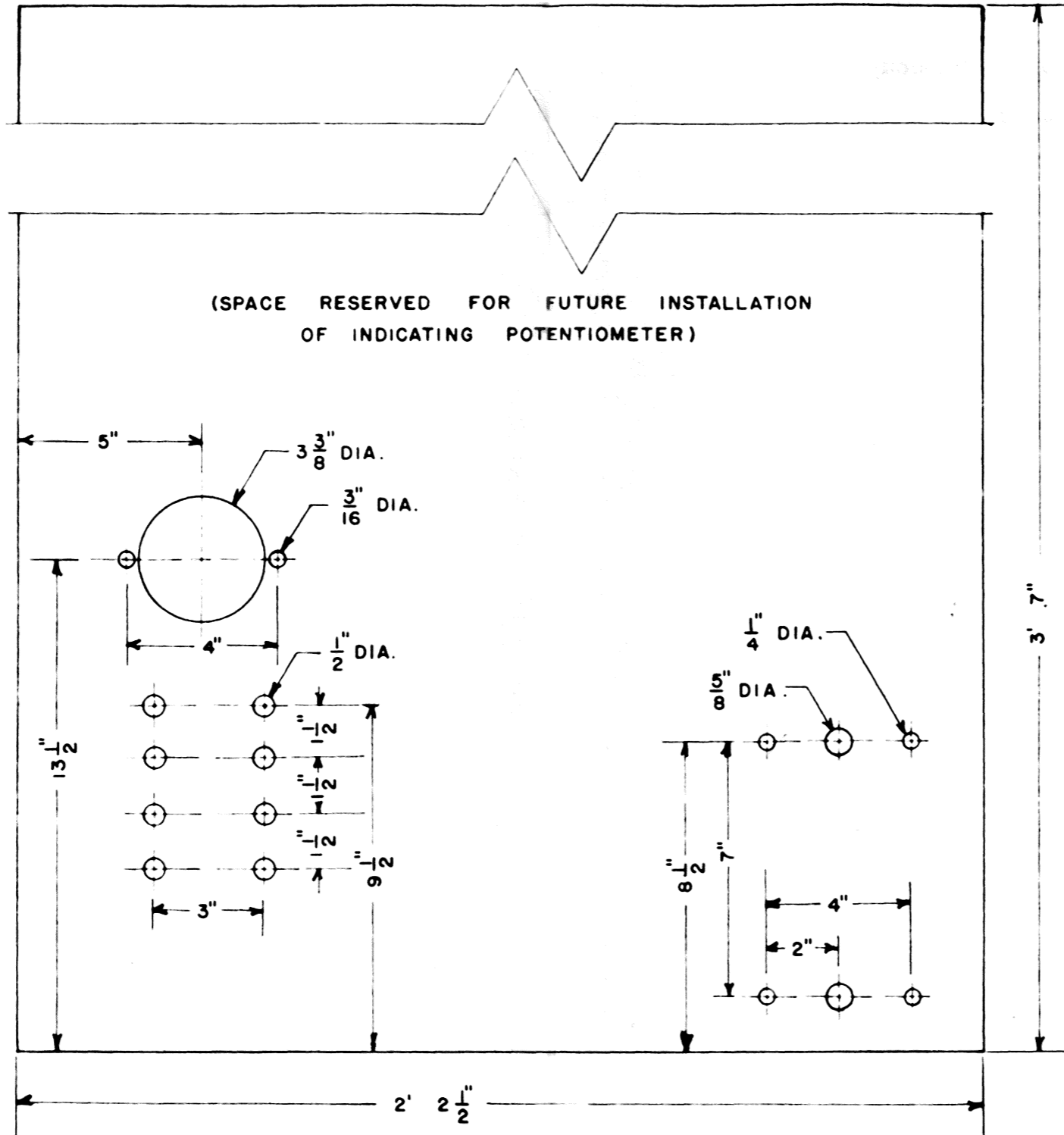


DETAIL 1
(NOT TO SCALE)
METHOD OF ATTACHING NICKEL SHEET
TO REACTOR BOTTOM

DEPARTMENT OF CHEMICAL ENGINEERING VIRGINIA POLYTECHNIC INSTITUTE BLACKSBURG, VIRGINIA		
MODIFIED REACTOR DESIGN FOR ULTRASONIC EXTRACTION		
SCALE: FULL	DATE	CASE NO: 55
DRAWN BY: <i>Haw.</i>	4/14/55	FILE NO: 599
CHECKED BY: <i>P.J.</i>	4/30/55	FIGURE NO: 21
APPROVED BY: <i>P.J.</i>	5/2/55	SHEET NO: 1 OF 1



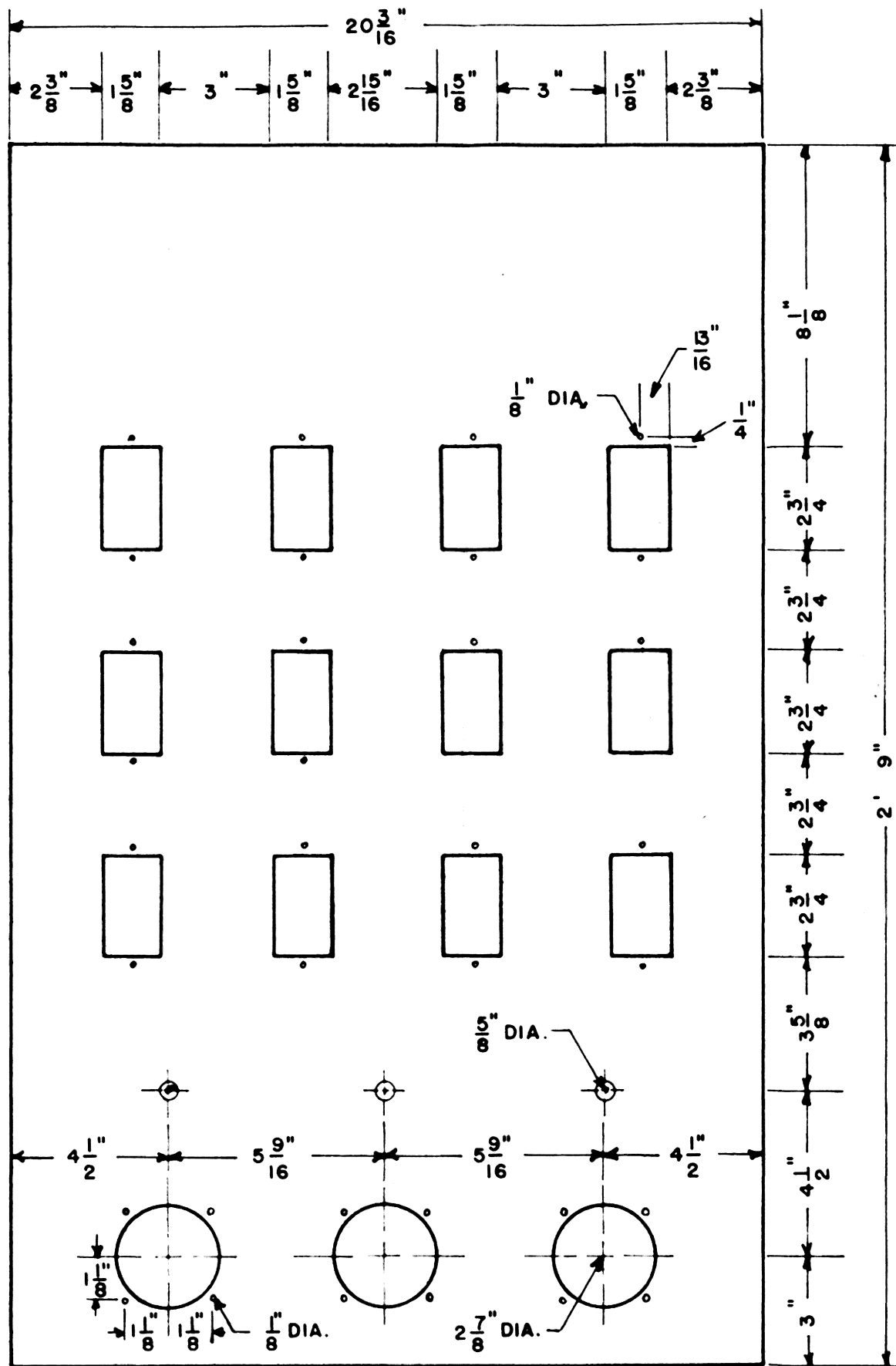
Figure 22. Glass Reactor of Modified Design



(SPACE RESERVED FOR FUTURE INSTALLATION
OF INDICATING POTENTIOMETER)

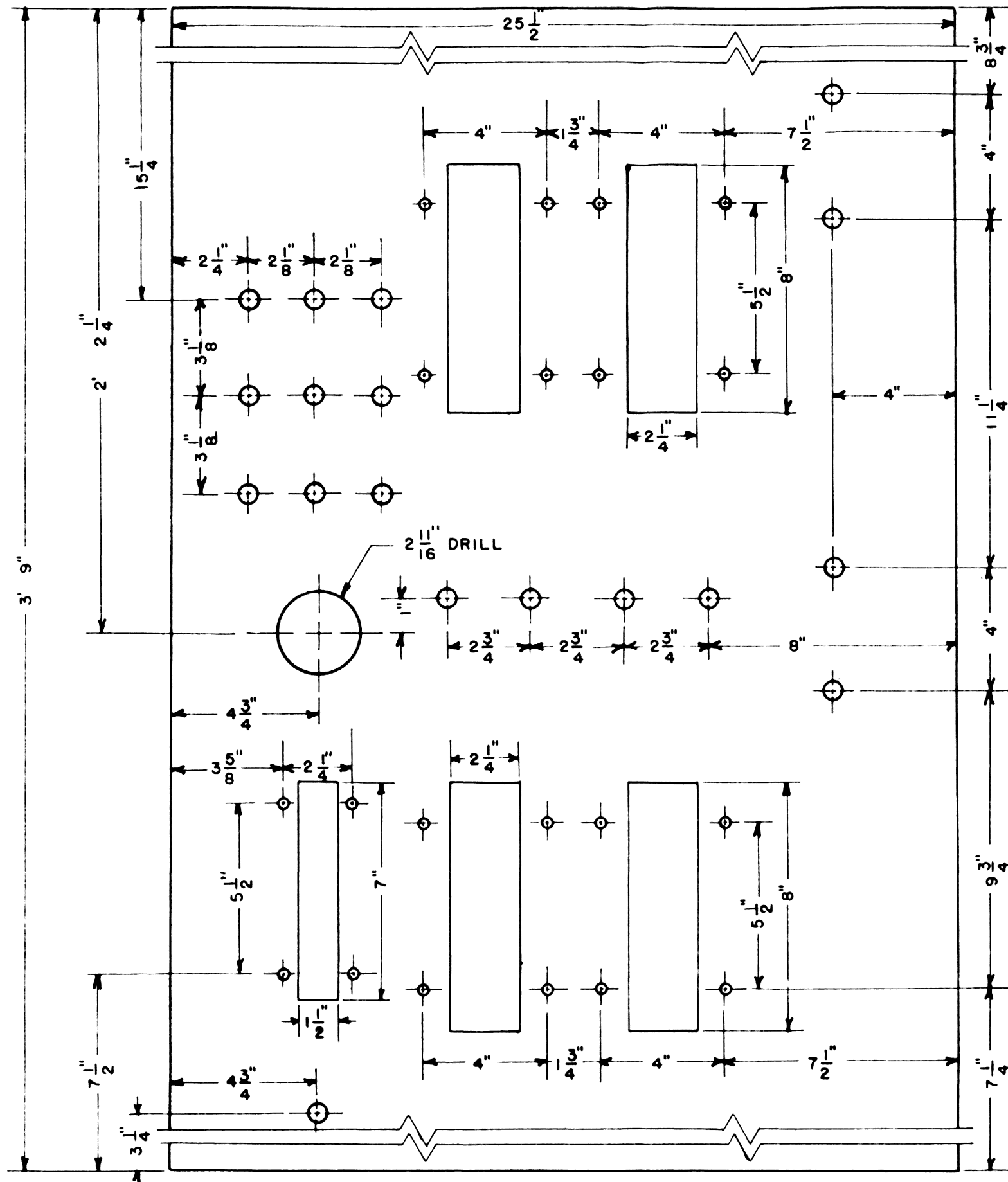
NOTE: PANEL CONSTRUCTED OF $\frac{1}{2}$ "
5 PLY PLYWOOD



DEPARTMENT OF CHEMICAL ENGINEERING VIRGINIA POLYTECHNIC INSTITUTE BLACKSBURG, VIRGINIA		
DRILLING PLAN FOR THERMOCOUPLE PANEL		
ULTRASONIC EXTRACTION		
SCALE: 1" = 4"	DATE	CASE NO: 55
DRAWN BY: <i>Hood</i>	5/5/55	FILE NO: 599
CHECKED BY: <i>DJ</i>	5/6/55	FIGURE NO: 23
APPROVED BY: <i>DJ</i>	5/6/55	SHEET NO: 1 OF 1



NOTE 1: PANEL CONSTRUCTED OF 5 PLY,
 $\frac{1}{2}$ " PLYWOOD

DEPARTMENT OF CHEMICAL ENGINEERING VIRGINIA POLYTECHNIC INSTITUTE BLACKSBURG, VIRGINIA		
DRILLING PLAN FOR ELECTRIC PANEL		
ULTRASONIC EXTRACTION		
SCALE: 1" = 4"	DATE	CASE NO: 55
DRAWN BY: <i>Haw.</i>	4/23/55	FILE NO: 599
CHECKED BY: <i>D.J.</i>	4/30/55	FIGURE NO: 24
APPROVED BY: <i>D.J.</i>	5/2/55	SHEET NO: 1 OF 1



- NOTE 1: PANEL CONSTRUCTION OF 5 PLY, 1/2" PLYWOOD
- NOTE 2: OPENINGS FOR VALVES, , ARE 5/8" DIAMETER
- NOTE 3: OPENINGS FOR MOUNTING BOLTS, , ARE 9/32" DIAMETER

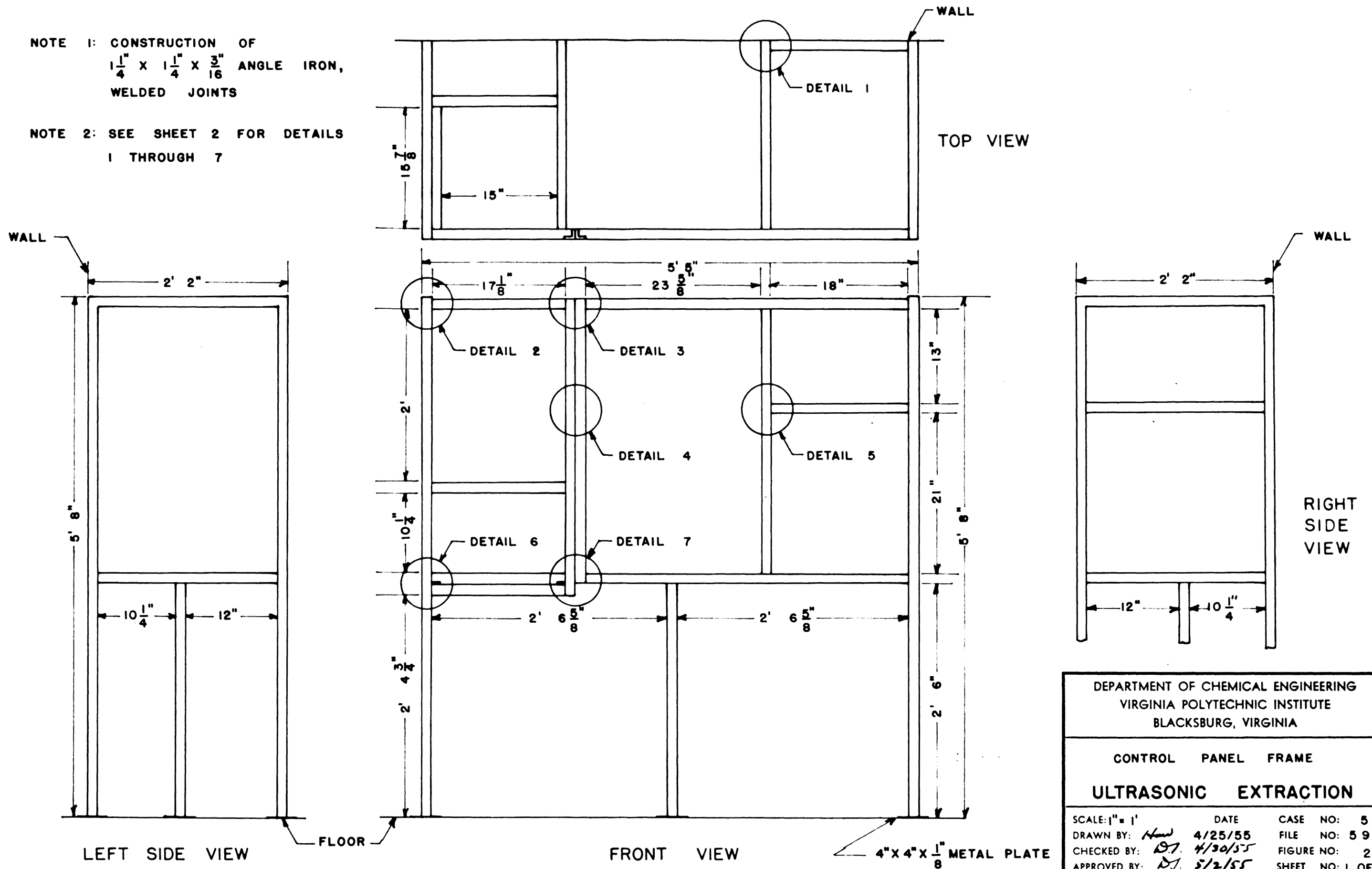
DEPARTMENT OF CHEMICAL ENGINEERING
 VIRGINIA POLYTECHNIC INSTITUTE
 BLACKSBURG, VIRGINIA

DRILLING PLAN FOR HYDRAULIC
 PANEL FOR
ULTRASONIC EXTRACTION

SCALE: 1" = 4"	DATE	CASE NO: 55
DRAWN BY: <i>Haw.</i>	5/6/55	FILE NO: 599
CHECKED BY: <i>P.J.</i>	5/6/55	FIGURE NO: 25
APPROVED BY: <i>P.J.</i>	5/8/55	SHEET NO: 1 OF 1

NOTE 1: CONSTRUCTION OF
 $1\frac{1}{4}$ " X $1\frac{1}{4}$ " X $\frac{3}{16}$ " ANGLE IRON,
 WELDED JOINTS

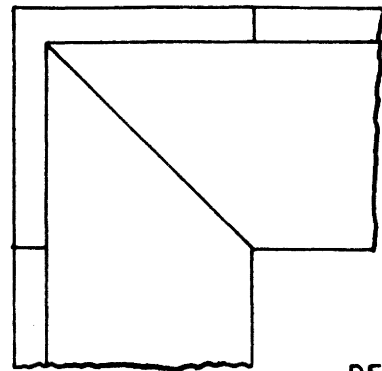
NOTE 2: SEE SHEET 2 FOR DETAILS
 1 THROUGH 7



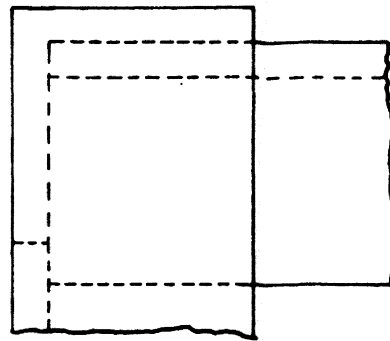
DEPARTMENT OF CHEMICAL ENGINEERING
 VIRGINIA POLYTECHNIC INSTITUTE
 BLACKSBURG, VIRGINIA

CONTROL PANEL FRAME
 ULTRASONIC EXTRACTION

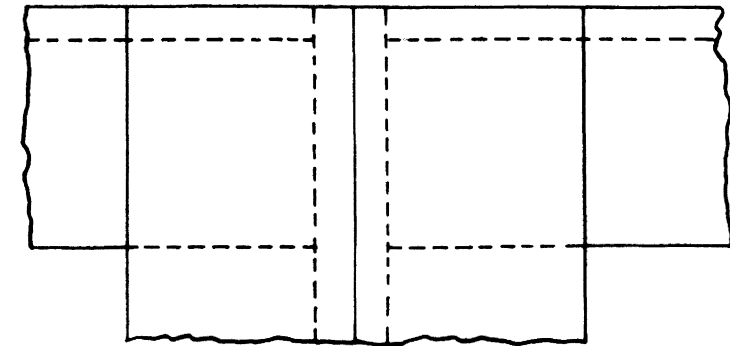
SCALE: 1" = 1'	DATE	CASE NO: 55
DRAWN BY: <i>Haw</i>	4/25/55	FILE NO: 599
CHECKED BY: <i>DJ</i>	4/30/55	FIGURE NO: 26
APPROVED BY: <i>DJ</i>	5/2/55	SHEET NO: 1 OF 2



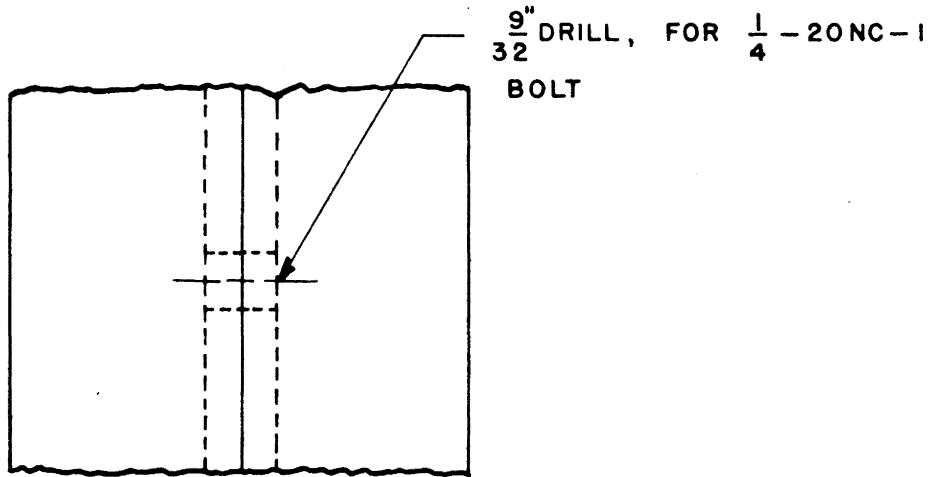
DETAIL 1



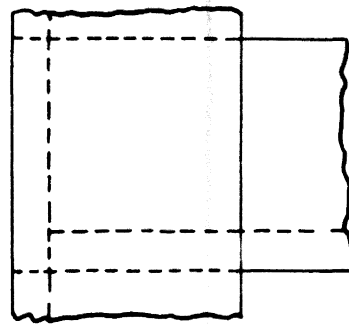
DETAIL 2



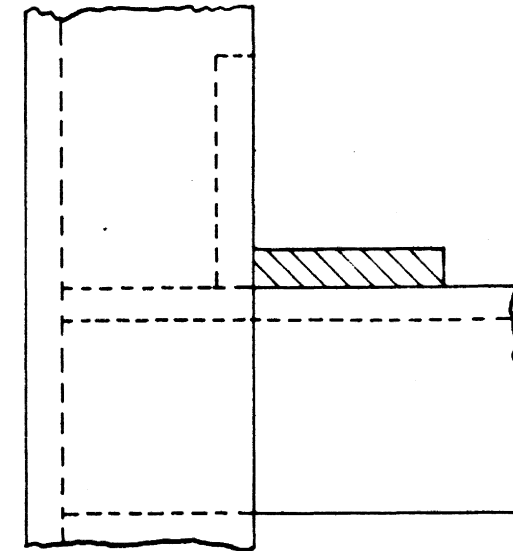
DETAIL 3



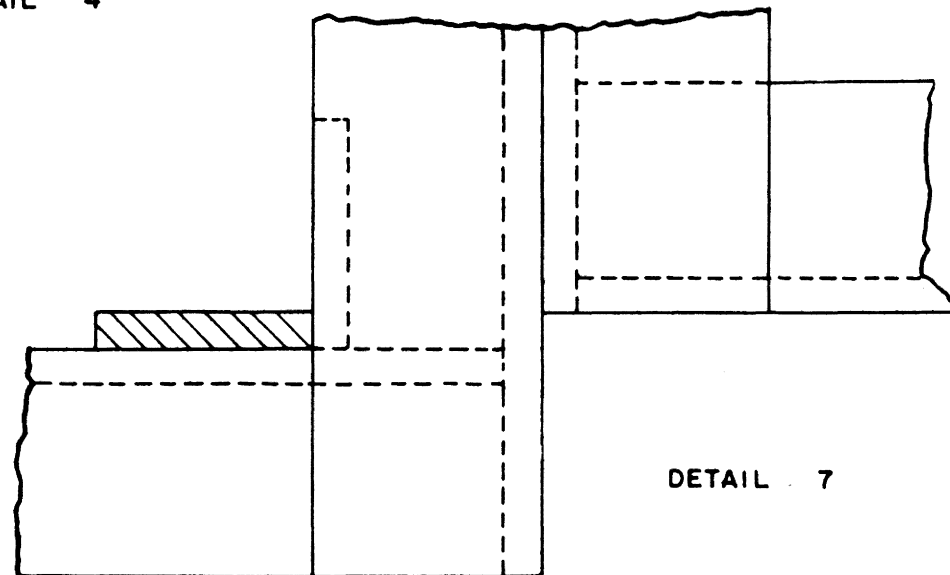
DETAIL 4



DETAIL 5



DETAIL 6



DETAIL 7

NOTE 1: DETAIL 4 ILLUSTRATES CONNECTION OF TWO SECTIONS OF FRAME (FOUR SUCH JOINTS REQUIRED)

