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CONTROLLING VARIABLES IN LIQUID-LIQUID EXTRACTION FROM SINGLE DROPS

By
William S. Farmer

Date Issued

APR 20 1950

OAK RIDGE NATIONAL LABORATORY
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Post Office Box P
Oak Ridge, Tennessee

NOTE: This report has been submitted in partial fulfillment of the requirements for the degree of Master of Science in Chemical Engineering at the University of Tennessee, 1949.

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CHAPTER I

ABSTRACT

Equilibrium distribution data and extraction rates are presented for water-solvent systems using acetic acid as a solute. Expressions are derived for correlating extraction rates for both the turbulent and transitional flow regime by means of hydrodynamic and diffusional concepts.

The extraction rate for falling drops has been investigated for stripping acetic acid from the dispersed solvents-carbon tetrachloride, benzene, toluene, chloroform, methyl isobutyl ketone, and isopropyl ether- into a continuous water phase. It is possible to define the rate of extraction in the turbulent regime by means of an overall mass transfer coefficient based on a fictive film in the dispersed phase. In the transitional regime of fall the rate of extraction obeys the diffusion law for spherical particles.

Over the turbulent regime of fall, which extends above a Reynolds number of 300 to 350, it is possible to express the overall extraction coefficient by the relation:

$$\frac{K_o d}{D_i} = 0.03 \left(\frac{dV\rho_d}{\mu_d}\right)^{0.8} \left(\frac{\mu_d}{\rho_d D_i}\right)^{0.44} \left(\frac{dV^2\rho_c}{I.T.}\right)^{0.8}$$

where K_o - overall mass transfer coefficient
d - drop diameter
D_i - molecular diffusivity
μ - viscosity
ρ - density
I.T. - interfacial tension
V - velocity
subscript d - dispersed phase
c - continuous phase

The resulting overall mass transfer coefficient must be multiplied by a correction factor, (2-n)/n, when stripping acetic acid from a dispersed non-polar organic liquid, where n is the degree of association. This factor represents the fraction of the solute molecules which exist as single molecules in the organic liquid. Since acetic acid exists in water, as unassociated molecules, the rate of extraction is approximately equal to the rate at which the single acetic acid molecules diffuse across the laminar boundary film of the dispersed organic liquid. The rate of dissociation of the double molecules in this same laminar film seems to be sufficiently slow, that its contribution to the rate of extraction is minor in relation to that of the single molecules.

In the transitional regime, over the range of Reynolds number from 100 to 300, the resulting rate of extraction agrees with the diffusion equation. The overall mass transfer coefficient is given by:

$$K_o = \frac{L}{A} \ln \left\{ \frac{\pi^2}{6} \frac{1}{\sum_{n=1}^{\infty} \frac{1}{n^2} e^{-\left(\frac{n\pi}{r_o}\right)^2 D_i \theta}} \right\}$$

where L - flowrate of the dispersed phase
A - interfacial area
θ - time of contact

No experimental data are given for the viscous range, since modifications would have had to be made in the apparatus to provide sufficient pressure to obtain drops of a small diameter. However, most industrial applications involve either the transitional or turbulent flow

regime and hence the previous correlations are more useful for design purposes. The relations involved in the viscous region would be expected to involve the diffusion equation with the resistance of the continuous phase also becoming important.

In investigating the hydrodynamics of the falling drop column, drop deformations were observed in the turbulent flow regime. The deformation increased with diameter, approaching a limiting value such that the major diameter is twice the minor. The velocity, as a function of drop diameter, passes through a maximum in the turbulent regime and then decreases in contrast to the behavior predicted by Newton's law for falling spheres. The effects of deformation on both the velocity and the rate of extraction have been taken into account in the correlation for the turbulent flow regime by including the Weber capillary group.

Large end-effects were found to occur in the falling drop column. Although no correlation was developed for this effect, it was found that the added extraction occurred during formation of the falling drop at the nozzle. The end-effects are not only a function of the properties of the dispersed phase but also depend on the size of the nozzle, the size of the drop formed, and the time of drop formation.

In order to correlate the rate of mass transfer in solvent extraction systems in terms of film coefficients, a thorough knowledge of not only the hydrodynamics but also the chemistry and molecular diffusivity is needed. The rate of extraction is governed not only by association and

and disassociation of the solute molecules, but also by forces of attraction between the solute and solvent molecules, such as with solvation. Insufficient data on these phenomena plus the lack of reliable correlations for the molecular diffusion coefficient hinder the evaluation of rate constants for many solvent extraction operations. Once the chemistry of solvent extraction systems is understood, and reliable hydrodynamic correlations have been obtained, the derivation of rate equations for packed columns should be possible.

CHAPTER II

INTRODUCTION

Liquid-liquid extraction has, (during the last few years) been the subject of many articles in the technical literature. However, most of these investigations have been concerned with performance data for specific conditions and equipment. Theoretical correlations, such as those of Colburn,[7] have been used with partial success in explaining the results. These correlations cannot be extrapolated outside the range of conditions for which the experiments were performed, since in general, the variables were not separated.

If solvent extraction is to be placed on as strong a basis as heat transmission, and if extraction rate coefficients are to be predicted, it will be necessary to develop more fundamental information. It will not be possible to correlate packed column extraction data in any more than an empirical fashion until the mechanisms involved are thoroughly understood.

The investigation of solvent extraction rates in industrial equipment involves not only the study of hydrodynamics but also the study of reaction rates and the study of mass transfer. In order to correlate solvent extraction data it must be possible to calculate or determine by measurement the hydrodynamics of the system involved. Hence, a packed column is not satisfactory for a fundamental investigation, since seldom is either the area or the manner of flow either measurable or static.

The relations governing mass transfer coefficients, which do not involve a kinetic reaction, should by analogy to heat and momentum transfer be a function of the viscosity, density, and interfacial tension of the fluids, as well as a function of the hydrodynamic variables fluid dimension and velocity.

The objectives of the program of investigation undertaken were thus defined as:

1. The selection of an extraction apparatus for which the hydrodynamics could be determined.

2. The choice of solvent-solute extraction systems whose chemical behaviors are similar.

3. The correlation of the rate of extraction in terms of the physical and chemical variables mentioned above by successive variable elimination.

4. Evaluation of the results, to obtain extraction rate coefficients suitable for design of spray and perforated plate columns.

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CHAPTER III

PREVIOUS INVESTIGATIONS

A preliminary literature search was made for information on the chemistry of solute-solvent systems, the hydrodynamics of countercurrent liquid-liquid flow, and the rate of extraction, in both experimental and industrial equipment. The chemistry of solute-solvent systems is discussed in most physical chemistry text books in a descriptive manner. There appeared to be no information on rates of reaction for the association - dissociation reaction in non-polar liquids. Also, the exact magnitude and effect of solvent-solute forces was mentioned in only a qualitative manner.

Although hydrodynamics has been the subject of many investigations, the hydrodynamics of liquid-liquid systems is not well understood. Correlations for the flow of fluids through packed beds can be found in books by Sherwood[23] and books by Perry,[20] These however, are empirical and do not explain the fluid mechanics of liquid-liquid flow. Strang, Hunter and Nash[24] established the hydrodynamics of flow in wetted-wall towers. Their work indicated that the change from streamline to turbulent flow occurs at a Reynolds number dependent upon the physical properties and motion of the two fluids in contact, and that as a result, turbulence occurs at a lower value of the Reynolds number than for flow in circular pipes.

The hydrodynamics of falling solid spheres has been extensively investigated and is presented in books by Vennard[26] and other authors. The data do not apply to fluid spheres when they deform in shape.

The available information most applicable to the hydrodynamics of liquid drops is that for systems of gas bubbles dispersed in liquids. By making use of analogies, the information of O'Brien and Gosline,[19] and Miyagi[18] can be used to interprete the hydrodynamics of liquid drop systems.

The hydrodynamics of liquid-liquid flow in a slanted tube column was investigated by Bergelin and Lockhart.[3] They observed in their glass column the effects of flow on the interface separating two counter-current flowing liquids. The interface was found to be well defined and probably stagnant when the mass velocities of the two liquids are the same. When the mass velocities are quite far apart, extreme turbulence and wave formation were observed at the interface.