DEPARTMENT OF CHEMICAL ENGINEERING
VIRGINIA POLYTECHNIC INSTITUTE
BLACKSBURG, VIRGINIA

CONTROL PANEL FRAME
(DETAILS 1 THROUGH 7)

ULTRASONIC EXTRACTION

SCALE: 1" = 1"	DATE	CASE NO: 55
DRAWN BY: <i>How.</i>	4/25/55	FILE NO: 599
CHECKED BY: <i>D.J.</i>	4/30/55	FIGURE NO: 26
APPROVED BY: <i>D.J.</i>	5/2/55	SHEET NO: 2 OF 2

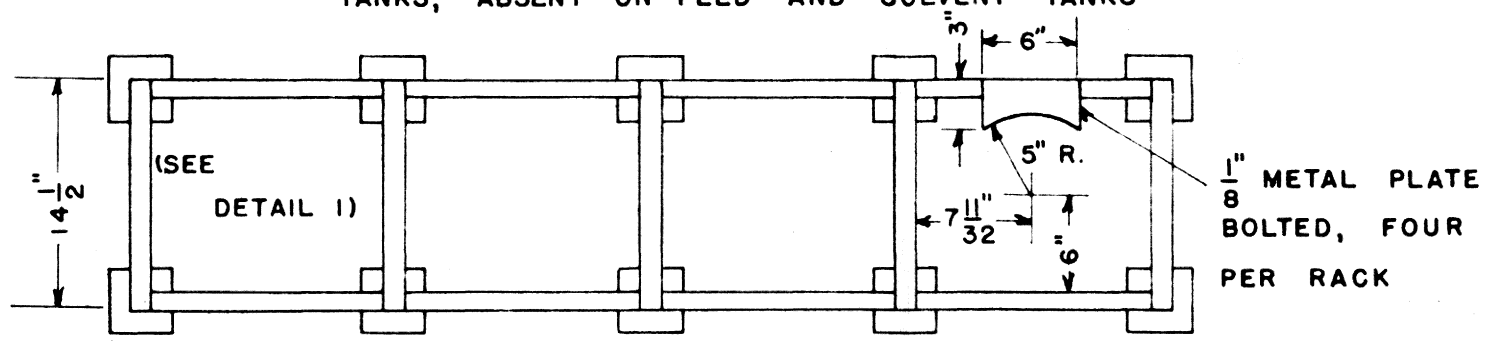
NOTE 1: CONSTRUCTION OF $1\frac{1}{4} \times 1\frac{1}{4} \times \frac{3}{16}$ ANGLE IRON, WELDED JOINTS

NOTE 5: SEE SHEET 2 FOR DETAILS 1 THROUGH 4

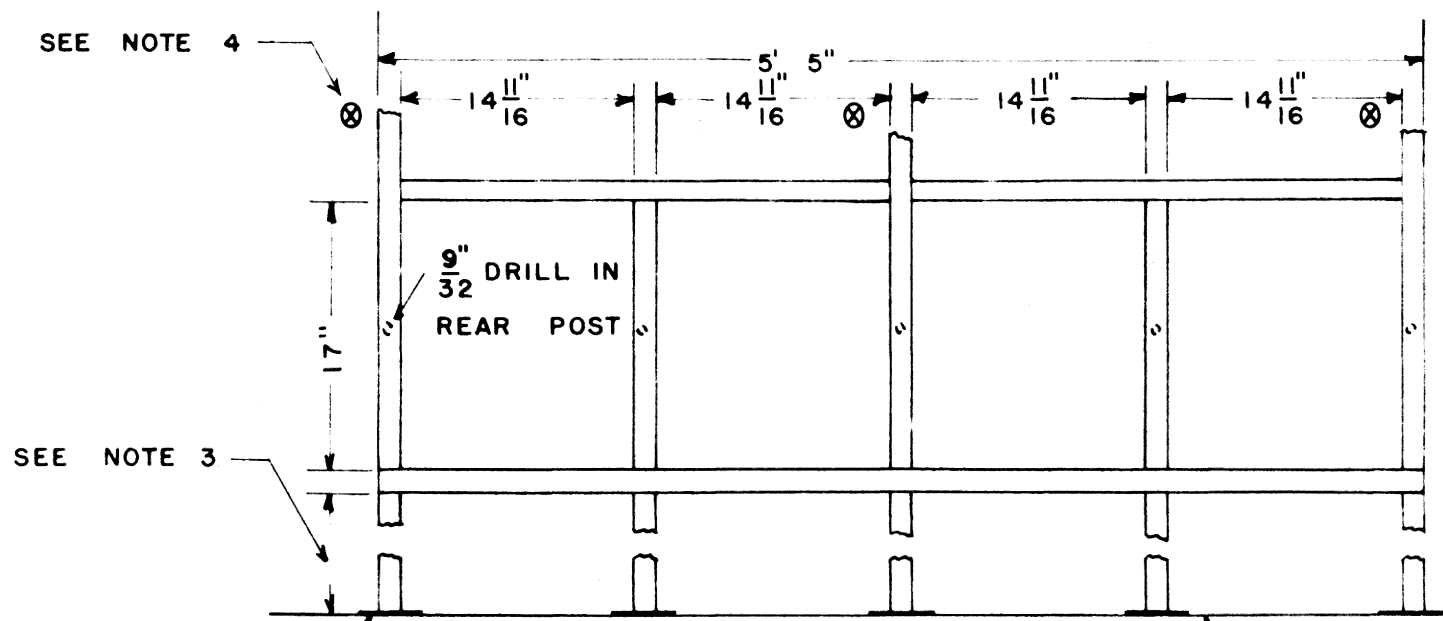
NOTE 2: THREE CENTER CROSS PIECES, $14\frac{1}{2}$ " LONG, PLACED FLAT SIDE UP TO FORM BEARING SURFACE

NOTE 3: DIMENSION IS 9" FOR FEED AND SOLVENT TANKS, $5\frac{1}{2}$ " FOR EXTRACT AND RAFFINATE TANKS

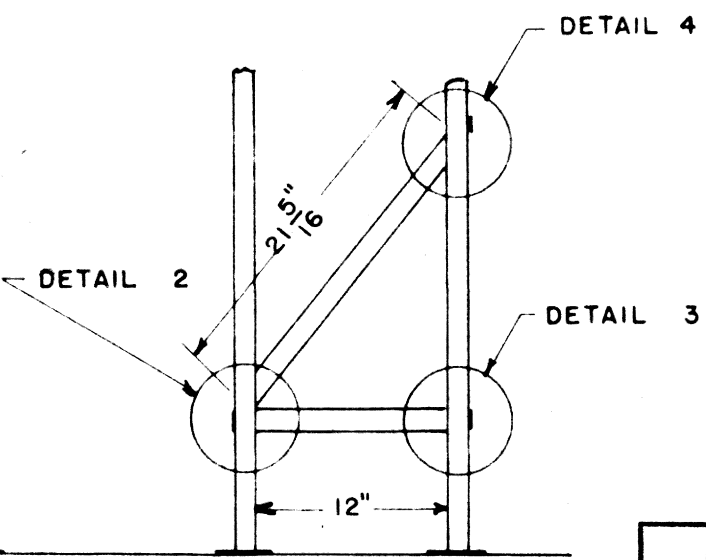
NOTE 4: POSTS \otimes EXTEND FROM PANEL FRAME FOR EXT. AND RAFF. TANKS, ABSENT ON FEED AND SOLVENT TANKS



TOP VIEW



FRONT VIEW

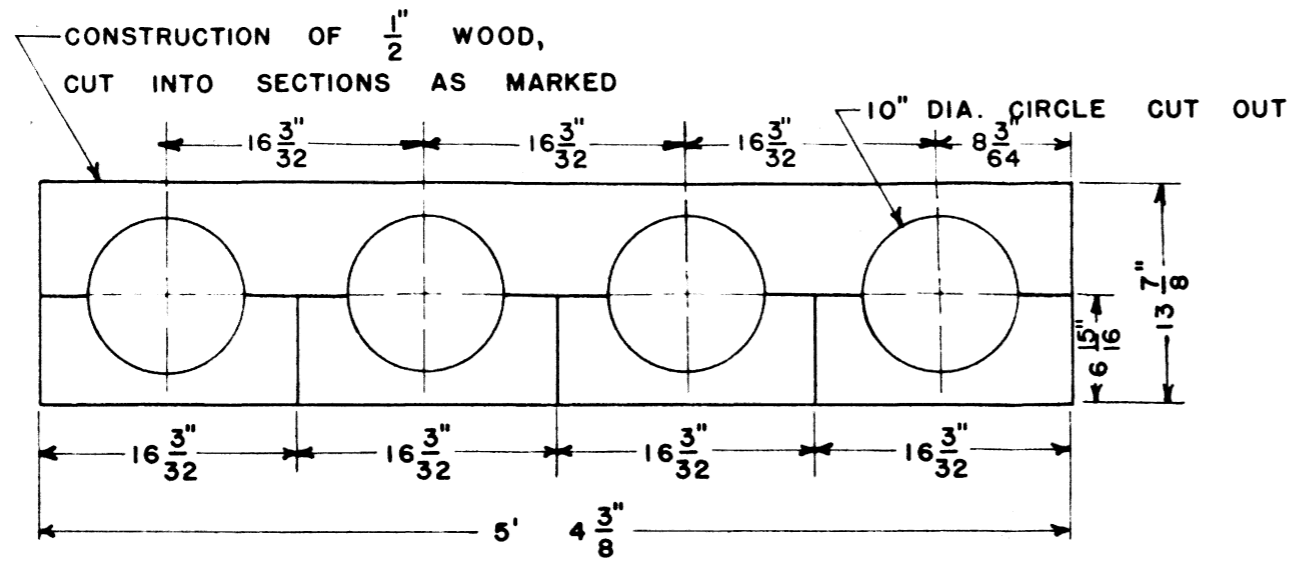


SIDE VIEW

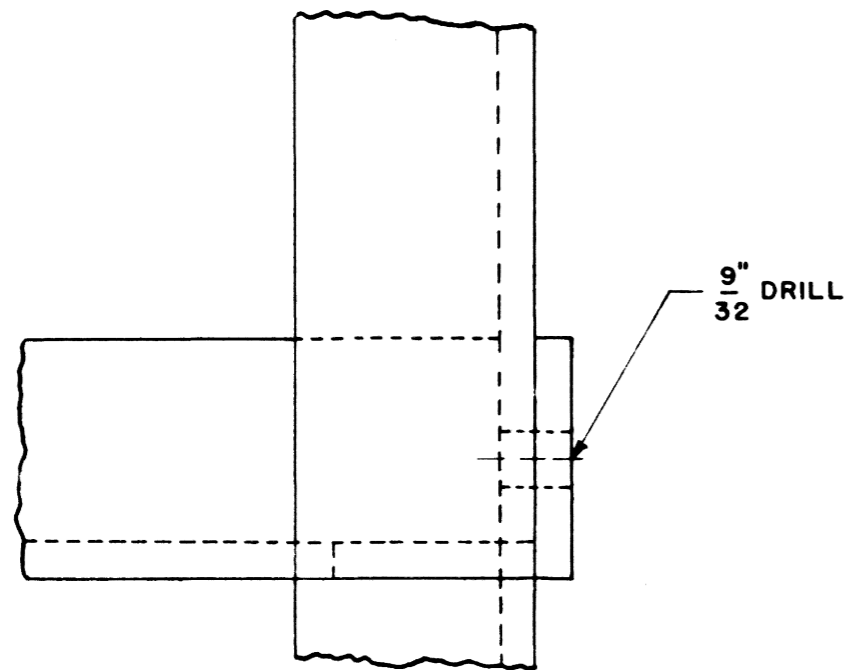
DEPARTMENT OF CHEMICAL ENGINEERING
 VIRGINIA POLYTECHNIC INSTITUTE
 BLACKSBURG, VIRGINIA

TANK RACKS FOR
ULTRASONIC EXTRACTION

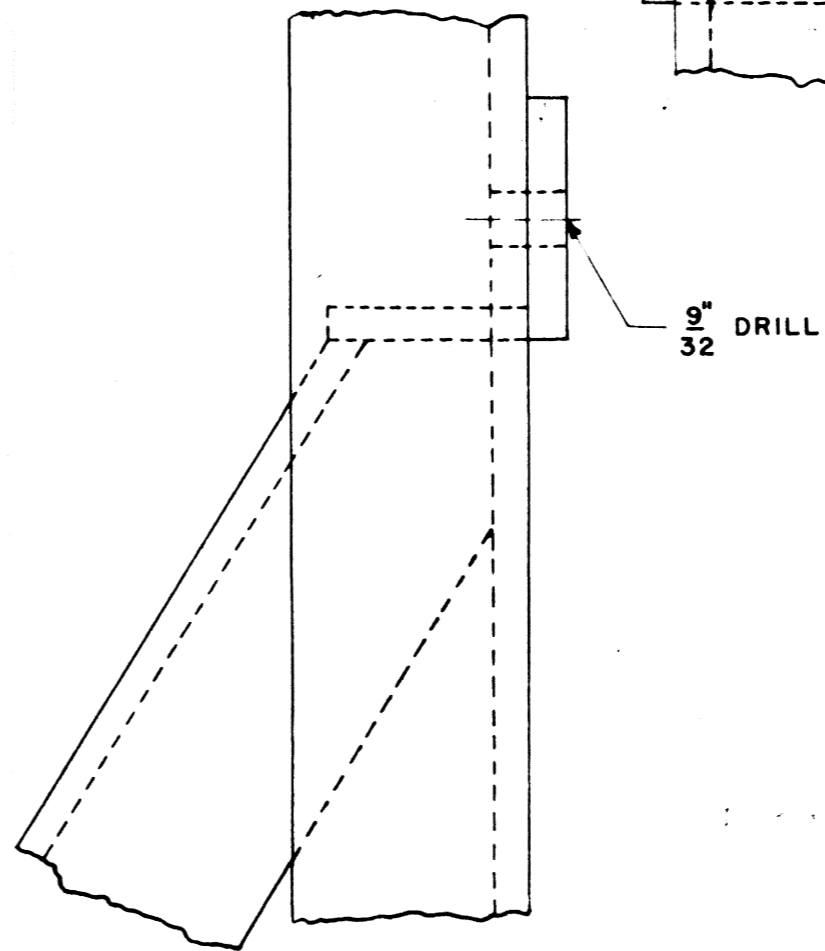
SCALE: 1" = 1' DATE: 5/2/55 CASE NO: 55
 DRAWN BY: *Haw* FILE NO: 599
 CHECKED BY: *DJ* 5/4/55 FIGURE NO: 27
 APPROVED BY: *PJ* 5/6/55 SHEET NO: 1 OF 2



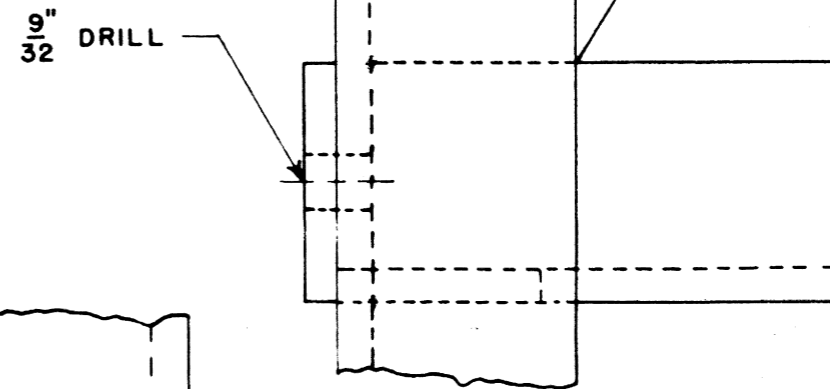
DETAIL 1
(SCALE: 1" = 1')



DETAIL 3



DETAIL 4

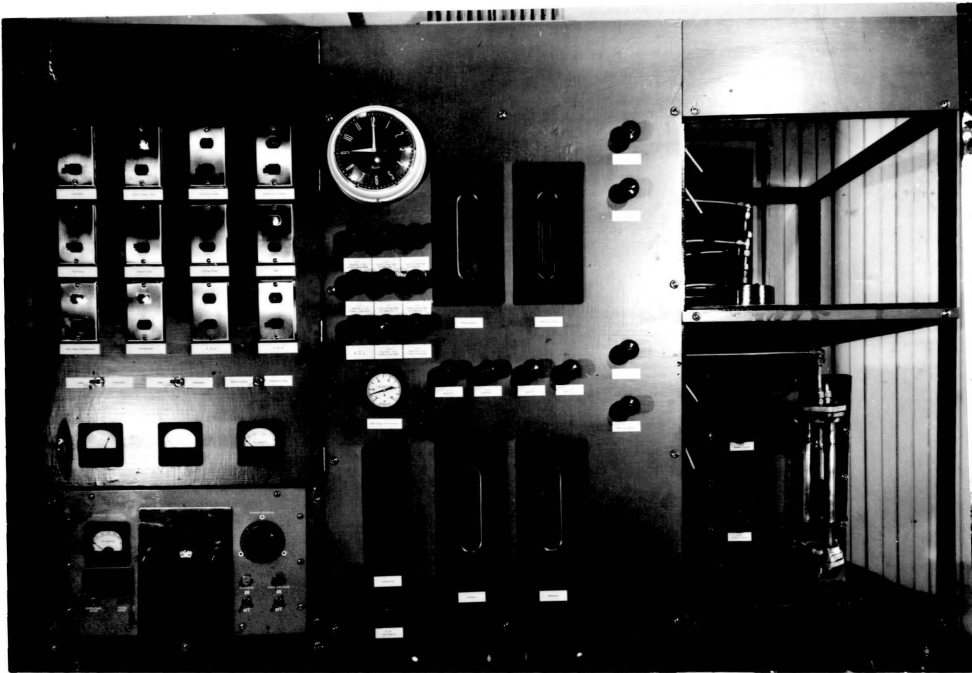


DETAIL 2

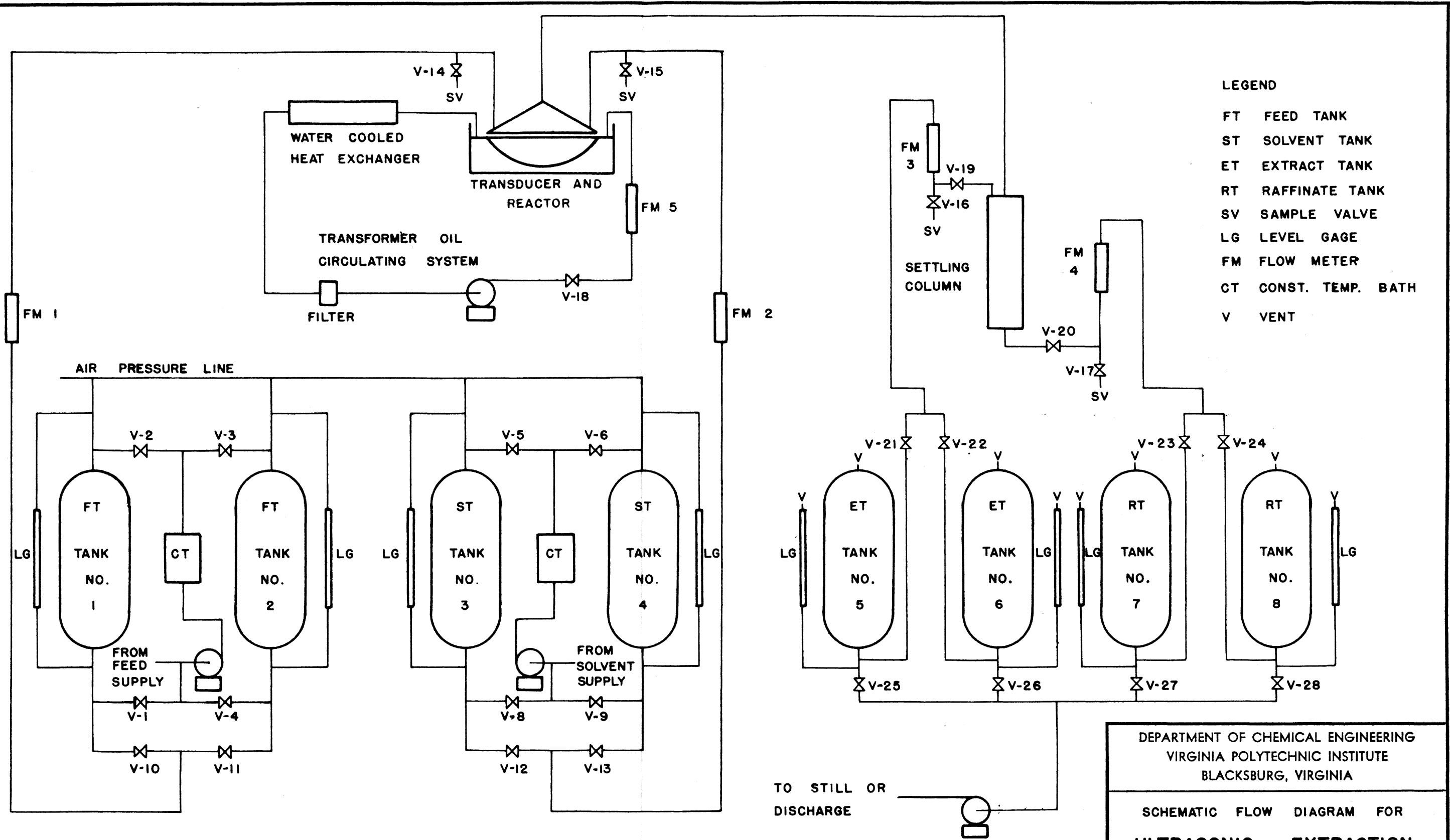
DEPARTMENT OF CHEMICAL ENGINEERING
VIRGINIA POLYTECHNIC INSTITUTE
BLACKSBURG, VIRGINIA

TANK RACKS FOR
ULTRASONIC EXTRACTION

SCALE: 1" = 1"	DATE	CASE NO: 55
DRAWN BY: <i>Haw</i>	5/2/55	FILE NO: 599
CHECKED BY: <i>D.J. 5/4/55</i>		FIGURE NO: 27
APPROVED BY: <i>D.J. 5/6/55</i>		SHEET NO: 2 OF 2