Investigations on the rate of mass transfer have been made for almost all the types of industrial equipment in use today. The vast majority of the results are reported in terms of such concepts as Colburn's[7] H.T.U., or as H.E.T.S. values, or by means of the volumetric overall transfer coefficient, Ka. Most of these are applicable to a particular situation and cannot be extrapolated far from the conditions for which the experiments were run.

Sherwood, Evans and Longcor[22] investigated extraction in both the single drop tower and in packed columns. Overall mass transfer coefficients

were determined as a function of drop diameter in a single drop tower for several solvents. Appreciable extraction was found to occur during drop formation, or within a short distance of the nozzle. The uncorrected overall mass transfer coefficients were compared with the results predicted by the laws of diffusion and were found to be much larger. No efforts were made to relate the results of the single drop tower to the variables of the systems employed. The remainder of their investigation was devoted to packed columns. The effect of both continuous and dispersed phase flow rates on the overall volumetric mass transfer coefficient was measured and the importance of these two variables in packed column performance noted.

Bergelin and Lockhart[3] also investigated the extraction of isopropyl alcohol from a tetrachlorethylene liquid phase to a water phase for counter-current liquid flow in a horizontal tube. They were able to estimate their interfacial area and thus to compute values of K_o. When the mass velocities of the two liquids were the same, the overall mass transfer coefficient passed through a minimum, due probably to a zero interfacial velocity which created a stagnant film in each phase. The highest rates of extraction were observed when the interface was in a condition of turbulence.

Important advances have been made in relating the basic mechanism of extraction to the properties of the liquids involved using wetted-wall columns. Fallah, Hunter and Nash[12] found the kerosene or wall-film

coefficient with the system water-phenol-kerosene in a wetted-wall column to be expressed by:

$$\frac{kd_i}{D_i} = 0.94 \left(\frac{d_i V \rho}{\mu}\right)^{0.8} \left(\frac{u}{D_i \rho}\right)^{0.46}$$

A major disadvantage with the use of wetted-wall columns is that the velocity of the wall liquid must be 5 to 20 times that of the core liquid in order to impart stability to the system. As a result, the overall mass transfer coefficient depends largely upon the rate of diffusion in the core liquid, and changes in the velocity of the core liquid produce larger changes in the overall mass transfer coefficient than do velocity changes in the wall liquid.

Studies on the effect of core and wall fluid flowrates on both the individual and overall extraction rates have been made by Colburn and Welsh,[8] Treybal and Work,[25] and Comings and Briggs.[9] Brinsmade and Bliss[5] determined the overall extraction coefficient for the extraction of acetic acid from methyl isobutyl ketone with water. By various simplifying assumptions they were able to separate the individual film coefficients to obtain:

$$\frac{k_o d}{D_c} = 1.07 \ (Re_c)^{0.67} \left(\frac{\mu}{\rho_c D_c}\right)^{0.62} \left(\frac{\mu_w}{\rho_w^2 g}\right)^{1/3}$$

$$\frac{k_w}{D_w} = 0.00135 \ (Re_w) \left(\frac{\mu_w}{\rho_w D_w}\right)^{0.62}$$

Ketone was used as the core liquid and water as the wall liquid in the above experiments.

Spray towers and sieve plate columns have been the subject of many investigations in recent years. Most of these have been made using multiple spray nozzles. As a result, the exact drop dimensions have not been known, and the results are usally expressed as volumetric overall transfer coefficients. Elgin and Browning[11] studied the transfer of acetic acid between water and isopropyl ether and presented an analysis for the theory of spray towers.

Johnson and Bliss[16] as well as Appel and Elgin[1] studied the variation of Ka with drop diameter. The overall volumetric mass transfer coefficient was found to be a maximum in the range of drop diameters from 0.1 to 0.2 inches. In addition changes in the dispersed phase velocity had little effect on the extraction coefficient over the range of drop diameters from 0.1 to 0.36 inches. A precise interpretation of the data in all of these experiments was impossible because of lack of information on the interfacial area.

It is apparent from the literature survey that it is necessary to determine the exact interfacial area in an extraction apparatus, if the fundamental relationships are to be evaluated. In the literature reviewed, the mass transfer coefficient appeared to depend upon both the molecular diffusivity and the flow rate. Thus:

$$K_L \propto D\ V^n \text{ where n varies from 0 to 1}$$

In equipment where mass transfer may be thought of as being across films, the overall mass transfer coefficient can be expressed in terms of the physical properties of the system by using analogies to heat and momentum transfer. In the case of spray columns, not only will these same physical properties affect the film coefficient, but also the surface tension should enter the correlation.

CHAPTER IV

PROBLEM APPROACH

From a revue of previous investigations and the objectives of this program it was possible to set up criteria for investigating the controlling variables in solvent extraction. It was apparent that the interfacial area must be known if the effect of variables such as the physical properties of the system on the extraction coefficient were to be determined.

The only apparatus available for which the interfacial area could be measured were the single drop tower, the slanting horizontal tube column and the wetted-wall column.

The slanted tube column was not used because of the large liquid flows resulting from the use of a column of sufficient diameter to eliminate wall effects. The wetted-wall column was not used because of operating difficulties associated with forming a thin liquid film on the wall and with controlling flow rates. Since the single drop tower such as employed by Sherwood, Evans and Langcor[22] had none of these disadvantages it was selected for the investigation.

The specific design of the falling drop tower was chosen to minimize the complexity of interpreting the data. Thus the tower diameter was made much larger than the drop diameter so that the wall would have no effect on the hydrodynamics of the falling drop. The

effective column height was varied using the same column by varying the distance that the nozzle extended into the column.

The dependence of the concentration analysis on an exact material balance was eliminated by using a small volume ratio of dispersed phase through-put to continuous phase. The material balance and driving force were then based on the dispersed phase analysis. Further, water was used as the continuous phase in the majority of experiments and the solute stripped from the dispersed phase. This made it possible to discard the continuous phase after each run.

From previous investigations it was apparent that the film coefficient for mass transfer is a function of the physical properties of the film and the film dimensions. In order to separate the affects of each of these variables, only one property was varied at a time. Then by mathematical analysis each property was introduced into the final correlation. This method of separating variables should give a more satisfactory correlation than that which could be obtained by making assumptions using dimensionless groups. Unfortunately, many data are needed to correlate a small number of factors by this method.

The chemical systems employed were selected for ease of solute concentration analysis in both phases. Two immiscible solvents were used as the two liquid phase and only one solute entered the diffusion process from one phase to the other. To facilitate the computation of the driving force a solute was chosen which was preferentially soluble

in the continuous water phase. Finally the two immiscible solvents were chosen with a large interfacial tension, since many reagents are available for lowering the interfacial tension but few for raising it.

Because water was to be used as the continuous phase it was necessary to select an organic liquid which had a large interfacial tension and low miscibility with water. There are many organic liquids which are immiscible with water, however, because of its large interfacial tension, carbon tetrachloride was chosen.

Iodine was first chosen as a solute because of its ease of chemical analysis and because it did not react with the two liquid solvents. However, due to its insolubility in water the iodine was soon discarded in favor of acetic acid as a solute. Unfortunately, acetic acid associates in many organic liquids. This obstacle is offset partially by the ease with which its concentration can be determined by chemical analysis. In addition the distribution ratio of acetic acid between water and carbon tetrachloride varies only slightly with the concentration change in each run.

The original technique of manipulating one variable at a time was not successful due to the complications introduced by the additives required to vary each of the physical properties. In order to determine the influence of the physical properties of the solvents, other organic liquids were substituted for carbon tetrachloride and experiments performed under identical conditions so that results could be evaluated by means of determinants.

CHAPTER V

THEORY

As was pointed out in Chapter III, there is little or no information on individual film coefficients for mass transfer in solvent extraction operations. As a result concepts have had to be developed for expressing the results of extraction operations. Most of the concepts have been obtained by analogy to gas absorption.