**Figure 26. Completed Control Panel and Adjacent
Equipment Sub-assemblies**



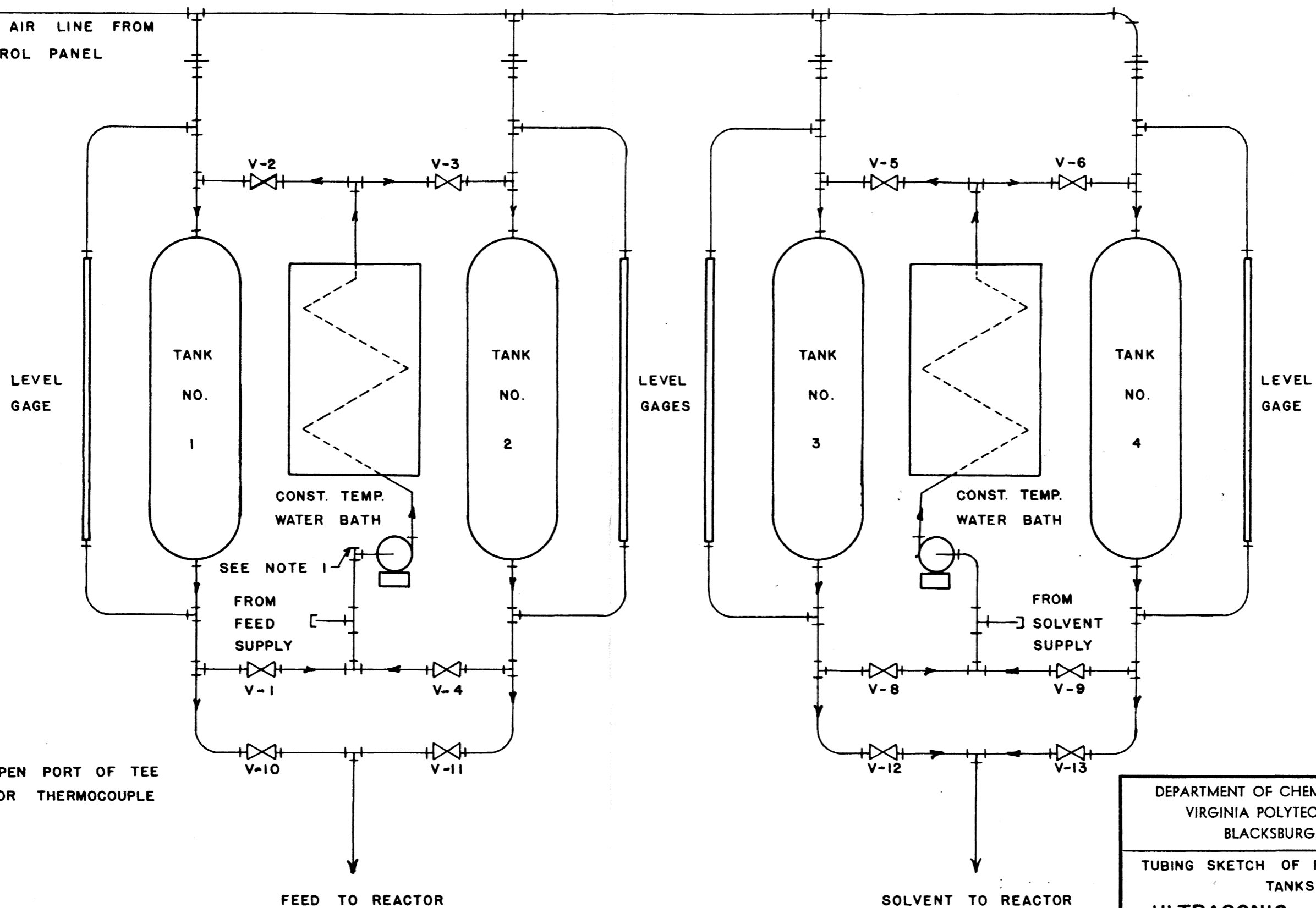
- LEGEND**
- FT FEED TANK
 - ST SOLVENT TANK
 - ET EXTRACT TANK
 - RT RAFFINATE TANK
 - SV SAMPLE VALVE
 - LG LEVEL GAGE
 - FM FLOW METER
 - CT CONST. TEMP. BATH
 - V VENT

DEPARTMENT OF CHEMICAL ENGINEERING
 VIRGINIA POLYTECHNIC INSTITUTE
 BLACKSBURG, VIRGINIA

**SCHEMATIC FLOW DIAGRAM FOR
 ULTRASONIC EXTRACTION**

SCALE: NONE DATE: 4/13/55 CASE NO: 55
 DRAWN BY: *Haw.* FILE NO: 599
 CHECKED BY: *DJ.* 4/30/55 FIGURE NO: 29
 APPROVED BY: *PJ.* 5/2/55 SHEET NO: 1 OF 1

1/4" PIPE, AIR LINE FROM
AIR CONTROL PANEL



NOTE 1: OPEN PORT OF TEE
FOR THERMOCOUPLE

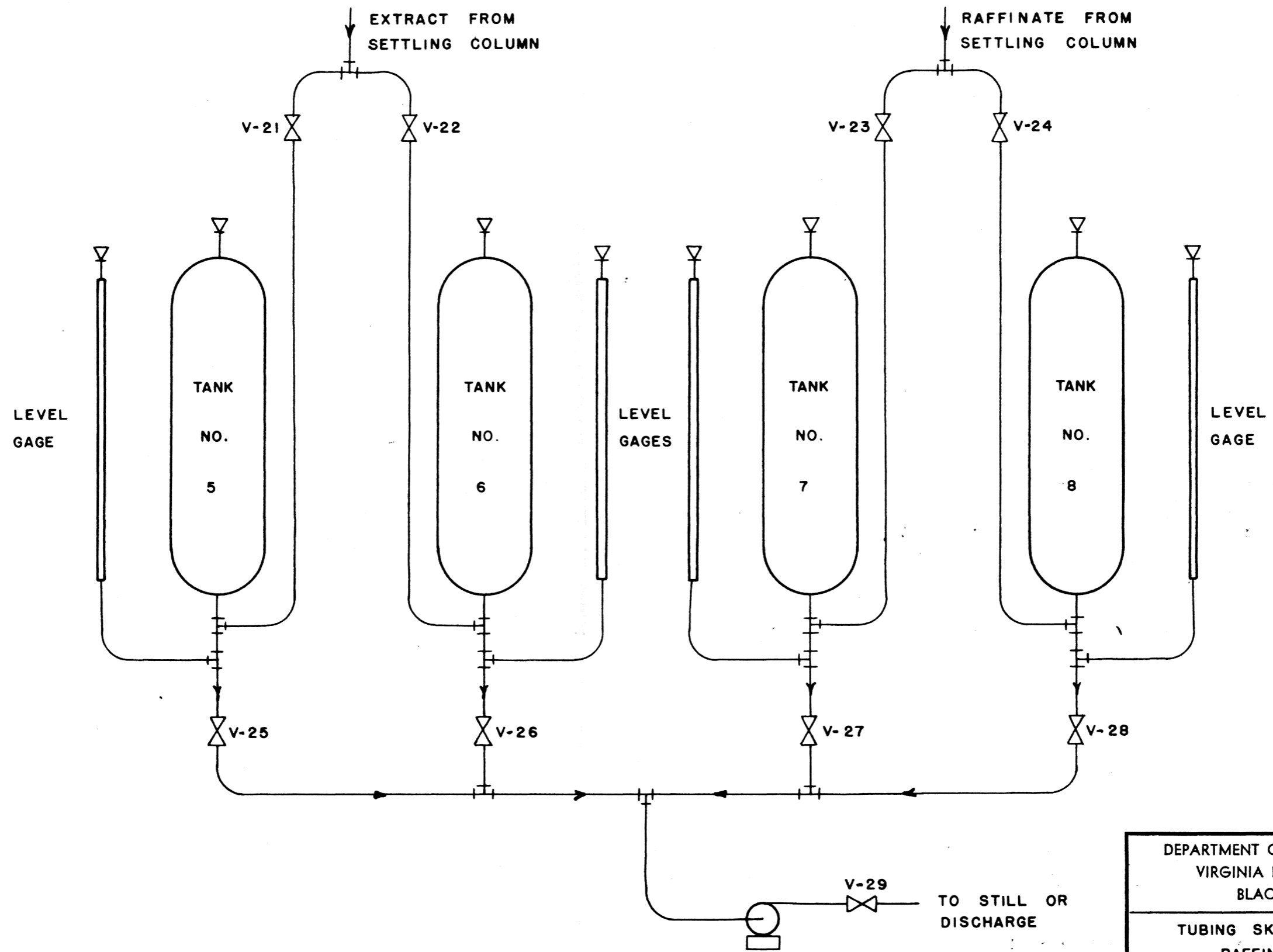
FEED TO REACTOR

SOLVENT TO REACTOR

DEPARTMENT OF CHEMICAL ENGINEERING
VIRGINIA POLYTECHNIC INSTITUTE
BLACKSBURG, VIRGINIA

TUBING SKETCH OF FEED & SOLVENT
TANKS FOR
ULTRASONIC EXTRACTION

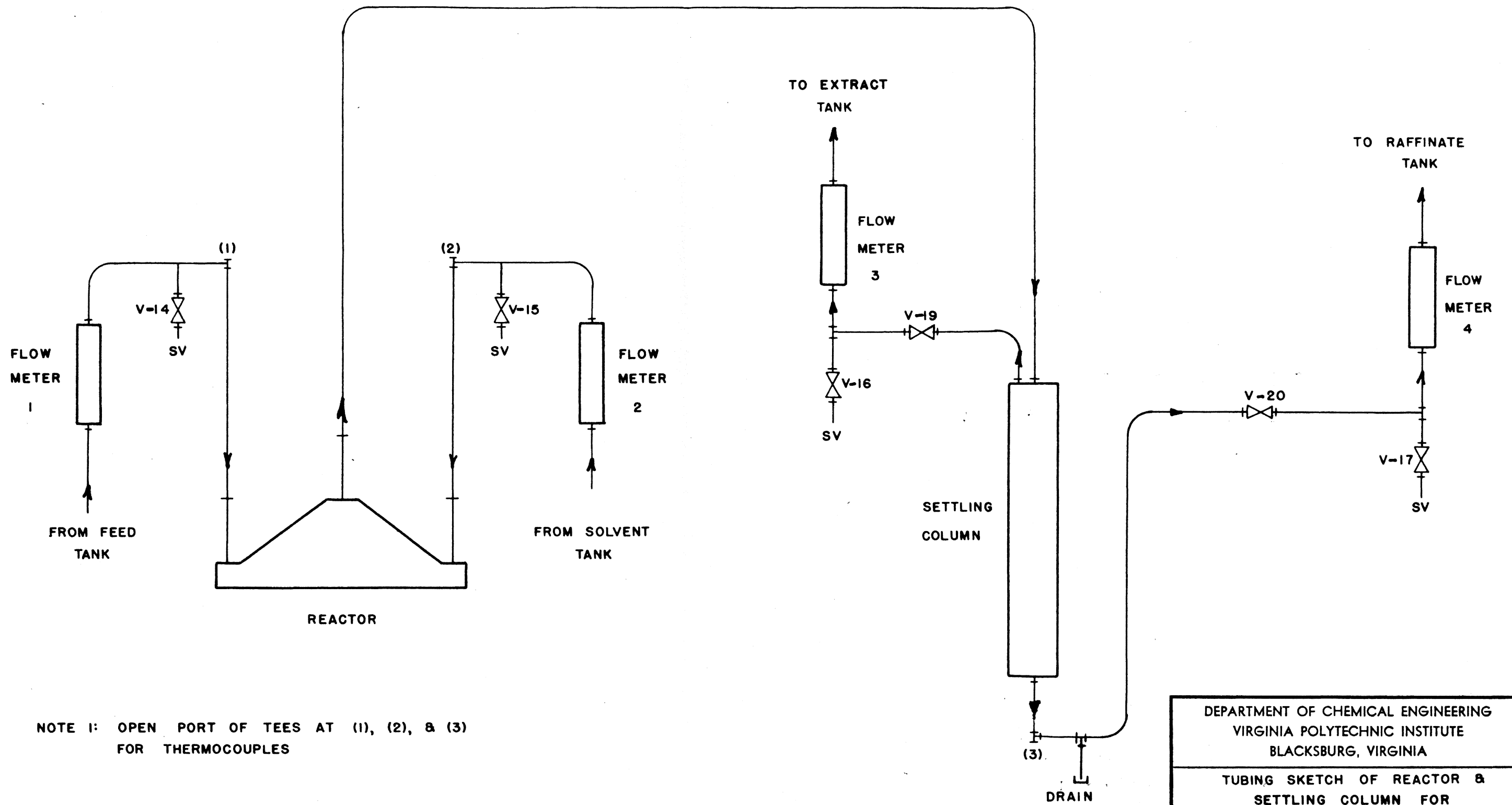
SCALE: NONE	DATE	CASE NO: 55
DRAWN BY: <i>How</i>	4/13/55	FILE NO: 599
CHECKED BY: <i>DJ</i>	4/30/55	FIGURE NO: 30
APPROVED BY: <i>DJ</i>	5/2/55	SHEET NO: 1 OF 1



DEPARTMENT OF CHEMICAL ENGINEERING
 VIRGINIA POLYTECHNIC INSTITUTE
 BLACKSBURG, VIRGINIA

TUBING SKETCH OF EXTRACT &
 RAFFINATE TANKS FOR
 ULTRASONIC EXTRACTION

SCALE: NONE	DATE	CASE NO: 55
DRAWN BY: <i>Haw</i>	4/12/55	FILE NO: 599
CHECKED BY: <i>DJ</i>	4/30/55	FIGURE NO: 31
APPROVED BY: <i>DJ</i>	5/2/55	SHEET NO: 1 OF 1

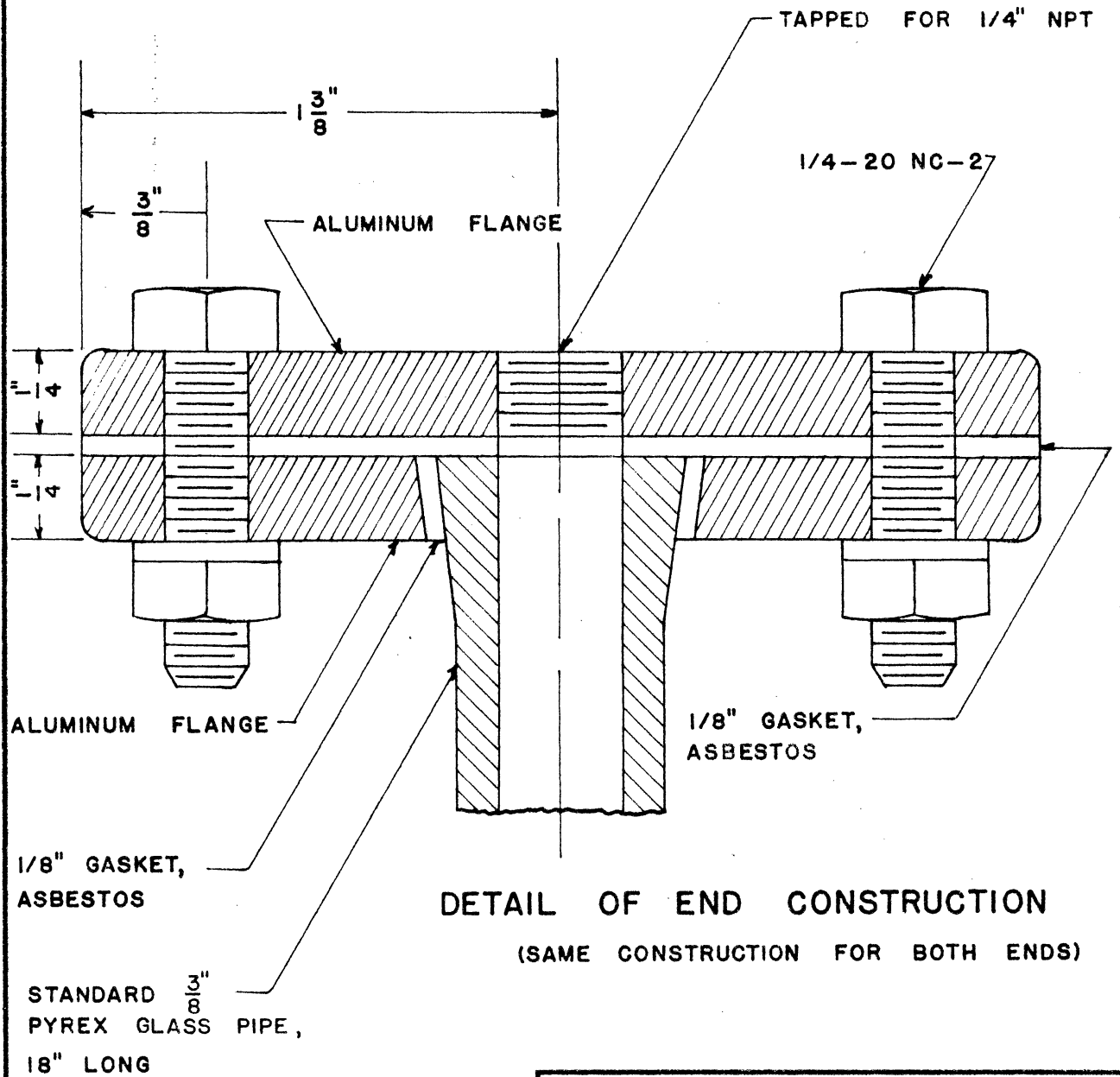


NOTE 1: OPEN PORT OF TEES AT (1), (2), & (3)
FOR THERMOCOUPLES

DEPARTMENT OF CHEMICAL ENGINEERING
VIRGINIA POLYTECHNIC INSTITUTE
BLACKSBURG, VIRGINIA

TUBING SKETCH OF REACTOR &
SETTLING COLUMN FOR
ULTRASONIC EXTRACTION

SCALE: NONE	DATE: 4/13/55	CASE NO: 55
DRAWN BY: <i>Handwritten</i>	4/13/55	FILE NO: 599
CHECKED BY: <i>D.J.</i>	4/30/55	FIGURE NO: 32
APPROVED BY: <i>D.T.</i>	5/2/55	SHEET NO: 1 OF 1



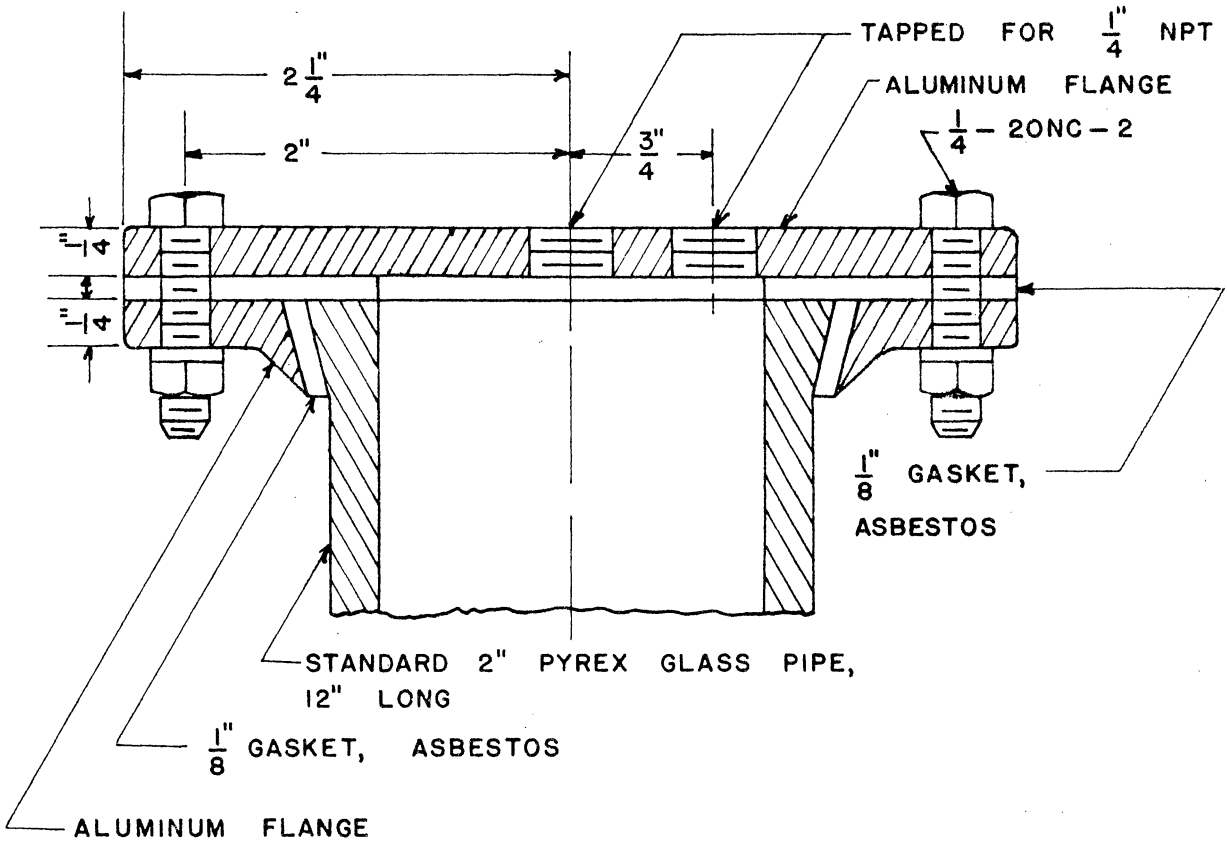
DEPARTMENT OF CHEMICAL ENGINEERING
VIRGINIA POLYTECHNIC INSTITUTE
BLACKSBURG, VIRGINIA

SIGHT GAGE FOR

ULTRASONIC EXTRACTION

SCALE: 1" = 1/2"	DATE	CASE NO:	55
DRAWN BY: <i>Hand</i>	4/21/55	FILE NO:	599
CHECKED BY: <i>DT</i>	4/30/55	FIGURE NO:	33
APPROVED BY: <i>DT</i>	5/2/55	SHEET NO:	1 OF 1

NOTE 1: DETAIL SHOWN IS FOR TOP OF COLUMN, BOTTOM CONSTRUCTION HAS ONLY CENTER TAPPED HOLE



DETAIL OF END CONSTRUCTION
(SEE NOTE 1)

DEPARTMENT OF CHEMICAL ENGINEERING
VIRGINIA POLYTECHNIC INSTITUTE
BLACKSBURG, VIRGINIA

SETTLING COLUMN

ULTRASONIC EXTRACTION

SCALE: 1" = 1"	DATE	CASE NO: 5 5
DRAWN BY: <i>Haw.</i>	4/30/55	FILE NO: 5 9 9
CHECKED BY: <i>D.T.</i>	4/30/55	FIGURE NO: 3 4
APPROVED BY: <i>D.T.</i>	5/2/55	SHEET NO: 1 OF 1