In order to study the basic mechanism of extraction the two film theory as proposed by Whitman[27] can be used. The use of this expression in gas absorption has been eminently successful. In the expression for the overall mass transfer coefficient for gas absorption, the gas film coefficient can be replaced by a second liquid film coefficient. This expression can be used then for liquid-liquid extraction.

$$K_{o_s} = \frac{L}{\frac{1}{K\,k_w} + \frac{1}{k_s}}$$

or

$$K_{o_w} = \frac{L}{\frac{K}{k_w} + \frac{1}{k_s}}$$

where $K = \Delta C_w / \Delta C_s$

In order to determine extraction coefficients on a sound basis, it is advantageous to analyse the results in terms of the individual film coefficients. The choice of the falling drop column is admirably suited for this purpose. As the dispersed falling drops descend through the continuous column phase they come into contact with fresh continuous phase. As a result a concentration gradient does not build up in the continuous phase film and almost all the resistance to transfer is in the dispersed phase. In addition, by dispersing the organic solvents and making the water phase continuous, it is possible to obtain large values of K, the distribution ratio, for the systems used here. Hence, $K_{o_s} \approx k_s$.

By means of the kinetic theory of mass transfer, the rate equation for the resistance of the dispersed film can be written:

$$L\, dC_s = k_s A \quad (C_i - C_s) \quad dA$$

where

- C = concentration of solute
- L = dispersed phase flow rate
- k_s = dispersed phase film coefficient
- A = interfacial area
- θ = time of contact
- H = column height
- V = dispersed phase or interfacial velocity

The solution of this equation is dependent upon a knowledge of the concentration at the interface. Although this cannot be measured, the systems used have been chosen in order to estimate this concentration. Since the continuous phase film coefficient is negligible; $C_i \approx C_w$. The ratio of the capacity of the continuous phase to that of the dispersed phase is large hence the concentration of the water phase is approximately zero or is at least a negligible value.

It is possible to derive a simple expression for the individual film resistance by integrating the equation above, setting $C_i = 0$.

$$L\, dC_s/C_s = -k_s DA$$

Integrating over column height H for a change in concentration of the dispersed phase from feed (C_f) to product (C_r) gives:

$$\ln (C_f/C_r) = k_s A/L$$

By rearranging the expression the overall mass transfer coefficient based on the dispersed phase can be expressed in terms of the individual dispersed phase film coefficient.

$$K_{o_s} = k_s = \frac{L \ln (C_f/C_r)}{A}$$

Another concept frequently used for correlating extraction rates is that of the height of a transfer unit or H.T.U. developed by Colburn.[7] The height of a transfer unit (H.T.U.) is expressed as:

$$\text{H.T.U.} = \frac{H}{\int_{C_f}^{C_r} \frac{dC}{C - C^*}} \qquad \frac{L}{KA}$$

In books by both Perry[20] and Sherwood[23] the relationships for the height of a transfer unit are fully developed for expressing extraction in terms of either or both phases.

The rate of extraction can also be calculated in terms of the height equivalent to a theoretical stage (H.E.T.S.). This has the distinct disadvantage that the values obtained vary with the solute

concentration. As a consequence, it is difficult to interpret the result in terms of the physical properties of the liquid phase.

In correlating the results of the experiments reported here, the method using the overall extraction coefficient, K_o, was employed. The concepts upon which this method is based are particularly applicable to the falling drop tower. In addition, the mathematics involved is simple and straight forward.

The concepts mentioned so far are most useful for correlating data in the turbulent flow regime. In this regime there is eddying and mixing in the turbulent liquid core and the primary resistance can be thought of as being in a thin outer laminar film across which the solute must transfer by molecular diffusion. In the regime of viscous or streamline flow there is no mixing or eddying and the fluid is either stagnant or moves in viscous layers. In this latter regime the laws of true diffusion apply.

If the dispersed drops are assumed to be perfect spheres, the rate of extraction in the viscous region of flow can be expressed by the diffusion equation in spherical coordinates.

$$\frac{\partial C}{\partial \theta} = D_i \left[\frac{\partial^2 C}{\partial r^2} + \frac{2}{r} \frac{\partial C}{\partial r} \right]$$

The solution of this problem is outlined in the Appendix. For the boundary conditions met with in the experiment reported here, the solution becomes:

$$\frac{C_r}{C_f} = \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} e^{-\left(\frac{n\pi}{r_o}\right) D_i \theta}$$

If the flow within the liquid drops consists of a viscous vortex, diffusion equations similar to the above in cylindrical coordinates can be derived in order to obtain the rate of extraction. The flow pattern for vortex formation is in the form of a doughnut and can be simulated by a cylinder of infinite length. A complete solution of this problem can be found in the Appendix.