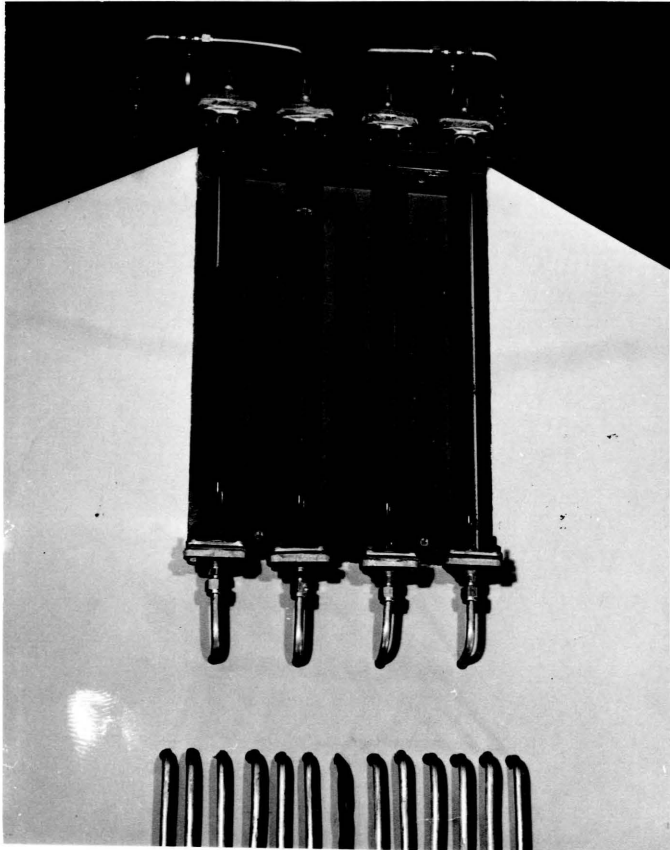


Figure 35. Mounting of Sight Gages for Feed and
Solvent Tanks

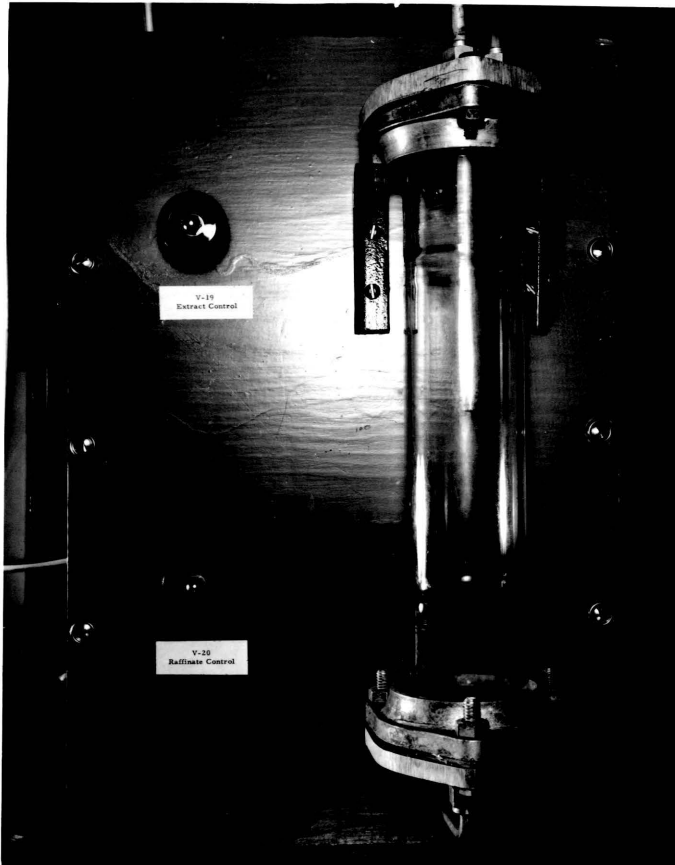
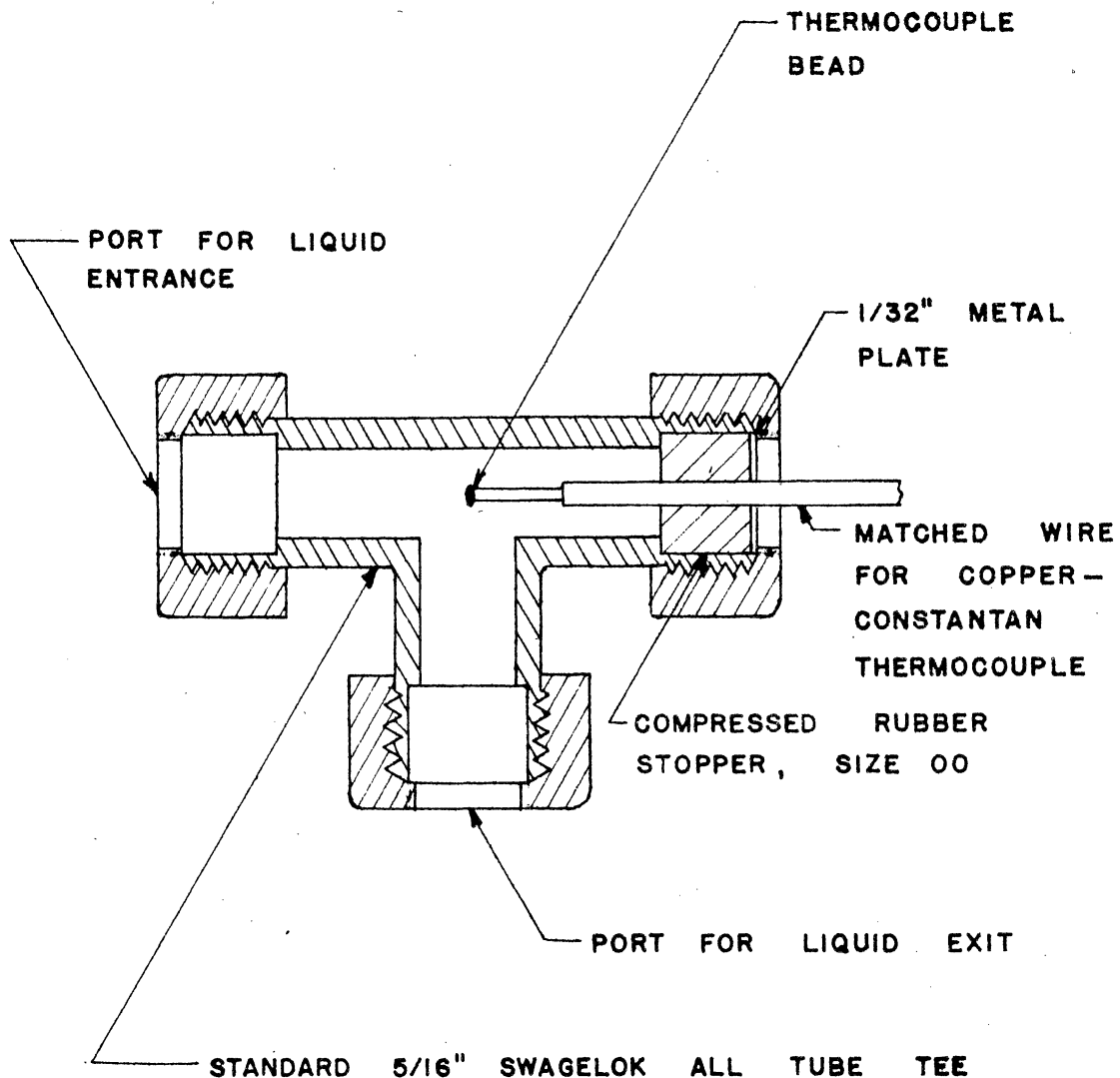


Figure 36. Installed Settling Column



DEPARTMENT OF CHEMICAL ENGINEERING
VIRGINIA POLYTECHNIC INSTITUTE
BLACKSBURG, VIRGINIA

SKETCH OF THERMOCOUPLE WELL
CONSTRUCTION

ULTRASONIC EXTRACTION

SCALE: NONE	DATE	CASE NO: 55
DRAWN BY: <i>New.</i>	4/30/55	FILE NO: 599
CHECKED BY: <i>D.T.</i>	4/30/55	FIGURE NO: 37
APPROVED BY: <i>D.T.</i>	5/2/55	SHEET NO: 1 OF 1

in Figure 38. The wiring diagram for the entire extractor is given in Figure 39.

Constant Temperature Bath. Arrangement of constant temperature bath, cooling coils, and circulating pumps is shown in Figure 40, specified in Table V, page 63.

Air Pressure Control Panel. Instrumentation and arrangement of small control panel for air pressure is shown in Figure 41.

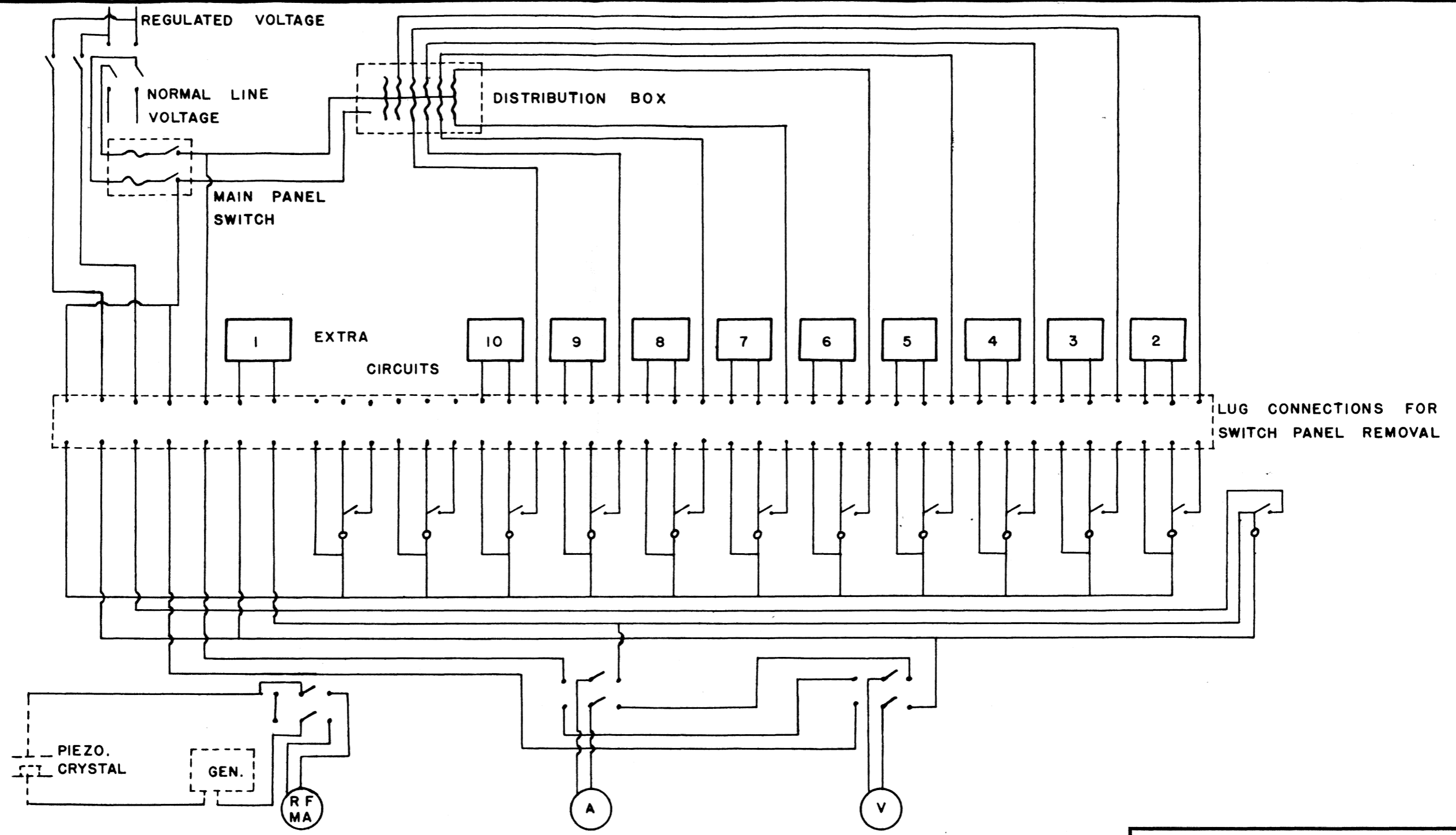
Design and Construction Procedure. Figures 23, 24, 25, 26, 27, 29, 30, 31, 32, 33, 34, and 37 show construction details for equipment sub-assemblies constructed using standard engineering techniques.

Reactor Design. The unique design and construction problem of this work was that of a suitable reactor, or contactor, for mixing feed and solvent in an ultrasonic field. To the knowledge of the author, no reactor of the type evolved for this problem has been employed in ultrasonic studies before.

Factors considered important in design and construction of the reactor were: (1) location of inlet and outlet tubes for liquids, (2) position inside the reactor of ultrasonic field of high intensity or focal point of transducer, (3) arrangements for transformer oil circulation for cooling transducer, (4) ease of making photographic studies of agitation and flow



**Figure 38. Arrangement of Hubicon Potentiometer in
Relation to Control Panel**



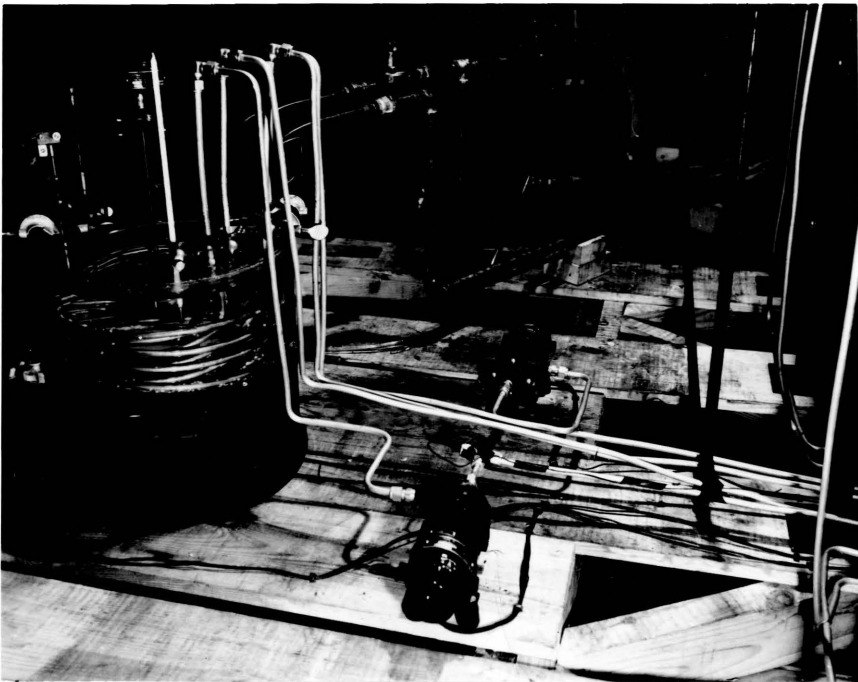
LEGEND

- | | | |
|-----------------------------|--------------------|-------------------------|
| 1 GENERATOR | 5 COOLING OIL PUMP | 9 ROTAMETER LIGHT |
| 2 CONSTANT TEMPERATURE BATH | 6 WASTE PUMP | 10 SIX VOLT TRANSFORMER |
| 3 FEED CIRCULATING PUMP | 7 FILLING PUMP | |
| 4 SOLVENT CIRCULATING PUMP | 8 FAN | |

DEPARTMENT OF CHEMICAL ENGINEERING
 VIRGINIA POLYTECHNIC INSTITUTE
 BLACKSBURG, VIRGINIA

PANEL WIRING DIAGRAM FOR
ULTRASONIC EXTRACTION

SCALE: NONE	DATE	CASE NO: 55
DRAWN BY: <i>How.</i>	4/12/55	FILE NO: 599
CHECKED BY: <i>DJ.</i>	4/30/55	FIGURE NO: 39
APPROVED BY: <i>DJ.</i>	5/2/55	SHEET NO: 1 OF 1



**Figure 40. Arrangement of Constant Temperature Bath,
Cooling Coils, and Circulating Pumps**

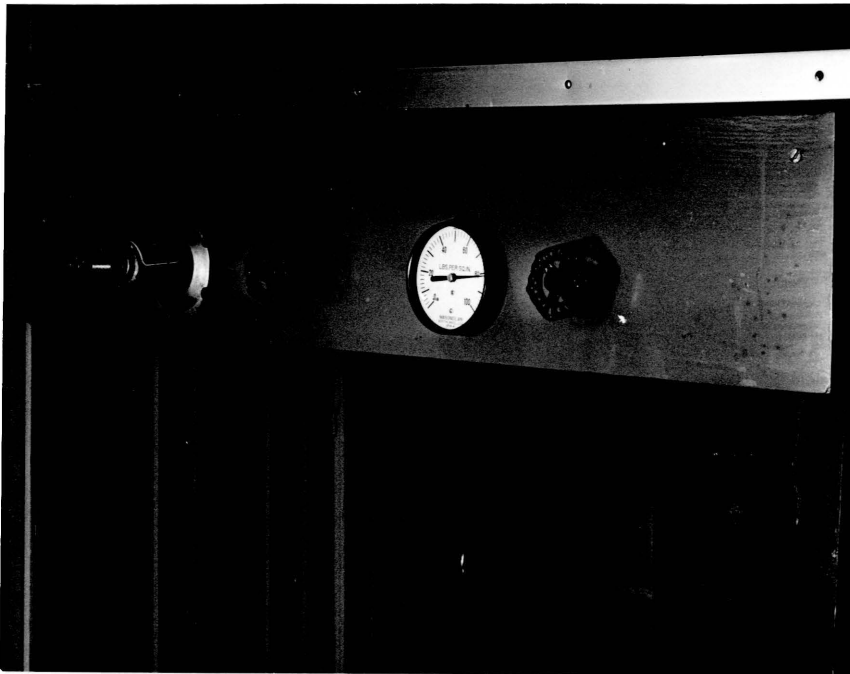


Figure 41. Air Pressure Control Panel

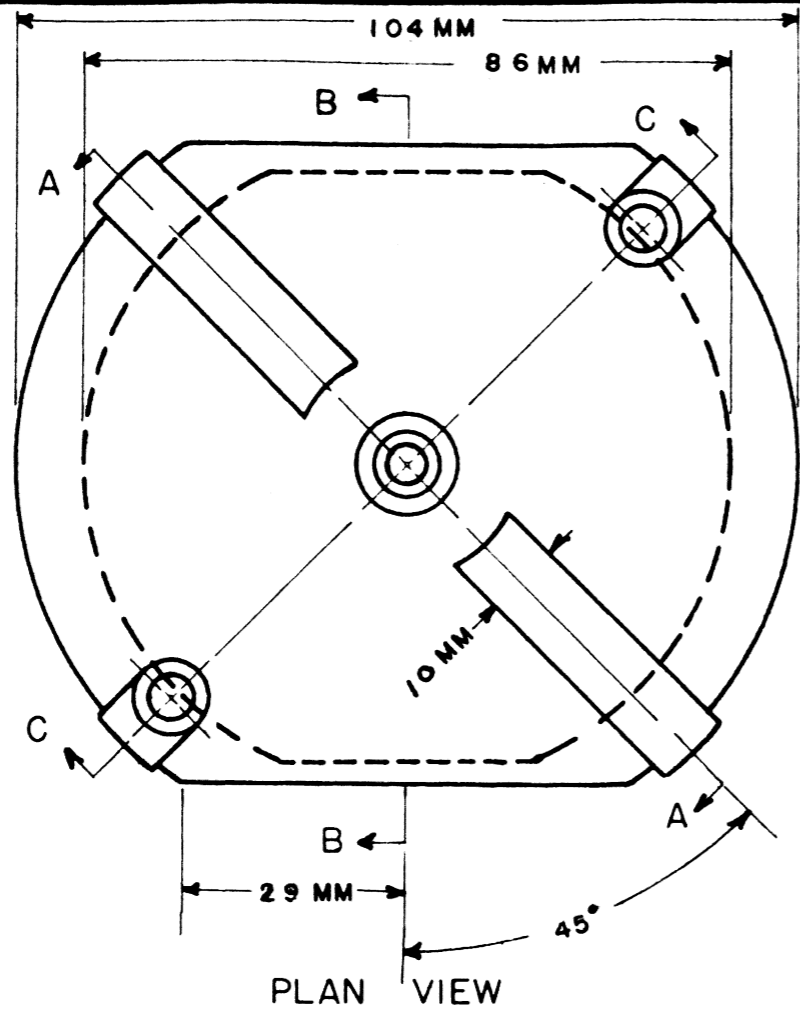
- patterns in reactor, (5) resistance to corrosion, and
(6) structural strength of finished reactor.

Reactor Shape. The reactor was designed in the shape of a cone to utilize the effect of focused waves from the transducer more efficiently than could be done with a vertical cylindrical type reactor. The cone shape of the reactor forces all liquid flowing through the reactor toward the field of high intensity, thereby giving greater liquid agitation by the ultrasonic waves for a given reactor volume.

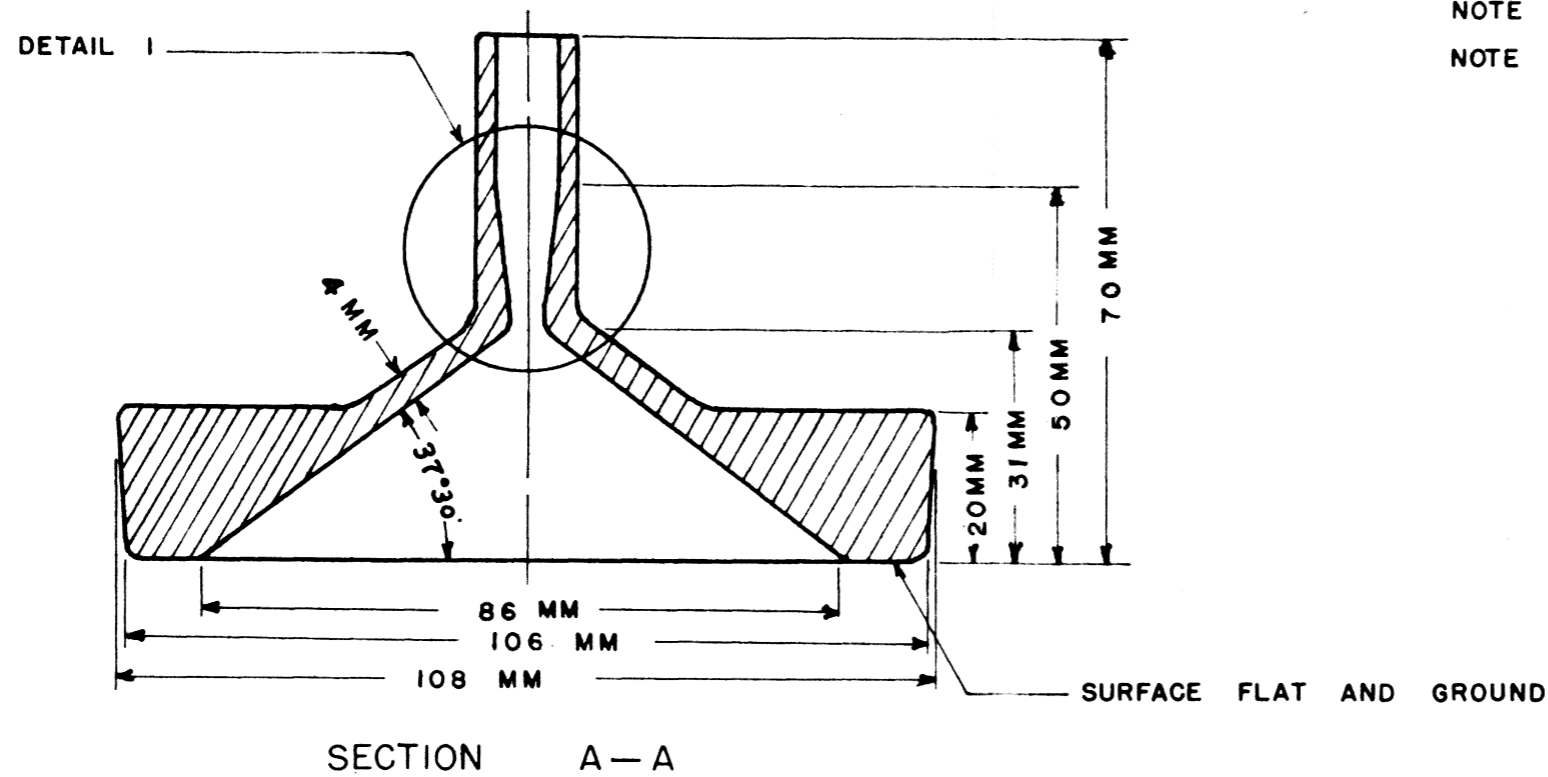
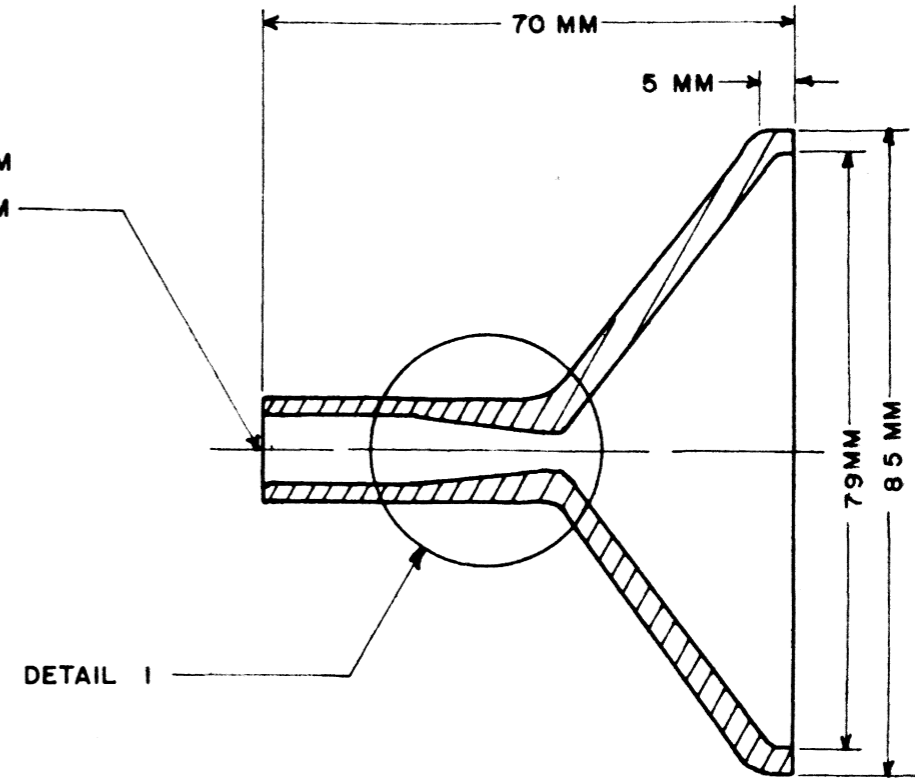
Material of Construction. Pyrex glass was selected for the original reactor as the material of construction. The glass, highly resistant to corrosion, allows photographs to be made of mixing and flow patterns inside the reactor.

Original Design. The original design for the reactor is shown in Figure 42. This design was considered to be impracticable, because of the close tolerances, for the manufacture of a glass reactor by a major glass company, and was never realized.

Plastic Reactor. A full-size reactor of original design, Figure 43, was carved from "Lucite" to furnish a model for reactor studies. This reactor was employed in



TUBING, O.D. 13 MM
I.D. 9 MM



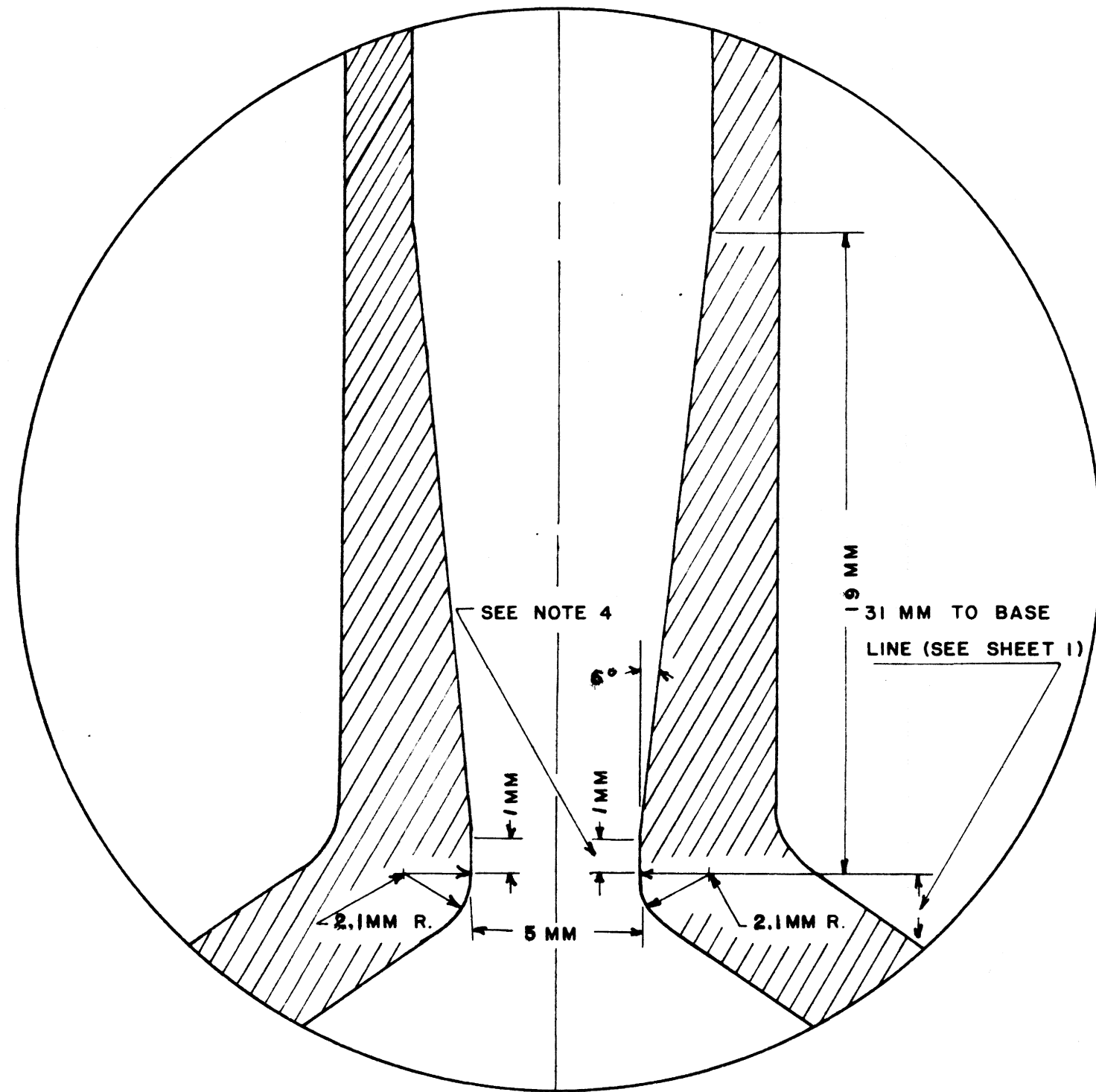
- NOTE 1: CONSTRUCTION OF PYREX GLASS
NOTE 2: DIMENSION TOLERANCES ± 0.10 MM
NOTE 3: UNMARKED FILLETS AND ROUNDS MIN. SIZE FOR EASE OF CONSTRUCTION

DEPARTMENT OF CHEMICAL ENGINEERING
VIRGINIA POLYTECHNIC INSTITUTE
BLACKSBURG, VIRGINIA

REACTOR DESIGN FOR
ULTRASONIC EXTRACTION

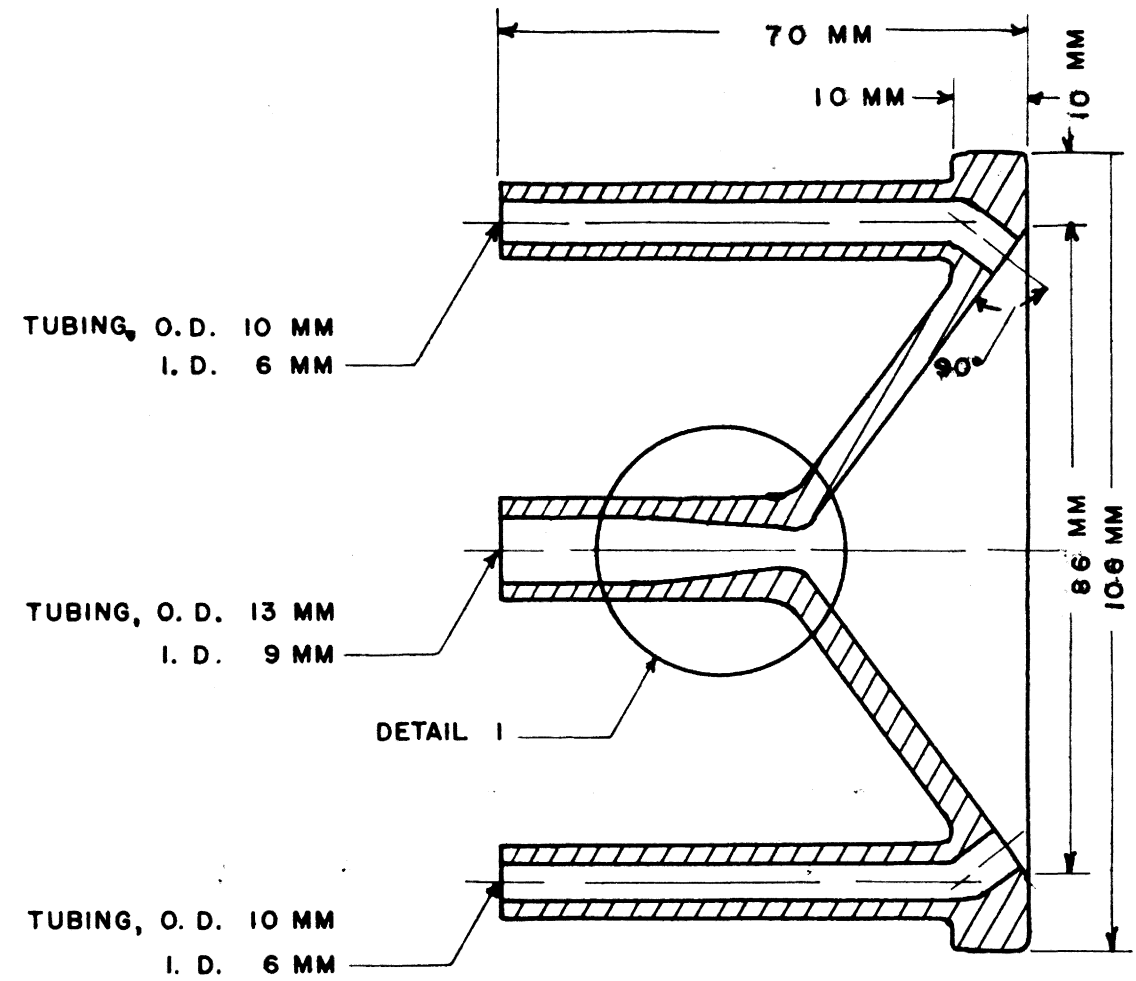
SCALE: FULL	DATE	CASE NO: 55
DRAWN BY: <i>Haw.</i>	4/15/55	FILE NO: 599
CHECKED BY: <i>D.T.</i>	4/30/55	FIGURE NO: 42
APPROVED BY: <i>D.T.</i>	5/2/55	SHEET NO: 1 OF 2

NOTE 4: 5 MM THROAT IS CYLINDRICAL AND VERTICAL



DETAIL I

(SCALE: 6 MM = 1 MM)



SECTION C-C

DEPARTMENT OF CHEMICAL ENGINEERING VIRGINIA POLYTECHNIC INSTITUTE BLACKSBURG, VIRGINIA			
REACTOR DESIGN FOR ULTRASONIC EXTRACTION			
SCALE: FULL	DATE	CASE NO:	5 5
DRAWN BY: <i>How.</i>	4/15/55	FILE NO:	5 9 9
CHECKED BY: <i>P.J.</i>	4/30/55	FIGURE NO:	4 2
APPROVED BY: <i>P.J.</i>	6/2/55	SHEET NO:	2 OF 2

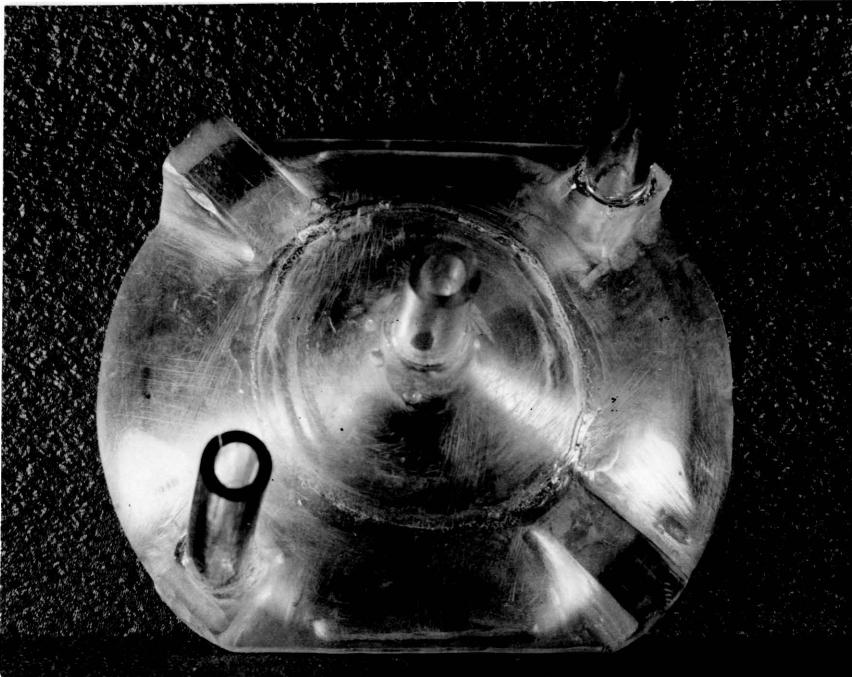


Figure 43. Plastic Reactor of Original Design

preliminary studies to determine flow patterns inside the reactor. When placed in operation, this plastic reactor was subjected to an insonation of intensity level approximately one-half that which was desired in future extraction studies. This original design was discarded when it was learned that placement of the reactor over the transducer was very critical, and that the small area of the exit tube, with the resulting high velocity of liquid, tended to favor the mechanical formation of an emulsion at this point.

Modified Design. Taking advantage of knowledge gained from preliminary reactor studies conducted with the plastic reactor, a modified reactor design was made based on a standard 60-degree pyrex glass funnel. Details of this modified design are given in Figure 21, page 72. A glass reactor of this design, with further modified cooling oil indentations, is shown in Figure 22, page 73.

Acoustical Window. Sheet nickel of 0.001-inch thickness was utilized as an acoustical window for the bottom of the reactor. Detail of the bond formed between the nickel sheet and glass reactor is illustrated in Figure 21. The bond between the nickel sheet and the glass of the reactor,

after being used in equipment evaluation test, is shown in Figure 44.

First attempted method of attaching the nickel sheet to glass was that of plating platinum on the glass edge and employing wire solder to bond the nickel sheet to the platinum. (The glass edge was painted with a saturated solution of chloroplatinic acid and fired to 1100 °F, at which temperature the platinum plated out.) This procedure worked very well for glass tubing of 32 millimeters outside diameter, but would not produce a bond strong enough to support the weight of liquid in the full size reactor.

A study of the conditions under which good bonding could be obtained with respect to thickness of paste and firing was conducted employing glass tubing sections and a DuPont silver paste number 6216. For application to the reactor, the paste was applied to the glass edge with a small brush, and the reactor fired to 1100 °F to plate out the silver. A layer of copper was then fixed on the silver by flashing from a cyanide copper bath. The nickel sheet was attached to the copper plate with wire solder. This procedure gave a bond between the nickel and glass that



**Figure 44. Example of Bond Between Nickel Sheet
and Glass Edge of Reactor**

was strong enough to resist pressures and liquid surges encountered in the equipment evaluation test.

Metal Reactor. A reactor of all metal construction was fabricated in an effort to obtain one that would stand possible rough treatment in first "shake-down" tests with the extractor. This reactor was constructed of tin and was conically-shaped in the dimensions of the glass reactor. The tin was copper plated, and the acoustical window of 0.001-inch nickel sheet attached to the bottom with wire solder. Entrance and exit area for circulation of cooling oil between the metal reactor and transducer was obtained by means of serrated edges at the lower edge of the metal reactor.

This metal reactor, Figure 45, was used in performing tests in evaluation of the extractor. The glass reactor previously described and shown in Figure 22, page 73, was also used in the evaluation test for making photographic studies of flow patterns.

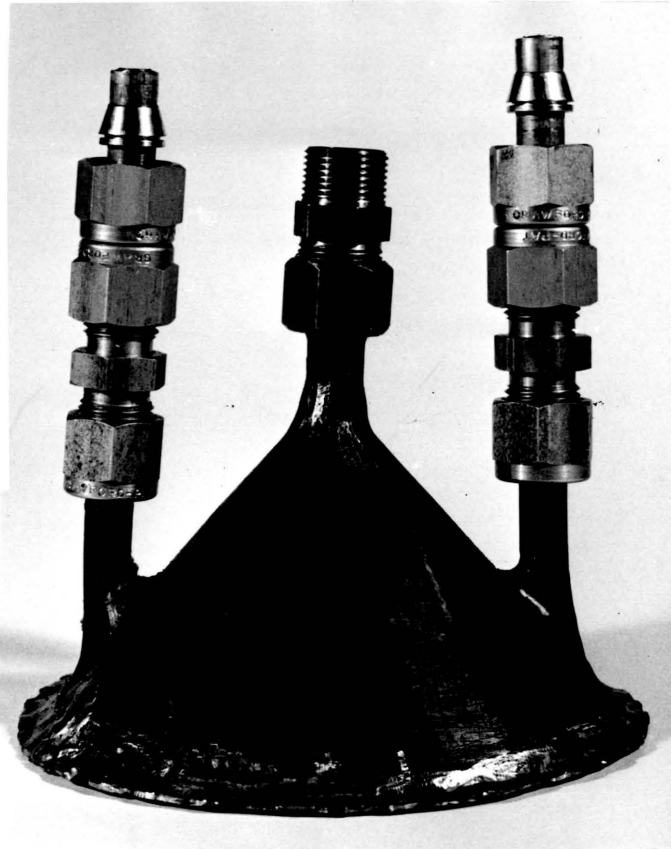


Figure 45. Metal Reactor Employed in Evaluation Test

Operational Procedure

It is recommended that any person intending to operate this extractor first become thoroughly familiar with the flow plan, wiring, and general characteristics. This may be done by physically checking the equipment and by studying the flow diagram and the wiring diagram. The flow diagram, Figure 33, page 86, wiring diagram, Figure 43, page 99, and general construction of the extractor, Figures 27 to 32, pages 79 to 85, and Figures 37 and 38, pages 90 and 92, will be found in an information folder that will be with the extractor at all times.

The extractor was designed and constructed for ease of operation and with the idea that it would be employed under a variety of experimental conditions. For this reason, all controls and instrumentation are located in such a manner that one person can operate the equipment.

Safety Measures. Safety precautions have been incorporated into the unit and include switches, fuses, indicating lights, tank vents, and cut off valves. A thorough check of the condition of general wiring, ultrasonic generator, and electrical instruments should be carried out before operation is started. Special attention should be given to the condition and amount of cooling oil in the transducer and the oil system. The extractor should never be operated until the oil circulating

system is operating and there is a constant level of oil at least one inch above the top of the barium titanate transducer. A forced air ventilation hood is in the area immediately adjacent to the extractor and should be employed whenever liquids are being handled that can produce obnoxious fumes. A CO₂ fire extinguisher is located close to the left side of the extractor and should be checked before any test is started.

Preparation for Start-up. In preparation for a start-up of the extractor, all hydraulic and air valves should be closed, and all electrical switches placed in "off" position. A written plan for a particular test, prepared notebook for recording data, sample bottles, and auxiliary equipment, the use of which is anticipated, should be on hand before operation begins. The following check list is given for initially operating the extractor:

1. Voltage regulator turned on
2. Main panel switch turned on
3. Feed and solvent tanks filled
4. Adjustment and operation of constant temperature bath and circulating feed and solvent pumps
5. Proper operation of cooling water system
6. Proper operation of cooling oil system
7. Adjustment of air pressure on feed and solvent tanks

8. Proper positioning of valves controlling flow of feed and solvent into reactor

9. Warm-up of generator.

Electrical Power Switches. Switch for voltage regulator is located on the first floor of the Ultrasonic Laboratory, in the Constant Temperature room. Main panel switch should not be turned on until all individual circuit switches are in "off" position.

Filling Feed and Solvent Tanks. Feed and solvent tanks are filled by use of pump controlled by panel switch 7. To fill the feed tanks, the discharge of the filling pump is connected to the end of "feed" tubing located on right side of panel frame and immediately below settling column tray. Valve V-1 is opened to fill tank 1, and valve V-4 is opened to fill tank 2. Tanks may be filled simultaneously. Valves V-2 and V-3 are left closed during filling cycle. Air line vent, located on air line to gage on panel board, is opened for venting when filling feed or solvent tanks. Solvent tanks are filled by connecting pump discharge to end of "solvent" tubing, located close to "feed" tubing beneath settling column tray, and valve V-8 or V-9 opened, depending on which tank is to be filled. Valves V-5 and V-6 remain closed during filling cycle.

Operation of Constant Temperature Bath. Stirrer and heater for constant temperature bath are controlled by switch 2, on the panel board. Adjustment to desired temperature is made by use of mercury contact and relay located on frame of the bath. Cooling water for the bath is controlled by valve V-32, located under sink, and valve V-34, located in right rear corner of shop. Water for filling bath is obtained from line controlled by valve V-35, located adjacent to the constant temperature bath. Three sets of tubing coils are immersed in the constant temperature bath. One coil is for cooling water, one is for solvent, and one is for feed.

Operation of Cooling Oil System. Cooling oil flow to transducer is regulated by valve V-18. The rate of flow is indicated on flow meter FM 5. Pump for cooling oil is controlled by switch 5 on the panel board. Flow of water for oil heat exchanger is controlled by valve V-33, located on shop wall behind the heat exchanger.

Feed and Solvent Circulation. Feed and solvent are circulated through respective tubing coils in the constant temperature bath by pumps controlled by panel switches 3 and 4, respectively. To circulate contents of tank 1, valve V-1 is opened entirely and valve V-2 is opened slightly. The circulating pump takes liquid from the tank, forces it

through the coil in the constant temperature bath, and back to the tank. A low flow rate through V-2 is required to keep liquid from backing up in air line, as the circulating pump is capable of pressures higher than the air pressure on the feed and solvent tanks, normally in the range of ten pounds per square inch, gage. Other feed and solvent tanks are circulated in the same manner, using appropriate valves as shown on flow diagram for each tank.

Regulation of Air Pressure. Air pressure is placed on the feed and solvent tanks by operation of valve V-30, which is located on the small air control panel. The latter is approximately two feet from the right side of the major control panel. Valve V-30 should not be turned on until the pressure regulator is closed. Full line pressure for air, 70 to 90 pounds per square inch, gage, may be read on the pressure gage on air control panel. Valve V-31 is then opened and the pressure regulator adjusted to give ten pounds per square inch on the gage located on the main panel board. The air vent on the line to the operating pressure gage should be closed before air pressure is placed on the feed and solvent tanks.

Extract and Raffinate Tanks. Extract from settling column is directed to tank 5 by opening valve V-21, and to tank 6 by opening valve V-22. Raffinate is directed to tank 7 by opening

valve V-23, and to tank 8 by opening valve V-24. Valves V-25, V-26, V-27, and V-28, should remain closed during test. Valves V-19 and V-20 should be open when liquid starts through the settling column, and then adjusted for desired interfacial level after liquid flow has commenced.

Generator Warm-up. Power to generator is controlled by switch 1 on the panel board. The filament switch only, located on the face of the generator, should be turned on initially. The fan, controlled by panel switch 8, should be in operation at any time the generator is used. Powerstat should be set at "0" before power switch is operated at a later step in the procedure.

Feed and Solvent Flow Rate Control. For normal operation, air pressure on the feed and solvent tanks should be adjusted to ten pounds per square inch, gage. As this pressure will give constant flow for all but the very highest rates, it should not be exceeded except for these unusual cases. Control of feed rate to reactor is accomplished by means of valves V-10 and V-11. Feed flow rate is indicated by flow meter FM 1. Solvent flow rate is controlled by valves V-12 and V-13, and the flow rate is indicated on flow meter FM 2. When opening valves for flow of feed and solvent into reactor, care should be taken to open them slowly, so as not to give a sudden surge of liquid through the

reactor. High pressures developed by sudden surges will endanger the acoustical window of the reactor.

Interfacial Control. Adjustment of interfacial level in the settling column is accomplished by valves V-19 and V-20. Valve V-20, regulating flow of the heavy phase, is closed to force a layer of the heavy phase liquid up to end of entrance tube in the settling column. The valve is then adjusted to allow a constant flow rate of the heavy phase out of the settling column. This adjustment keeps the interfacial level constant.

Sample Collection. Samples of feed and solvent are taken through valves V-14 and V-15, respectively. Extract and raffinate samples are taken through valves V-16 and V-17, respectively. Samples should be taken slowly over a period of time, instead of running full flow into sample bottle. This procedure minimizes disturbance of flow rates and equilibrium.

Power to Transducer. After the two liquid phases are flowing through the reactor and settling column, power to the barium titanate ceramic transducer from the ultrasonic generator may be turned on. This is done by the power switch and powerstat on the face of the HYPERSONIC generator. High power, 70 to 80 on the powerstat scale, should not be employed when first operating the generator. Power tubes should be allowed to warm up for four or five minutes after the power switch is thrown. Adjustment in

power level is then accomplished with the powerstat. Critical frequency tuning after the generator and transducer have warmed up is done by utilization of the large tuning knob on the generator face. Current input to the transducer is measured by a radio frequency milliammeter mounted on the panel just above the generator.