In selecting the systems for investigation, one of the idealized criteria is that of a solute which functions as the same species in all liquids under investigation. In addition secondary reactions should not occur. In many practical experiments this condition cannot be obtained and reactions occur sometimes in series or parallel with the diffusion of the solute. The handling of such data is extremely difficult but several methods of doing so have been presented by both Wilhelm[29] and Sherwood[23] for the simpler situations.

The main resistance whether it be thought of as a film, for turbulent flow, or the entire mass, for viscous flow, involves transfer by molecular diffusion. Hence the computation of mass transfer coefficients is dependent on the accuracy with which the molecular diffusion can be predicted. The mechanism of diffusion of a solute molecule through

a liquid involves the kinetics of liquids. Since little is known on this subject, only empirical correlations are presented for computing the diffusion coefficients, no one of which is precise.

Arnold[2] developed an empirical correlation for liquid diffusion coefficients given by:

$$D_i = \frac{B}{A_a A_b (\mu_b)^{\frac{1}{2}}} \quad \frac{1}{(V_a^{1/3} + V_b^{1/3})^2} \quad \sqrt{\frac{1}{M_a} + \frac{1}{M_b}}$$

where

A_a = solute abnormality factor
A_b = solvent abnormality factor
B = constant = 0.0156 at 68° F.
= 0.0143 at 59° F.

The usefulness of Arnold's equation is quite limited however, due to a lack of sufficient information to evaluate the abnormality factors and other terms involved in the expression above.

The relationship between diffusion and liquid viscosity was brought out by the classical Stokes-Einstein equation which was derived for the diffusion of large spherical molecules through a solvent made up of small molecules.

$$D_i = \frac{KT}{6\, r \mu_b}$$

By applying the absolute rate theory to diffusion; Powell, Roseveare, and Eyring[21] derived modified the Stokes-Einstein equation in order to fit the observed diffusion coefficients more closely.

$$D_i = \frac{KT}{b(2r)\mu_b}$$

where b varies from 3π for large spherical molecules to unity for self diffusion and has a value of 2π for large cylindrical molecules. The use of the equation is limited by lack of specific information on the shape of the solute molecules.

Although purely empirical, the recent correlation of diffusion coefficients presented by Wilke[30] is more workable than any of the others.

$$D_i = T\,F/\mu$$

Wilke developed expressions for evaluating F as a function of temperature, solute and solvent properties, and concentration. This equation is used in the appendix to compute molecular diffusion coefficients for correlating the overall extraction coefficients.

The hydrodynamics of falling solid spheres has been extensively investigated and correlated by using a drag coefficient defined as a function of the Reynolds number. An evaluation of the drag coefficient (c) as a function of Reynolds number can be found in books by Perry[20] or Vennard.[26] Unfortunately, in the turbulent flow regime liquid drops do not behave as perfect spheres. No information on the hydrodynamics of dispersed liquid drops in a continuous liquid medium could be found. However, many useful deductions can be drawn by analogy to correlations presented for gas bubbles rising through a liquid. This latter phenomenon has been extensively investigated by O'Brien and Gosline[19] and many others. The hydrodynamic relations are completely developed in the Appendix.

CHAPTER VI

APPARATUS

The experimental equipment for measuring the rate of liquid-liquid extraction is shown in Figures 1 through 6. Two columns of similar construction but different length were employed in determining the overall mass transfer coefficients. The columns could be assemblied in two different arrangements depending upon the relative densities of the dispersed and continuous medium. They were unpacked in all experiments.

The long column shown in Figures 4 and 5 is the same tower with different auxiliary equipment and with a different arrangement. The column consisted of a 3 inch outer pyrex glass tube surrounding a 2 inch inner tube. The outer tube was sealed to the inner at top and bottom to provide for a water cooling jacket. Tits made from 8 mm. tube were provided at the top and bottom of the jacket for attaching rubber hose which were in turn connected to the plant water supply. Thermometer wells were provided at the top and bottom of the jacket for insertion of Weston metal thermometers in order to measure the jacket water temperature.