Temperature Measurements. Temperature measurements are made with seven copper-constantan thermocouples wired to the thermocouple selector switch and the Rubicon potentiometer. Conversion of potential to temperature is accomplished through thermocouple calibration charts.

Electrical Power Measurements. Electrical power measurements are made with a voltmeter and an ammeter mounted on the panel just above the generator. Power input to either generator or load, consisting of pumps, lights, and other electrical equipment, can be ascertained by proper manipulation of the double-pole, double-throw switch for each meter.

General Operation. After the extractor is placed in operation, flow rates, feed to solvent ratios, and insonation intensity can be adjusted for a particular test. Samples of liquid streams may be obtained and thermal conditions ascertained.

Level of Feed and Solvent Tanks. As a note of caution, the operator should pay strict attention to the levels in the

feed and solvent tanks. No tank should be emptied completely. The reserve tank of feed or solvent should be utilized before the first tank is emptied. Employment of tanks in this manner will eliminate air getting in flow lines during a test and disturbing the equilibrium conditions that have been established.

Shut-down Procedure. When a test is completed, the following procedure should be employed for shut-down. The ultrasonic generator should be turned off as soon as the test is complete. After the generator is turned off, the cooling oil system and the cooling water system both may be turned off. The feed and solvent tanks should all be put on stream gradually and emptied. If large amounts of feed and solvent remain in the tanks at the completion of a test and continued mixing of the two is not desired, the tanks should be emptied singularly. This is accomplished by disconnecting the feed and solvent lines going into the reactor and attaching in place of the reactor lines that lead to a storage carboy or container. If corrosive feed or solvent was used in a test, the entire system should be flushed with water or other washing liquid.

Emptying Extract and Raffinate Tanks. Extract and raffinate tanks are emptied by use of waste pump, controlled by panel switch 6. Valves V-25, V-26, V-27, and V-28 control these tanks. Only one tank should be emptied at a time.

Shut-down and Checking. After all required pumping is completed, main panel electrical switch and air control valve V-30 should be turned off. A thorough check for any leaks or damage to extractor should be conducted before it is left idle. A periodic check for leaks should also be conducted during test.

Extractor Evaluation

An evaluation test with this extractor was carried out to ascertain its operating characteristics. The rate of mass transfer occurring in the test system was not of major concern in this test. Detailed studies of mass transfer in several systems will be undertaken at a later date.

System Employed for Evaluation. The system acetone-water-1,1,2-trichloroethane was chosen for making the original test with the extractor. Availability of system components, ease of analysis, relatively low corrosion, and available data on previous extraction work employing this system are factors that led to this choice. Liquid equilibria data for this system at 25 °C are given in Figure 46.

Make-up and Handling of Feed and Solvent. A feed mixture of water and acetone, approximately 20 per cent acetone by weight, was made up for the evaluation test. Regular tap

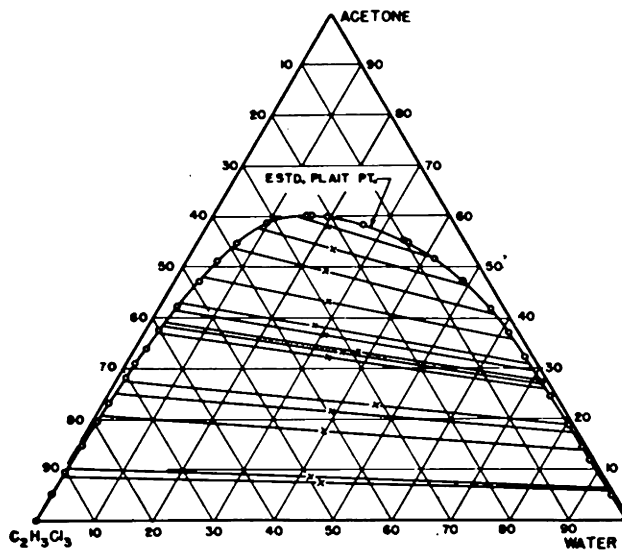


Figure 46. Liquid Equilibria of System Acetone-Water-
1,1,2-Trichloroethane at 25 °C

Treybal, R. E., L. D. Weber, and J. F. Daley: The System
Acetone-Water-1,1,2-Trichloroethane, *Ind. Eng. Chem.*,
38, 817 (1946).

water from Virginia Polytechnic Institute water supply was employed as the carrier solvent. A 15-gallon ceramic crock held the feed solution while mixing was accomplished with a Lightnin mixer. During addition of feed components to the vessel, mixing, and pumping it into tanks 1 and 2, the top of the crock was covered with heavy towels saturated with water to reduce evaporation.

The solvent for this test was 1,1,2-trichloroethane saturated with water. A ceramic crock similar to the one utilized for the feed was employed for the preparation of the solvent. A known excess of water was placed in the crock with the 1,1,2-trichloroethane, and the two were mixed with the Lightnin mixer. After separation of the two phases in the vessel, the solvent was pumped to tanks 3 and 4. Wet towels were also used in this instance to lessen evaporation of solvent.

After the feed and solvent were pumped into tanks 1 and 2, and 3 and 4, respectively, they were circulated in closed systems through the constant temperature bath until a temperature of 25 °C was reached. Intermittent circulation was carried on during actual test to maintain this temperature.

Solvent-to-Feed Ratio. A solvent-to-feed ratio of approximately 1:1 was chosen because it is practical and known to be in the order of magnitude that will be considered in future studies.

Flow Rates. Minimum flow rate of each phase was set at approximately 100 grams per minute, with maximum flow rate approximately 300 grams per minute. Five different rates within this range were investigated. Exact rates employed for each test conducted will be found in Table VI.

Number of Tests Made. Five test runs of different flow rates were conducted without insonation, and five were made with insonation. The second set of tests had flow rates that were approximately the same as the corresponding test in those without insonation.

Insonation Intensity. Current input to the transducer was measured as a function of generator plate current for this evaluation test. A constant input voltage of 120 volts and a plate current of 160 milliamperes were used as the measure of intensity of insonation. This level of insonation, 120 volts and 160 milliamperes, was employed in all tests conducted with insonation.

Sample Collection. Operating samples of feed, solvent, extract, and raffinate were taken ten minutes after starting a test of a particular flow rate. These operating samples were checked with an Abbe refractometer, and samples to represent the test conditions were taken after two operating samples taken ten minutes apart had a constant analysis. Samples of

TABLE VI

Operational Data and Results for Extractor Evaluation Test
(Extraction of Acetone from Water with 1,1,2-Trichloroethane^a)

Test	Plate Current, ^b ma	Phase	Flow Rate, gm/min	S/F Ratio	Refractive Index, n_D^{25}	Acetone, wt %	Stage Efficiency, %
1	0	Feed, Solvent, Extract, Raffinate,	93 105 143 65	1.13:1	1.3467 1.4644 1.4406 1.3404	19.2 0.0 12.1 10.3	110.0
2	0	Feed, Solvent, Extract, Raffinate,	115 145 167 93	1.26:1	1.3467 1.4644 1.4454 1.3401	19.2 0.0 10.6 10.2	104.0
3	0	Feed, Solvent, Extract, Raffinate,	165 190 185 152	1.15:1	1.3467 1.4644 1.4442 1.3402	19.2 0.0 11.3 10.2	106.5
4-1	0	Feed, Solvent, Extract, Raffinate,	215 250 280 175	1.16:1	1.3467 1.4644 1.4461 1.3415	19.2 0.0 11.3 12.4	106.5
4-2	0	Feed, Solvent, Extract, Raffinate,	210 235 277 165	1.12:1	1.3467 1.4644 1.4454 1.3413	19.2 0.0 10.6 12.3	95.5
5	0	Feed, Solvent, Extract, Raffinate,	250 300 345 200	1.20:1	1.3467 1.4644 1.4452 1.3389	19.2 0.0 10.6 8.8	105.0
6	160	Feed, Solvent, Extract, Raffinate,	93 85 110 65	0.92:1	1.3450 1.4644 1.4413 1.3400	17.8 0.0 12.0 10.2	109.0
7	160	Feed, Solvent, Extract, Raffinate,	135 135 149 127	1.00:1	1.3450 1.4644 1.4452 1.3402	17.8 0.0 10.9 10.2	103.8
8	160	Feed, Solvent, Extract, Raffinate,	215 250 301 158	1.16:1	1.3450 1.4644 1.4472 1.3400	17.8 0.0 9.6 10.2	93.0
9	160	Feed, Solvent, Extract, Raffinate,	243 295 336 215	1.21:1	1.3450 1.4644 1.4476 1.3390	17.8 0.0 9.6 9.0	101.1
10	160	Feed, Solvent, Extract, Raffinate,	152 155 188 130	1.02:1	1.3450 1.4644 1.4671 1.3405	17.8 0.0 9.9 11.0	94.3

^a Temperature, 25 °C

^b Brush HYPERSONIC Generator. Model No BU-204. Serial No 52-B-071.
400 kilocycle insonation.

approximately 40 cubic centimeters were taken in large test tubes, the test tube fitted with a tight cork, and covered with aluminum foil.

Analysis of Samples. Refractive index was employed as method of analysis. Samples in corked test tubes were placed in a constant temperature bath at 25 ± 0.1 °C and after reaching constant temperature, were analyzed by means of a dipping refractometer.

Information⁽⁷⁹⁾ is available in the literature for conversion of refractive index to per cent acetone by weight for the system acetone-water-1,1,2-trichloroethane. 1,1,2-Trichloroethane employed as solvent in this test was checked and was found to have a refractive index corresponding to a two per cent acetone by weight mixture on the refractive index versus concentration curve for acetone-1,1,2-trichloroethane mixtures taken from the literature. A new concentration curve was constructed using the same slope as that of the published curve, but placing the point of the curve referring to zero per cent acetone by weight at the refractive index observed for the particular lot of 1,1,2-trichloroethane employed as solvent.

A check of tap water used as carrier solvent showed that the refractive index was the same as that published⁽⁷⁹⁾ for

water with no acetone present, so the refractive index versus concentration curve for acetone in the water-rich phase was utilized as published. Refractive index versus acetone concentration per cent by weight for 1,1,2-trichloroethane-rich phase is given in Figure 47, and for the water-rich phase in Figure 48.

Calibration of Flow Meters. Calibrations were made for different liquid phases, or streams, in order to measure flow rates more accurately than could be done with factory calibrations. Method employed in calibrating is that recommended by Fischer and Porter Company for the "TRI-FLAT" flow meter⁽⁹³⁾. Special calibrations were made for solvent and for the 1,1,2-trichloroethane-rich phase leaving the settling column. Factory calibrations were employed for the feed and water-rich phase leaving the settling column. The calibration curve for solvent is given in Figure 49, for the 1,1,2-trichloroethane-rich phase in Figure 50, and for the feed and water-rich phase in Figure 51.

Calibration of Thermocouples. Thermocouples were calibrated by standard method, measuring the potential generated by the thermocouple in freezing water and in water boiling at a known vapor pressure and temperature. Calibration curve for copper-constantan thermocouples is given in Figure 52.

TABLE VII

Refractive Index Data for Acetone-Water-1,1,2-Trichloroethane
Mixtures at 25 °C

1,1,2-Trichloroethane	Water	Acetone	Refractive Index 25 n _D
wt %	wt %	wt %	
99.89	0.11	0.00	1.4683
94.73	0.26	5.01	1.4588
90.11	0.36	9.53	1.4504
34.65	0.59	14.76	1.4416
79.58	0.76	19.68	1.4338
75.72	0.99	23.29	1.4285
70.36	1.43	28.31	1.4218
67.52	1.44	31.04	1.4178
64.17	1.87	33.96	1.4139
60.06	2.11	37.33	1.4088
54.88	2.93	42.14	1.4032
48.78	4.01	47.21	1.3972
43.88	5.00	51.12	1.3927
38.31	6.34	54.35	1.3888
31.67	9.73	58.35	1.3829
26.39	13.35	60.26	1.3792
24.04	15.37	60.59	
25.20	16.65	60.17	1.3792
20.71	19.31	59.98	1.3758
15.39	26.23	58.33	1.3695
10.00	34.96	55.04	1.3672
9.63	35.38	54.99	1.3672
6.77	41.35	51.88	1.3652
4.35	48.47	47.13	1.3626
2.18	55.97	41.65	1.3601
1.72	61.11	37.17	1.3578
1.17	66.58	32.25	1.3543
1.02	71.30	27.13	1.3518
0.92	74.54	24.54	1.3501
0.78	80.40	18.62	1.3460
0.70	84.94	14.36	1.3430
0.65	87.63	11.72	1.3412
0.52	94.66	4.32	1.3382
0.44	99.56	0.00	1.3328

Treybal, R. E., L. D. Weber, and J. F. Daley: The System
Acetone-Water-1,1,2-Trichloroethane, Ind. Eng. Chem.,
38, 817-821 (1946).

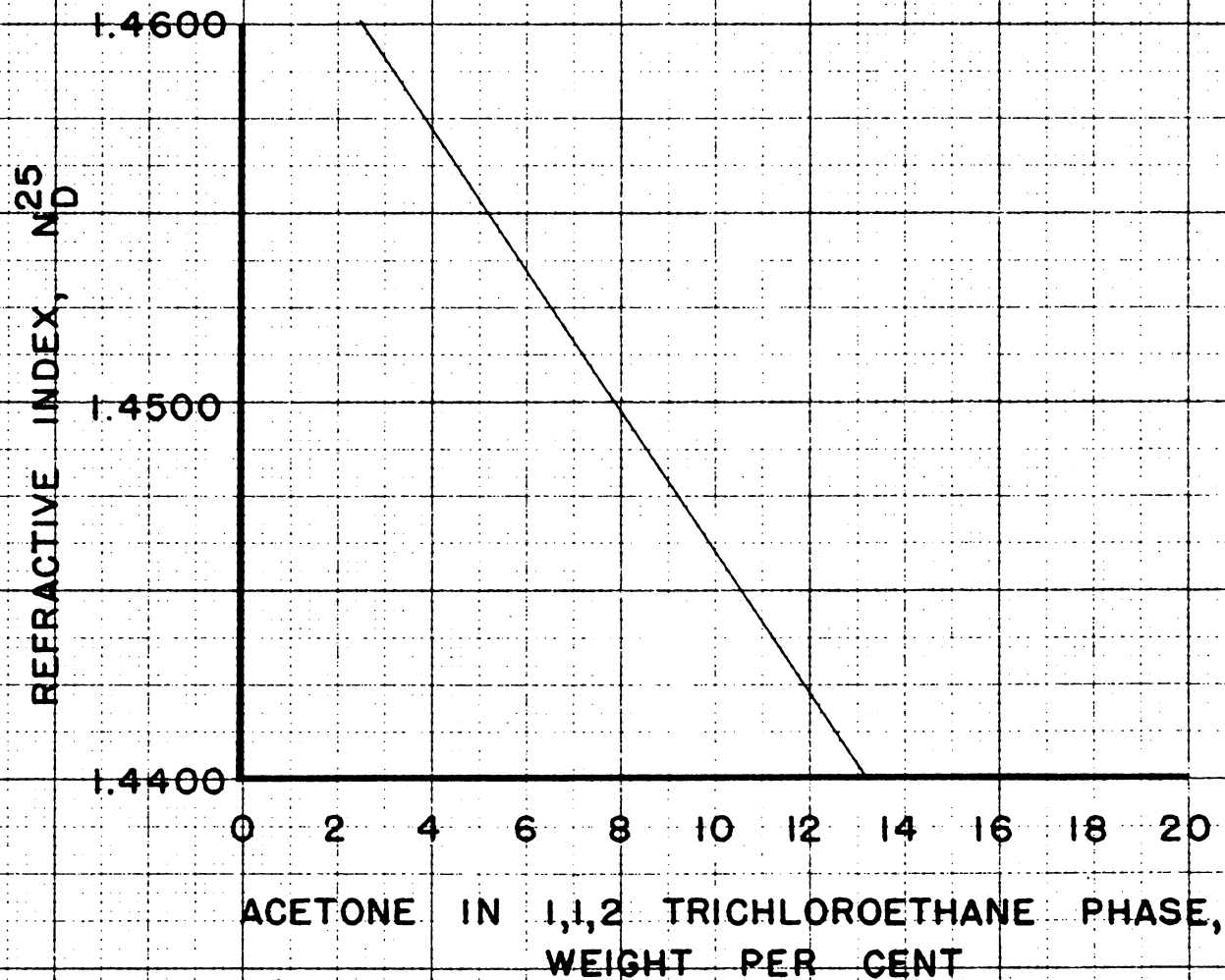


FIGURE 47. ACETONE CONCENTRATION OF 1,1,2-TRICHLOROETHANE PHASE FOR SYSTEM ACETONE-WATER-1,1,2-TRICHLOROETHANE

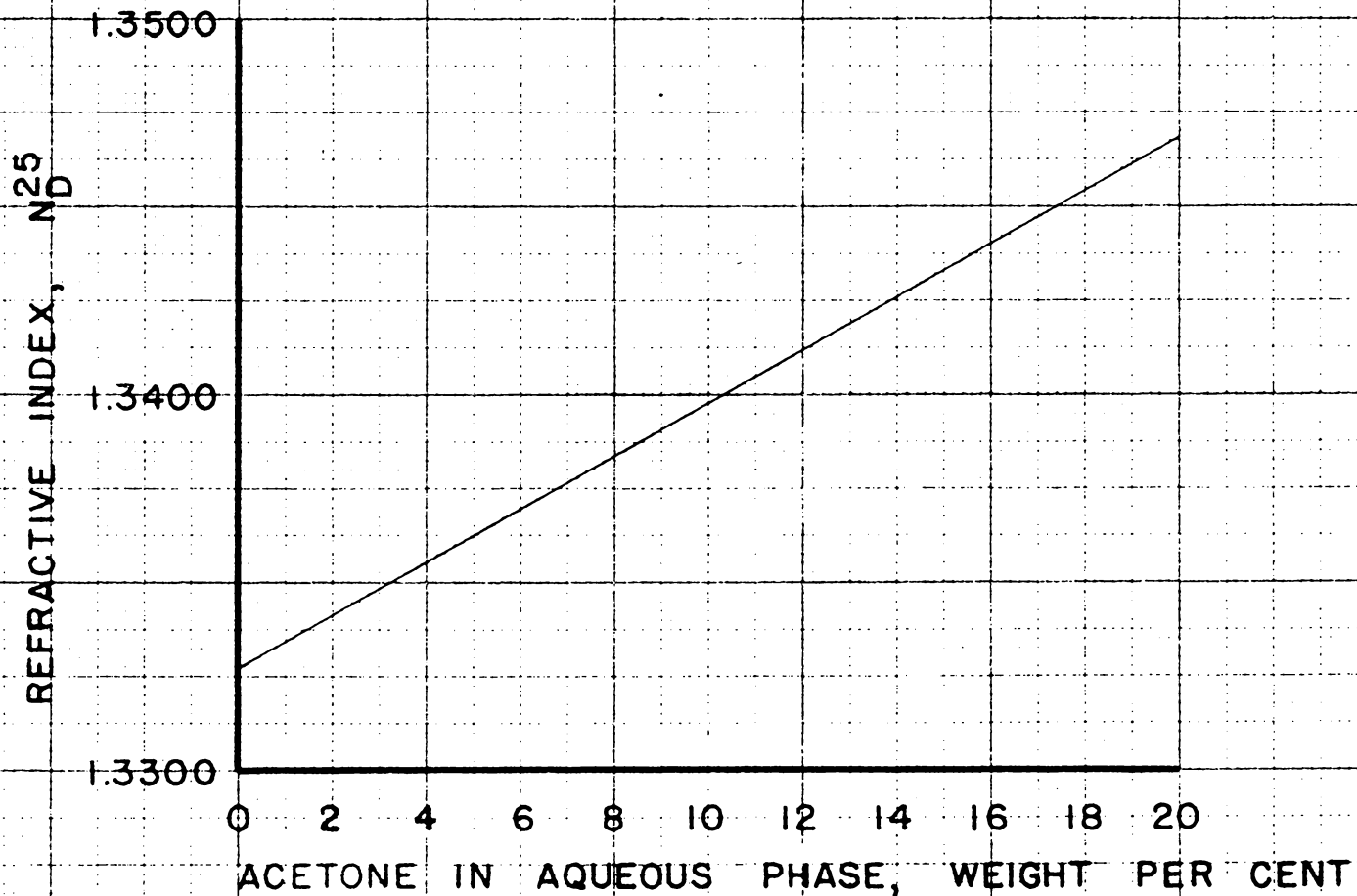
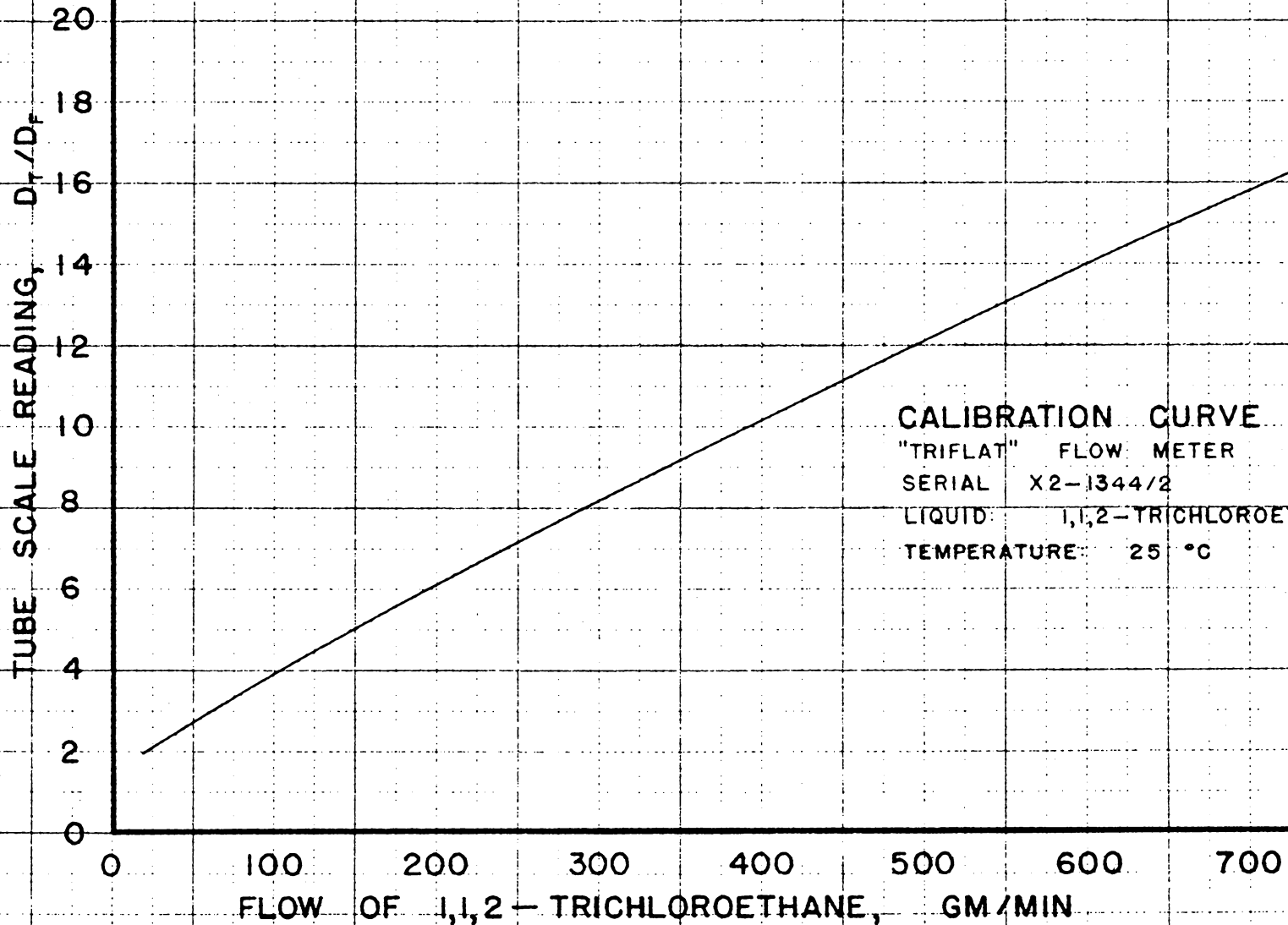


FIGURE 48. ACETONE CONCENTRATION OF AQUEOUS PHASE FOR SYSTEM ACETONE - WATER - 1,1,2-TRICHLOROETHANE

TABLE VIII

Calibration of "TRI-PLAT" Flow Meter, Serial X2-1344/2,
for 1,1,2-Trichloroethane at 25 °C

<u>Tube Scale Reading</u>	<u>Flow Rate,</u> <u>gm/min</u>
2	28.7
4	105.0
6	197.0
8	292.0
10	397.0
12	497.0
14	607.0
16	710.0
18	825.0
20	940.0



-125-

FIGURE 49. FLOW METER CALIBRATION FOR SOLVENT

TABLE IX

Calibration of "TRI-FLAT" Flow Meter, Serial X2-1344/4,
for 1,1,2-Trichloroethane Rich Extract Phase at 25 °C

<u>Tube Scale Reading</u>	<u>Flow Rate,</u> <u>gm/min</u>
2	14.3
4	60.9
6	120.0
8	181.0
10	252.0
12	318.0
14	389.0
16	460.0
18	537.0
20	609.0

TUBE SCALE READING, D_t/D_f

20
18
16
14
12
10
8
6
4
2
0

0 100 200 300 400 500 600 700
FLOW OF 1,1,2-TRICHLOROETHANE RICH PHASE FROM SETTLING
COLUMN, GM/MIN

CALIBRATION CURVE
"TRIFLAT" FLOW METER
SERIAL X2-1344/4
LIQUID: 1,1,2-TRICHLOROETHANE RICH
PHASE, 11% ACETONE
TEMPERATURE: 25 °C

-127-

FIGURE 50. FLOW METER CALIBRATION FOR EXTRACT

TABLE X

Calibration of "TRI-PLAT" Flow Meters, Serials X2-1344/1 & 3,
for Feed and Water Rich Raffinate Phase at 25 °C

<u>Tube Scale Reading</u>	<u>Flow Rate,</u> <u>ga/min</u>
2	16.0
4	67.0
6	125.0
8	186.0
10	258.0
12	325.0
14	397.0
16	471.0
18	546.0
20	625.0

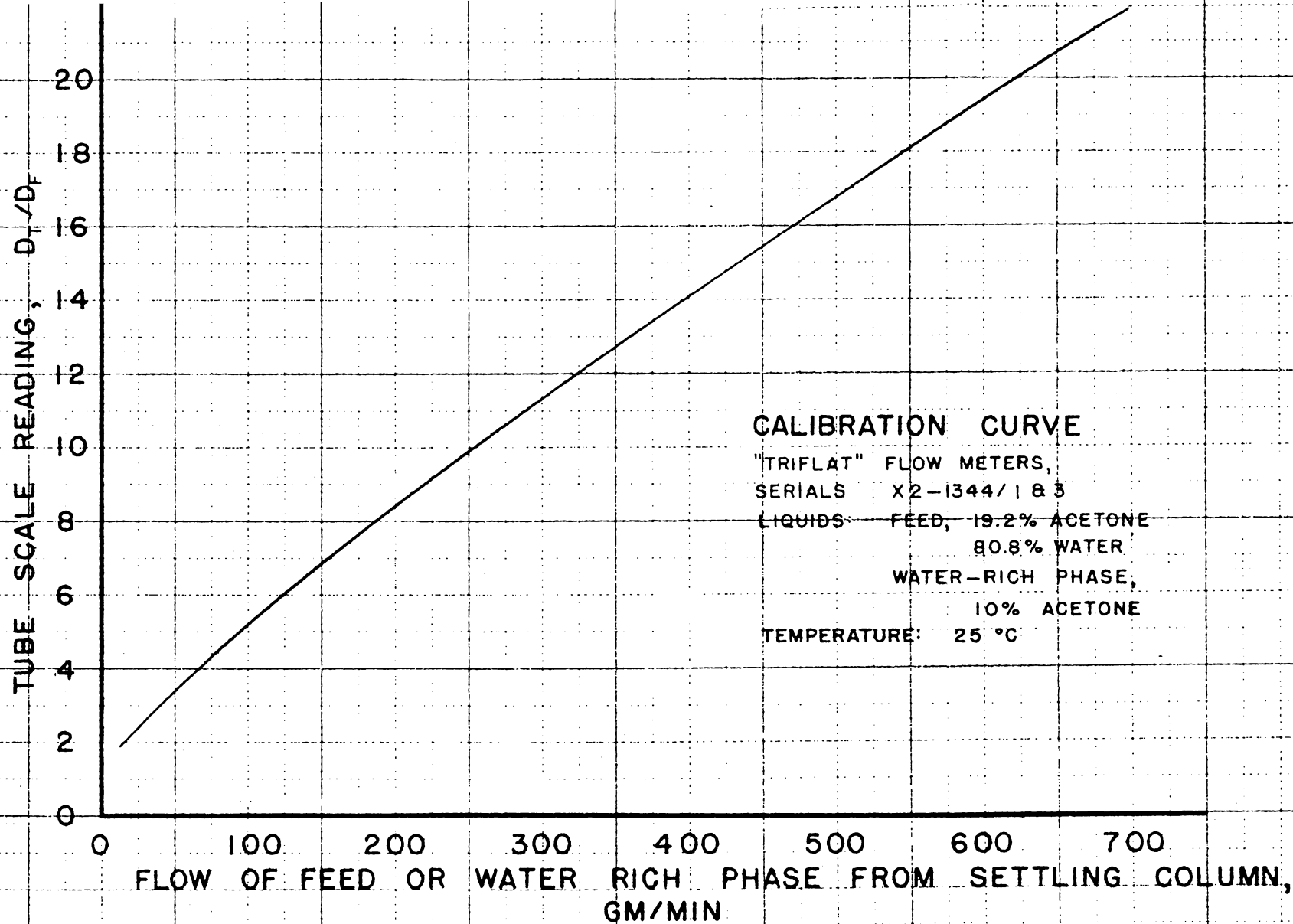


FIGURE 51. FLOW METER CALIBRATION FOR FEED AND RAFFINATE

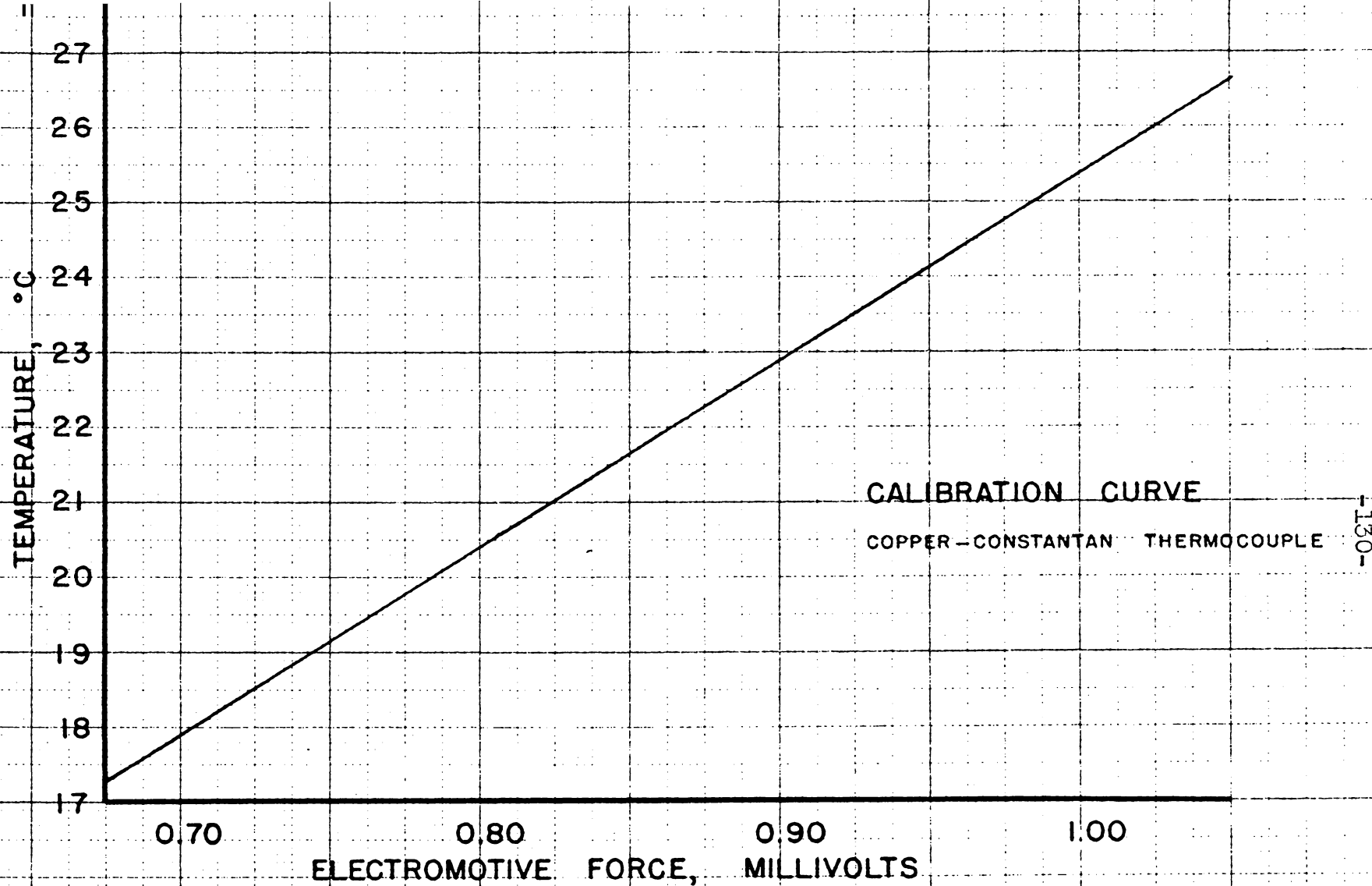


FIGURE 52. THERMOCOUPLE CALIBRATION

Sample Calculations

Examples of the different type calculations made in this investigation are presented in this section. Two sets of calculations will be discussed: (1) calculations for calibration of flow meters, and (2) calculation of stage efficiencies for the different tests made with the system acetone-water-1,1,2-trichloroethane.

Calibration of Flow Meters. Calculations for flow meter calibration were made as suggested by Fischer and Porter Company ⁽⁹³⁾. Calculations presented here are for calibration of the flow meter employed to measure the rate of flow of extract leaving the settling column. The calculations involve six major steps, as shown below:

Step 1. Statement of fluid properties and meter dimension values:

- | | |
|-------------------------|---------------------------------|
| 1. Float material: | glass |
| 2. Float diameter: | 0.375-inch |
| 3. Float density: | 2.28 grams per cubic centimeter |
| 4. Float weight in air: | 1.04 grams |
| 5. Fluid viscosity: | 0.95 centipoise |
| 6. Fluid density: | 1.28 grams per cubic centimeter |

Step 2. Calculation of viscous influence number:

$$N = \frac{\mu}{\sqrt{W_f \left(\frac{\rho_f - \rho}{\rho_f} \right) \rho}} \quad (6)$$

where:

N = viscous influence number

μ = fluid viscosity, centipoises

W_f = float weight in air, grams

ρ_f = float density, grams per cubic centimeter

ρ = fluid density, grams per cubic centimeter

$$N = \frac{(0.95)}{\sqrt{(1.04) \left(\frac{2.28 - 1.28}{2.28} \right) (1.28)}} \quad (7)$$

$$N = 1.24 \quad (8)$$

Step 3. Calculation of instrument constants:

$$I. C. = D_f \sqrt{W_f \left(\frac{\rho_f - \rho}{\rho_f} \right) \rho} \times (\text{flow unit conversion from pounds per minute to desired units}) \quad (9)$$

where:

- I. C. = instrument constant
- D_f = diameter of float, inches
- ρ_f = float density, grams per cubic centimeter
- ρ = fluid density, grams per cubic centimeter
- W_f = weight of float in air, grams

$$I. C. = (0.375) \sqrt{(1.04) \left(\frac{2.28 - 1.28}{2.28} \right) (1.28)} \times (453.6) \quad (10)$$

(Note: Value of 453.6 is flow unit conversion factor for converting pounds per minute to grams per minute, as taken from Fischer and Porter instructions⁽⁹³⁾.)

$$I. C. = 129.5 \quad (11)$$

Step 4. Use of Ball Float Characteristic Curve X-1006,
Figure 53:

Locate viscous influence number of ordinate of curve X-1006, and read up to intersection with various D_t/D_f ratio curves. Read across from intersections and locate values of the simplified flow coefficient, K, for the various ratios of D_t/D_f .

Step 5. Tabulate values of D_t/D_f , K, I. C., and the resulting flow in desired units:

$$\text{Flow} = (K) (I. C.) \quad (12)$$

See Table XI.

Step 6. Plot of a calibration curve, diameter ratio tube scale versus flow:

See Figure 50, page 127.

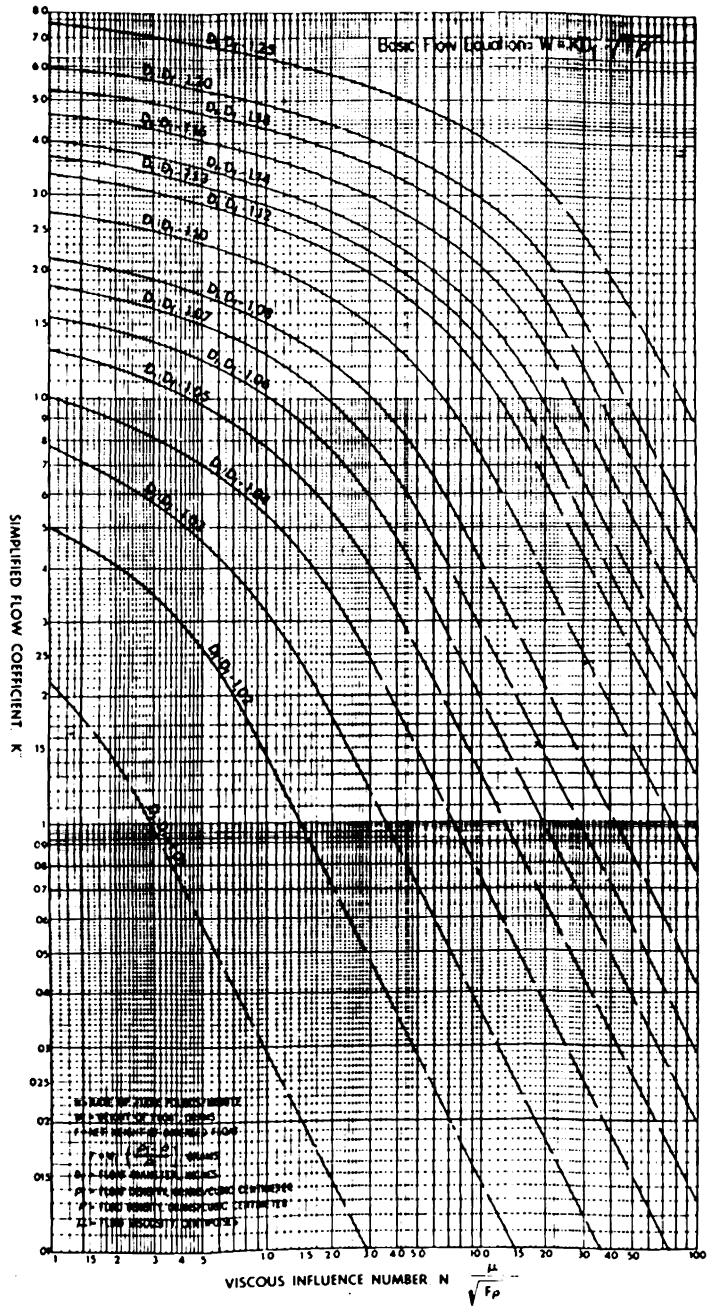


Figure 53. Ball Float Characteristic Curve X-1006

The F & P TRI-FLAT Variable-Area Flow Meter Handbook,
p. 14, Catalog 10-A-91, Fischer and Porter Company,
Hatboro, Pa., 1953.

TABLE XI

Calibration Data for Extract Flow Meter

D_v/D_f	K	I. C.	Flow gm/min
1.25	6.10	129.5	790.0
1.20	4.70	129.5	609.0
1.18	4.15	129.5	537.0
1.16	3.55	129.5	460.0
1.14	3.00	129.5	389.0
1.12	2.45	129.5	318.0
1.10	1.95	129.5	252.0
1.08	1.40	129.5	181.0
1.06	0.93	129.5	120.0
1.04	0.47	129.5	60.9
1.02	0.11	129.5	14.3

Calculation of Stage Efficiency. Data for test 4-2 are used for these calculations.

$$S = \frac{y_A - y_0}{y_E - y_0} \times 100 \quad (13)$$

where:

- S = stage efficiency, per cent
- y_A = actual concentration of acetone in the extract phase, weight per cent
- y_0 = concentration of acetone in the solvent, weight per cent
- y_E = ideal concentration of acetone in the extract phase, weight per cent

(Note: y_E for this test is 11.1 weight per cent, calculated by graphical methods as presented by Bull and Coli⁽⁹⁾.)

$$S = \frac{10.6 - 0.0}{11.1 - 0.0} \times 100 \quad (14)$$

$$S = 95.5 \% \quad (15)$$

IV. DISCUSSION

The following section includes the discussion of results, recommendations, and limitations imposed on the performance of the designed and constructed liquid-liquid extractor.

Discussion of Design and Construction

Performance of the extractor, as designed and constructed, was basically that anticipated. The over-all design and construction was found to be of a sound nature. A question of optimum design for sub-assemblies arose in two instances during the evaluation test. One case was that of the circulating lines to and from the constant temperature bath for the feed and solvent, and the second was that of the electrical leads from switches on the swinging electrical panel to the individual circuits.

Constant Temperature Circulating Lines for Feed and Solvent.

It was learned through operation of the extractor that the feed and solvent liquids that were circulated through the constant temperature bath could be forced back into the air pressure line unless careful attention was given to attaining a low flow rate. The lines returning to the tanks from the constant temperature bath utilized the same inlet pipe fitting at the top of the tanks as the air line. This arrangement should be

changed and the line containing the circulating liquid should be run directly into the tanks through their own fittings and not through the same one employed by the air line.

Electrical Wiring. Present arrangement is not the most suitable for the electrical leads going from the individual circuit switches located on the swinging electrical panel to the small vertical panel for connection to the corresponding circuits. The present wiring is satisfactory, however, the bending of the wires, which takes place when the swinging panel is opened and closed, was responsible for several broken wires. Flexible braided wire only should be employed where it is subject to bending.

Discussion of the Reactor

The reactor was the sub-assembly of the extractor that required the greatest time and consideration.

Design. The glass reactor employed in photographic studies, Figure 22, page 73, evolved from numerous considerations, the principal one was that a conical shape was employed to utilize the energy of the conically-focused ultrasonic waves.

Construction. Glass was preferred as the construction material because its transparency allowed photographs to be

made of the two-phase liquid mixing taking place in the reactor. Attachment of the acoustical window, 0.001-inch nickel sheet, to the glass reactor body was the difficult part of the reactor construction. A technique employing a silver surface, deposited on the glass reactor by heating a silver paste painted on the edge to 1100 °F, and a layer of copper plated on the silver permitted a bond to be obtained between the nickel and glass that withstood the high pressures and liquid surges found in the reactor during operation. First attempts at bonding the nickel sheet to the glass reactor were not satisfactory and prompted the construction of a reactor from sheet tin. This metal reactor was fabricated to withstand the extreme conditions of pressure and flow rates that could appear in an evaluation test of an extensive nature.

Liquid Mixing in the Reactor. A study was initiated of flow patterns that occurred in the reactor, the prime consideration of which was to observe the mixing effects in the reactor both in the presence and absence of insonation. Cavitation occurring in the liquid during insonation was observed also.

Operation without Insonation. Figure 54 shows the reactor operating with a solvent to feed ratio of approximately 1:1 and flow rates for feed and solvent of approximately 300 grams per minute each. Mixing of the liquids



Figure 54. Reactor in Operation without Insonation

in the reactor without insonation was accomplished by the turbulence created by the entering two phases.

Operation with Insonation. Figure 55 shows the reactor operating under insonation of 400 kilocycles frequency and an intensity level equivalent to 190 milliamperes plate current. The flow rates of feed and solvent were the same as in the previous test, Figure 54. Small cavities were formed in the liquid by action of the ultrasonic waves, and could be distinguished, by turbulence, from the particles of the light phase dispersed in the heavy phase. A continued investigation of cavitation by means of high-speed motion pictures would illustrate uniquely the mixing and gross stirring effect obtainable with ultrasonic insonation which cannot be adequately represented by still photography or in descriptive phrases.

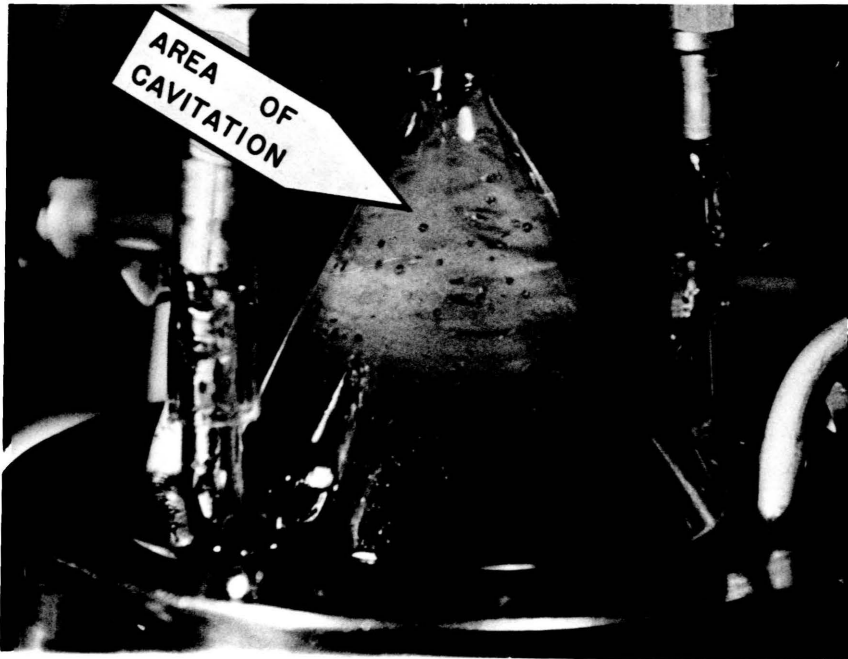


Figure 55. Reactor in Operation with Insonation

Discussion of Extractor Operation

The two-phase system acetone-water-1,1,2-trichloroethane was employed in an evaluation test designed for a study of the operating characteristics of the extractor. Inasmuch as this was the first operation of the extractor as a unit, its performance was quite satisfactory. As anticipated, there were operational characteristics observed that should be altered for improvement.

Collection of Samples. Operation of the sample valves was satisfactory. However, occasionally a siphon effect was created in the liquid lines when a sample valve was opened and air was drawn in, disturbing equilibrium conditions of the system. A simple re-design of the placement of the sample valves will remedy this difficulty.

Electrical Measurements. The total current drawn by electrical equipment of the extractor was in the range of one-fifth that estimated for maximum load. For this reason, readings on the ammeter were on the lower portion of the non-linear scale where the accuracy of the meter reading was not satisfactory. Incidentally, meters with linear scales were ordered but could not be supplied within the time required. The ammeter presently in use has a rating of ten amperes. An ammeter with a linear

scale and rating on the order of two amperes should be installed in the extractor.

Discussion of Extraction Results

Mass transfer which occurred in the test system was investigated to the extent of determining stage efficiencies for each individual test conducted.

Variance of Stage Efficiency with Flow Rates and Insonation.

Of eleven individual tests, eight calculated stage efficiencies were over 100 per cent, ranging from 101.1 to 110.0 per cent. The remaining three tests had efficiencies ranging from 94.3 to 98.0 per cent, in the range anticipated for this extractor for this system. There was no definite order to the change in stage efficiencies of the different tests. No reliable correlation can be made between extraction efficiency and flow rates or insonation using data from this particular series of extraction tests on the system acetone-water-1,1,2-trichloroethane.

Content of Extract Samples. A clear, light yellow color, similar to that of freshly distilled nitrobenzene, was very evident in the samples taken of the extract phase during the evaluation test. This color indicated an acquired impurity in the sample, as the three components of the feed and solvent under study were clear. It is thought that some foreign matter

such as process oil, pipe dope, or a protective coating inside the tanks was present in the system and was dissolved by the 1,1,2-trichloroethane. This impurity in the sample would cause a refractive index to be read that was not representative of the acetone content of the sample. As the nature and concentration of impurities were not known, the refractive index of each sample was reported as found. To eliminate the possibility of such an occurrence happening during other extraction studies employing this extractor, it is suggested that the extractor first be flushed with the different solvents and the solvents analyzed for possible contamination before study of the particular system is undertaken.

Recommendations

This extractor was specifically designed and constructed for use with the Brush HYPERSONIC Generator and 400-kilocycle barium titanate, bowl-shaped transducer. The following recommendations are made on the basis of continued use of this equipment. Utilization of other ultrasonic equipment would require modifications of the extractor that will not be discussed.

Reactor Design. A study should be continued as to the possibilities of using the conically-shaped glass reactor for extraction studies. Introduction of feed and solvent streams into the reactor by means of jets should be investigated. The turbulence thus provided by the design of the reactor added to that provided by the insonation should provide a more effective method of extraction than either one alone.

Pumps. Centrifugal pumps capable of developing approximately 30 to 40 pounds per square inch pressure, gage, and delivering five to ten gallons liquid per minute, should be substituted for the small pumps presently in use. These pumps should be specifically designed for continuous operation.

Feed and Solvent Circulation Lines. It is recommended that the tubing arrangement of the lines handling circulation of feed and solvent through the constant temperature bath be redesigned.

The lines going back to the tanks from the constant temperature bath should go directly into the tank and not into the common tubing line coming out of the one 1/4-inch pipe fitting at the top of each tank. This proposed arrangement would eliminate any possibility of liquid being forced back into the air line.

Sample Valves. Present tubing leading from main streams to sample valves should be changed. Tubing for liquid streams to be sampled should be connected on the run of the tubing tee, and tubing to the reactor or receiving tanks should be connected to the side port of the tee. This arrangement would cause the sample valve, when open, to be the line of least resistance to liquid flow and would eliminate any difficulties in sample collection due to a siphon effect.

Flow Meter Filters. Individual line filters should be placed in the inlet line of each flow meter to trap any foreign matter in the lines that could contaminate the floats in the meters.

Recovery Column. A recovery system for solvents is needed in the immediate vicinity of the extractor. This arrangement would allow the operator to carry on purification of any accumulated extract or raffinate while attending to test being conducted with extractor. Needed area for such recovery equipment is available.

Ammeter. An ammeter of two ampere rating and with a linear scale should be installed in the extractor to replace the present ammeter of ten ampere rating and non-linear scale.

Limitations

Performance of the liquid-liquid extractor as designed and constructed was controlled by the following limitations.

Feed and Solvent Capacity. Operating tank capacity for each was 20 gallons.

Flow Rates. Using a liquid of specific gravity of 1.0 as basis of measure, flow rates were obtainable for any one liquid, feed or solvent, of 0.132 pound per minute minimum to 12 pounds per minute maximum.

Solvent-to-Feed Ratio. On basis of above flow rates for one liquid, solvent-to-feed ratios could be varied over the range of 1:100 to 100:1. A ratio of 1:1 was employed in the extractor evaluation test.

Reactor. The reactor, or contacting vessel, for this extractor was a special design of conical shape, with a volume of 12.8 cubic inches (0.21 liter).

Settling Column. The settling column employed has a total volume of 37.4 cubic inches (0.61 liter) and an interface between

phases of 3.14 square inches. Samples were taken from single phase lines after the extract and raffinate left the settling column.

Ultrasonic Equipment. The Brush HYPERSONIC Generator employed in this investigation has a rated maximum output of 250 watts, with variable frequency from 100 to 1000 kilocycles. The transducer used in conjunction with the HYPERSONIC Generator was a focused, bowl-shaped, barium titanate ceramic element of approximately 400 kilocycles frequency.

Temperature Measurements. Temperature measurements were made at seven points in the extractor with copper-constantan thermocouples.

System Studied. Mass transfer in the system acetone-water-1,1,2-trichloroethane was observed in the evaluation test. Feed mixtures of approximately 20 weight per cent acetone, water as the carrier solvent, were employed.

V. CONCLUSIONS

A liquid-liquid extractor utilizing ultrasonic energy was designed, constructed, and tested with the two-phase, three-component system acetone-water-1,1,2-trichloroethane. A special conically-shaped glass reactor was developed and used in conjunction with a Brush HYPERSONIC Generator of 250 watts power and a bowl-shaped, barium titanate ceramic transducer. This investigation led to the following conclusions:

1. A liquid-liquid extractor having maximum combined flow rates of feed and solvent of approximately 24 pounds per minute and incorporating an ultrasonic generator of 250 watts power output for insonation of the liquid system under study was constructed and operated on a practical basis for laboratory scale, experimental work in the field of mass transfer.

2. Four-hundred kilocycle insonation of the two phases of the liquid system acetone-water-1,1,2-trichloroethane while contained by the special conically-shaped reactor created a mixing of the two phases that was in excess of that mixing observed solely from fluid flow turbulence in the reactor without insonation.

VI. SUMMARY

This investigation was conducted to provide laboratory scale equipment that will facilitate an accurate study of the effect of ultrasonic energy on mass transfer in two-phase, multiple-component liquid systems.

A liquid-liquid extractor incorporating an ultrasonic generator and transducer was designed and constructed to the following specifications: (1) insonation frequency of 400 kilocycles per second, (2) insonation intensities equivalent to plate currents of 0 to 200 milliamperes, and (3) flow rate of solvent and feed through the reactor ranging approximately from one-half to 24 pounds per minute, in varying solvent-to-feed ratios.

A special glass reactor, or contactor, was constructed from a standard, 60° pyrex glass funnel and fitted with an acoustical window of 0.001-inch sheet nickel. Photographic studies were made of the two-phase mixing taking place inside the reactor in both the presence and absence of ultrasonic insonation. An all metal reactor, of the same general design as the glass reactor, was constructed for use with the extractor when investigations were to be made that would involve high pressures or sudden liquid surges through the reactor.

An evaluation test of the extractor was conducted employing the system acetone-water-1,1,2-trichloroethane. Stage efficiencies calculated for the individual tests, eleven in all, varied from 94.3 to 110.0 per cent. An observed yellow color in the extract complex, probably due to dissolved impurities in the 1,1,2-trichloroethane, could have been responsible for the observation of refractive index readings that did not give true representation of the acetone concentration of the sample.

Cavitation was observed in the reactor while the test system was undergoing ultrasonic insonation. The gross stirring effects resulting from cavitation in the liquids caused a mixing of the two phases that was more intense than that taking place in the reactor without insonation.

VII. BIBLIOGRAPHY

1. Austerweil, O. V.: Applying Elastic Untrasound Waves, L'Auxiliaire des chemins de fer et de l'industrie, Fr. Patent 841,359, May 17, 1939; C. A., 34, 4189 (1940).
2. Benedict, M.: Multistage Separation Processes, Trans. Am. Inst. Chem. Engrs., 43, 41-61 (1947).
3. Bergmann, L.: "Der Ultraschall und seine Anwendung in Wissenschaft und Technik," pp. 66-150. VDI-Verlag, Berlin, Germany, 1942. 3 ed.
4. _____: "Der Ultraschall und seine Anwendung in Wissenschaft und Technik." Zurich, S. Hirzel Verlag, 1954. 6 ed.
5. Brinsmade, D. S. and H. Bliss: Extraction of Acetic Acid in Methyl Isobutyl Ketone with Water in a Wetted Wall Tower, Trans. Am. Inst. Chem. Engrs., 39, 679-713 (1943).
6. Brown, G. G., et al: "Unit Operations," pp. 302-319. John Wiley and Sons, Inc., New York, N. Y., 1950.
7. Bryant, W. H. D., J. C. Elgin, J. H. Perry, F. D. Rossini, and J. C. Whitwell: Physical and Chemical Principles, "Chemical Engineers' Handbook" (J. H. Perry, Editor), p. 318. McGraw-Hill Book Co., Inc., New York, N. Y., 1950. 3 ed.
8. Buchanan, R. H.: Liquid-liquid Equilibrium Data for the System Carbon Tetrachloride-Acetone-Water, Ind. Eng. Chem., 44, 2449-2450 (1952).
9. Bull, F. W. and G. J. Coli: Graphical Methods as Applied to Extraction Problems, Virginia Polytechnic Institute, Blacksburg, Va., Engr. Exp. Sta. Bull., No 72 (1949).
10. Butterworth, J. W.: Kilowatts of Ultrasonic Power for Industry, "Ultrasonics--two symposia." Chem. Eng. Prog. Sym. Series, No 1, 47, pp. 69-75 (1951).

11. Cady, W. G.: "Piezoelectricity," p. 2. McGraw-Hill Book Co., Inc., New York, N. Y., 1946. 1 ed.
12. *ibid*, p. 4.
13. *ibid*, p. 683.
14. Campbell, H. and C. A. Long: Emulsification by Ultrasonics, *Pharm. Jour.*, 163, 127-128 (1949).
15. Carlin, B.: "Ultrasonics," p. 259. McGraw-Hill Book Co., Inc., New York, N. Y., 1949. 1 ed.
16. _____: Material Testing and Gaging with Ultrasonics, "Ultrasonics--two symposia." *Chem. Eng. Prog. Sym. Series, No 1*, 47, pp. 43-46 (1951).
17. Colburn, A. P.: The Simplified Calculation of Diffusional Processes, General Consideration of Two-Film Resistances, *Trans. Am. Inst. Chem. Engrs.*, 35, 211-236 (1939).
18. _____: Simplified Calculation of Diffusional Processes, *Ind. Eng. Chem.*, 33, 459-467 (1941).
19. _____ and R. L. Pigford: General Theory of Diffusional Operations, "Chemical Engineers' Handbook" (J. H. Perry, Editor), p. 459. McGraw-Hill Book Co., Inc., New York, N. Y., 1950. 3 ed.
20. Coplan, B. V., J. K. Davidson, and E. L. Zebroske: The "Pump-Mix" Mixer Settler, a New Liquid-liquid Extractor, *Chem. Eng. Prog.*, 50, 403-408 (1954).
21. Danser, H. W.: Eliminate Stack Dusts and Mists, *Chem. Eng.*, 57, 5--158 (1950).
22. _____ and E. P. Neumann: Industrial Sonic Agglomeration and Collection Systems, *Ind. Eng. Chem.*, 41, 2439-2442 (1949).
23. Elgin, J. C.: Design and Applications of Liquid-liquid Extraction, *Chem. and Met. Eng.*, 49, 5--110 (1942).

24. Elgin, J. C. and R. Wynkoop: Solvent Extraction, "Chemical Engineers' Handbook" (J. H. Perry, Editor), p. 715. McGraw-Hill Book Co., Inc., New York, N. Y., 1950. 3 ed.
25. *ibid*, pp. 729-746.
26. *ibid*, pp. 747-752.
27. Francis, A. W.: Solvent Extraction with Liquid Carbon Dioxide, *Ind. Eng. Chem.*, 47, 230-233 (1955).
28. Frankel, Y.: *Acta Physicochim*, U. R. S. S., 12, 317-322, (1940); *C. A.*, 34, 7682 (1940).
29. Glasstone, S.: "Textbook of Physical Chemistry," p. 735. D. Van Nostrand Co., Inc., New York, N. Y., 1946. 2 ed.
30. Gray, W. C.: The Effect of Ultrasonic Irradiation on the Specific Reaction Rate Constant in the Acid Hydrolysis of Ethyl Acetate. Unpublished Ph. D. Thesis, Library, Virginia Polytechnic Institute, Blacksburg, Va. (1952).
31. Haul, R., H. H. Rust, and J. Lutzow, *Naturwissenschaften*, 37, 523 (1950); Treybal, R. E.: Solvent Extraction, *Ind. Eng. Chem.*, 45, 58-67 (1953).
32. Hayford, D. A.: The Design, Construction and Operation of a Liquid-liquid Pulse Extractor. Unpublished M. Sc. Thesis, Library, Virginia Polytechnic Institute, Blacksburg, Va. (1953).
33. Hiedemann, E. A.: Basic Principles of the Application of Sonic and Ultrasonic Energy, "Ultrasonics--two symposia." *Chem. Eng. Prog. Sym. Series*, No 1, 47, 51-56 (1951).
34. Hunter, T. G. and A. W. Nash: The Application of Physicochemical Principles to the Design of Liquid-liquid Contact Equipment, Part I, *J. Soc. Chem. Ind.*, 51, 285T (1942).
35. _____: The Application of Physicochemical Principles to the Design of Liquid-liquid Contact Equipment, Part II, *J. Soc. Chem. Ind.*, 53, 95T (1944).

36. Judah, M. A., E. M. Burdick, and R. G. Carroll: Chlorophyll by Solvent Extraction, *Ind. Eng. Chem.*, 46, 2262-2271 (1954).
37. Kinsler, L. E. and A. R. Frey: "Fundamentals of Acoustics," p. 133. John Wiley and Sons, Inc., New York, N. Y., 1950.
38. Laddha, G. S. and J. M. Smith: Mass-transfer Resistances in Liquid-liquid Extraction, *Chem. Eng. Prog.*, 46, 195-202 (1950).
39. Lewis, J. B.: Liquid-liquid Extraction, Atomic Energy Research Establishment Report CE/R 910, Her Majesty's Printing Office, Great Britain (1953).
40. _____ and H. R. C. Pratt, *Nature*, 171, 1155 (1953); Lewis, J. B.: Liquid-liquid Extraction, Atomic Energy Research Establishment Report CE/R 910, Her Majesty's Printing Office, Great Britain (1953).
41. Lewis, W. K. and W. G. Whitman: Principles of Gas Absorption, *Ind. Eng. Chem.*, 16, 1215-1220 (1924).
42. Loomis, A. L. and R. W. Wood: The Physical and Biological Effects of High Frequency Sound Waves of Great Intensity, *Phil. Mag.*, 4, 7--417 (1928).
43. Maloney, J. C. and A. E. Schubert: The Application of Rectangular Coordinate Methods to Solvent Extraction Design, *Trans. Am. Inst. Chem. Engrs.*, 36, 741-757 (1940).
44. Mason, W. P.: "Piezoelectric Crystals and Their Application to Ultrasonics," pp. 234-389. D. Van Nostrand Co., New York, N. Y., 1950.
45. Mattiat, O.: The Processing Possibilities of Ultrasonics, *J. Acous. Soc. Amer.*, 26, 241-243 (1954).
46. Morello, V. S. and N. Foffenberger: Commercial Extraction Equipment, *Ind. Eng. Chem.*, 42, 1021-1035 (1950).

47. Murray, L. P.: Effect of 400-kilocycle Insonation on the Extraction of Acetone from Carbon Tetrachloride with Water. Unpublished M. Sc. Thesis, Library, Virginia Polytechnic Institute, Blacksburg, Va. (1954).
48. National Research Corp.: Liquid-liquid Extractor, Chem. Engr., 61, 1--286 (1954).
49. Raven, F. A., A. M. Feiler and Anna Jespersen: An Annotated Bibliography of Cavitation, U. S. Navy Dept. Report R-81, David Taylor Model Basin, Washington, D. C. (1947).
50. Rayleigh, L.: On the Pressure Developed in a Liquid During the Collapse of a Spherical Cavity. Phil. Mag., 34, 94-98 (1917).
51. Rees, J. H.: The Effect of Ultrasonic Vibrations on Diffusion. M. Sc. Thesis, M. I. T. (1949).
52. Richards, W. T.: The Chemical Effects of High Frequency Sound Waves II. A Study of Emulsifying Action, J. Amer. Chem. Soc., 51, 1724-1729 (1929).
53. _____: Supersonic Phenomena, Rev. Mod. Phys., 11, 54-64 (1939).
54. Richardson, C. N.: Ammonia Synthesis. U. S. Pat. 2,500,008, March 7, 1950.
55. Richardson, W. G.: "Ultrasonic Physics," p. 1. Elsevier Publishing Company, New York, N. Y., 1952. 1 ed.
56. Richardson, M. L. F.: Apparatus for Warning a Ship of Its Approach to Large Objects in a Fog, British Pat. 9423 (1912); Bergmann, L.: "Der Ultraschall und seine Anwendung in Wissenschaft und Technik," p. 301. VDI-Verlag, Berlin, Germany, 1942. 3 ed.
57. Rushton, J. H.: Mixing of Liquids in Chemical Processing, Ind. Eng. Chem., 44, 2931-2936 (1952).

58. Saal: World Petroleum Congress, London, 1933, Proceed.,
2, 354 (1933); Varteressian, E. A. and M. R. Fenske:
Liquid-liquid Extraction, Exact Quantitative Relations,
Ind. Eng. Chem., 28, 1353-1360 (1936).
59. Samsel, R. W.: Physical and Economic Limitations in the
Application of Sonic and Ultrasonic Energy to Industrial
Processing, "Ultrasonics--two symposia." Chem.
Eng. Prog. Sym. Series, No 1, 47, pp. 77-81 (1951).
60. Scheibel, E. C.: Absorption Theory and Design Calculations,
Chem. and Met. Eng., 49, 5--102 (1942).
61. Segs, G. and F. W. Woodfield: Pulse Column Variables,
Chem. Eng. Prog., 50, 396-402 (1954).
62. Sherwood, T. K. and R. L. Pigford: "Absorption and Extrac-
tion," p. 393. McGraw-Hill Book Co., Inc., New York,
N. Y., 1952. 2 ed.
63. Skrzec, A. E.: Mass Transfer Studies in Countercurrent
Liquid-liquid Extraction Systems. Unpublished Ph. D.
Thesis, Library, Virginia Polytechnic Institute,
Blacksburg, Va. (1953).
64. Sellner, K.: Sonic and Ultrasonic Waves in Colloid
Chemistry, "Colloid Chemistry," Vol 5, (J. Alexander,
Editor), pp. 337-342. Reinhold Publishing Corp.,
New York, N. Y., 1944. 1 ed.
65. _____: Colloidal Effects of Ultrasonics, "Ultra-
sonics--two symposia." Chem. Eng. Sym. Series No 1,
47, pp. 28-37 (1951).
66. _____ and C. Bondy: On the Mechanism of Emulsifica-
tion by Ultrasonic Waves, Trans. Fara. Soc., 31,
835-842 (1935).
67. _____: The Influence of Gases on
Mercury Emulsions Prepared by Ultrasonic Waves,
Trans. Fara. Soc., 31, 843-846 (1935).
68. _____: Quantitative Experiments on
Emulsification by Ultrasonic Waves, Trans. Fara.
Soc., 32 (1936).

69. Sollner, K. and C. Bondy: The Mechanism of Coagulation by Ultrasonic Waves, Trans. Fara. Soc., 32, 616 (1936).
70. Stokes, C. A. and J. E. Vivian: Applications of Sonic Energy in the Process Industries, "Ultrasonics-- two symposia." Chem. Eng. Prog. Sym. Series, No 1, 47, pp. 11-21 (1951).
71. Thompson, D.: Ultrasonics-- a New Chemical Engineering Tool, Chem. Eng. Prog., 46, 3-6 (1950).
72. _____: Effect of Ultrasonic Energy on Settling of Solids in Phosphate Tailing, Ind. Eng. Chem., 46, 1172-1180 (1954).
73. _____: Personal Communication, December 1, 1954. Blacksburg, Virginia.
74. _____: Effect of Ultrasonic Insonation on Solvent Extraction of Oil from Peanuts. Paper given at Christmas Symposium of Ch E Div ACS, Cincinnati, Ohio, January 7 and 8, 1955.
75. Treybal, R. E.: "Liquid Extraction," p. 97. McGraw-Hill Book Co., Inc., New York, N. Y., 1951.
76. *ibid*, p. 125.
77. *ibid*, p. 204.
78. *ibid*, p. 290.
79. _____, L. D. Weber, and J. F. Daley: The System Acetone-Water-1,1,2-Trichloroethane, Ind. Eng. Chem., 38, 817-821 (1946).
80. Twissaus, H. C. and N. J. Ehlers: Caustic Purifications by Liquid-liquid Extraction, Chem. Ind., 63, 231 (1948).
81. Ultrasonic Scoreboard, Chem. Week, 76, 7-52 (1955).
82. Varteressian, K. A. and M. R. Fenske: Liquid-liquid Extraction, Exact Quantitative Relations, Ind. Eng. Chem., 28, 1353-1360 (1936).

83. Varteressian, K. A. and M. R. Fenske: The System Methylcyclohexane-Aniline-n-Heptane, Ind. Eng. Chem., 29, 270-277 (1937).
84. Vermeulen, T., G. N. Williams, and G. E. Langlois: Interfacial Area in Liquid-liquid and Gas-liquid Agitation, Chem. Eng. Prog., 51, 85F-94F (1955).
85. Von Berg, R. L. and H. F. Wiegandt: Liquid-liquid Extraction, Chem. Eng., 59, 6--189 (1952).
86. Wiegandt, H. F. and R. L. Von Berg: Key to Better Extraction, Chem. Eng., 61, 7--183 (1954).
87. Weinhardt, A. E. and A. N. Hixson: Separation of Chromium and Vanadium Salts by Liquid-liquid Extraction, Ind. Eng. Chem., 43, 1676-1689 (1951).
88. Weisser, A.: Chemical Effects of Intense Ultrasonic Waves in Liquids, Naval Research Laboratory Report S-3483, Washington, D. C., June 15, 1949.
89. _____: Sonochemistry: The Production of Chemical Changes with Sound Waves, J. Acous. Soc. Amer., 25, 651-657 (1953).
90. Whitman, W. G.: The Two-film Theory of Gas Absorption, Chem. and Met. Eng., 29, 146-148 (1923).
91. Willard, G. W.: Ultrasonically Induced Cavitation in Water-- a Step-by-step Process, J. Acous. Soc. Amer., 25, 669-687 (1953).
92. Wood, A. B.: "A Textbook of Sound," p. 3. G. Bell and Sons, Ltd., London, England, 1946. 2 ed.

Addenda

93. Fischer, K.: How to Predict Calibration of Variable-Area Flow Meters, Chem. Eng., 59, 6--180 (1952).

VIII. ACKNOWLEDGMENTS

Dr. Dudley Thompson is responsible for the origin of this particular investigation. Without his guidance and understanding of the many general and special problems confronted by the investigator during the past year, this present goal would never have been reached. Sincere appreciation from the author, along with a desire for continued professor-student association, goes to Dr. Thompson.

Without the constructive criticism and many helpful suggestions given by Dr. F. W. Bull, other members of the Department of Chemical Engineering teaching staff, and various Chemical Engineering graduate students, many important features of this equipment would not have been realized at this time. A very personal "Thanks!" goes to each person in the above groups. Special appreciation goes to Mr. M. A. Price for information and assistance during construction of the unit.

Co-workers of the author in the Ultrasonic Laboratory, Mr. L. P. Murray and Mr. N. N. Bakhshi, have contributed in many ways to the accomplishment of this investigation. Their assistance has been very important in the execution and completion of this project. Appreciation goes to Mr. Murray for his assistance in the actual construction and operation of this unit.

The final typing of this thesis was done by Mrs. E. P. Ellison, a very important person to most graduate students of the Chemical Engineering Department. Along with many others, the author wishes to express his appreciation for her part in helping to reach the final point of a particular graduate program.

Dr. J. W. Murray of the V. P. I. Chemistry Department has given gratuitously of his time and knowledge in construction of the special reactor. Without the work of Dr. Murray, it is doubtful that the reactor desired would have been realized.

This project has been sponsored by the Virginia Engineering Experiment Station, with an equipment grant from Socony-Vacuum Oil Company. The author is indebted to both organizations for financial support during this investigation.

To Dr. C. E. Littlejohn, Department of Chemical Engineering, Clemson College, goes a special appreciation for his original interest in undergraduate education of the author and his untiring efforts directed towards the author's accomplishment of graduate work.

The author's parents, Mr. and Mrs. H. A. Woodle, deserve the greatest and warmest appreciation for their inspiration throughout the years, especially during the past year. Without

their influence and backing, the author would never have been able to accomplish the work represented by this thesis.

To Sue goes an appreciation only she can understand. The understanding and interest she has shown in this work have been very important to the author. Her influence during the course of this work, although not appearing directly in this thesis, has had a great and lasting effect on the author.

**The three page vita has been
removed from the scanned
document. Page 1 of 3**

**The three page vita has been
removed from the scanned
document. Page 2 of 3**

**The three page vita has been
removed from the scanned
document. Page 3 of 3**