Due to the hardening of plastics and the swelling of rubber by solvents it was necessary to use interchangeable or ball joints for all connections. Thus male ball joint fittings slightly greater than 2 inches in diameter were attached to the top and bottom of the large column. Finally 7 mm. tits were provided for hose connections about

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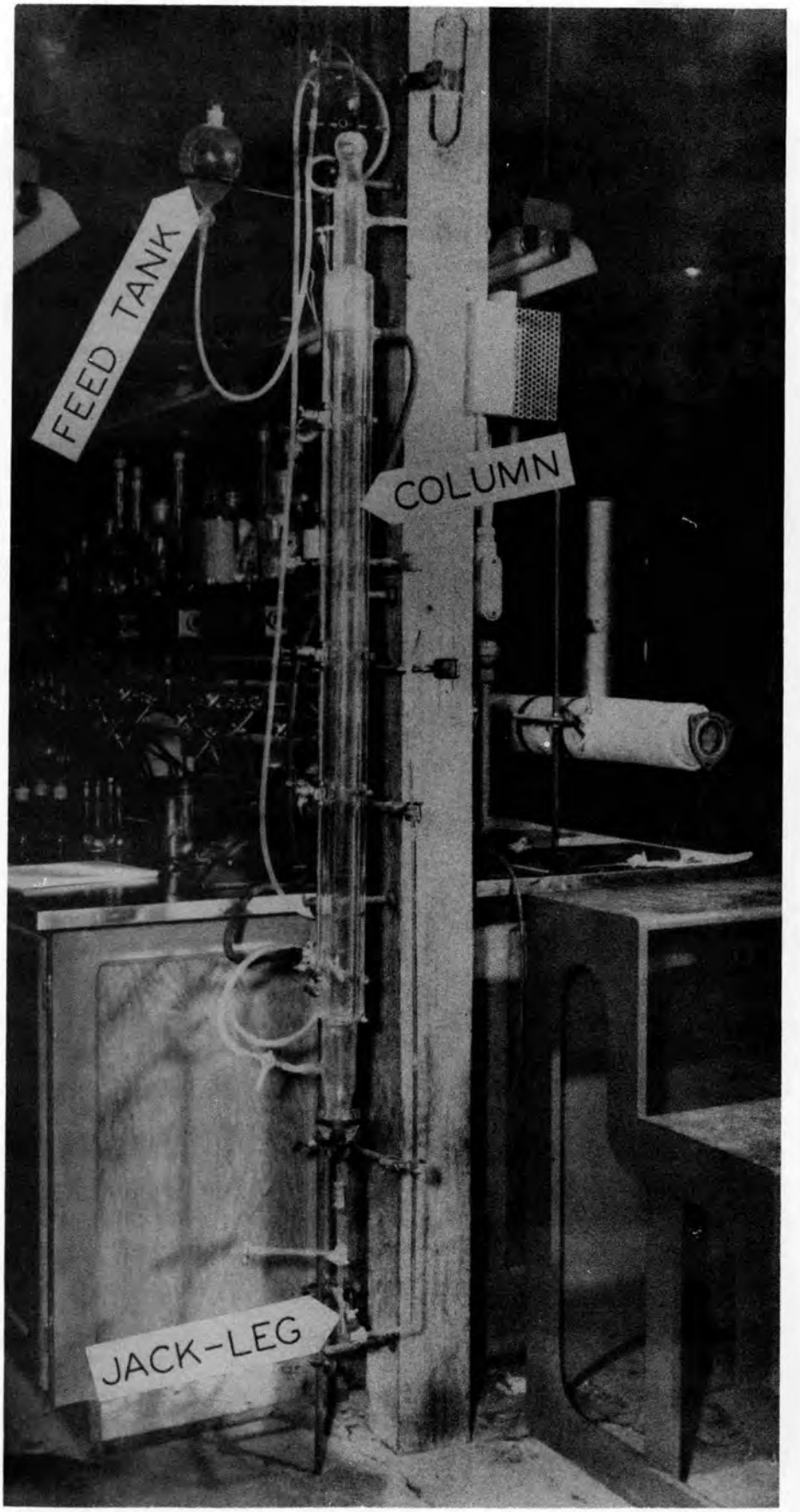

FIG. 1 EXTRACTION COLUMN AND APPARATUS FOR DISPERSING SOLVENTS HEAVIER THAN WATER

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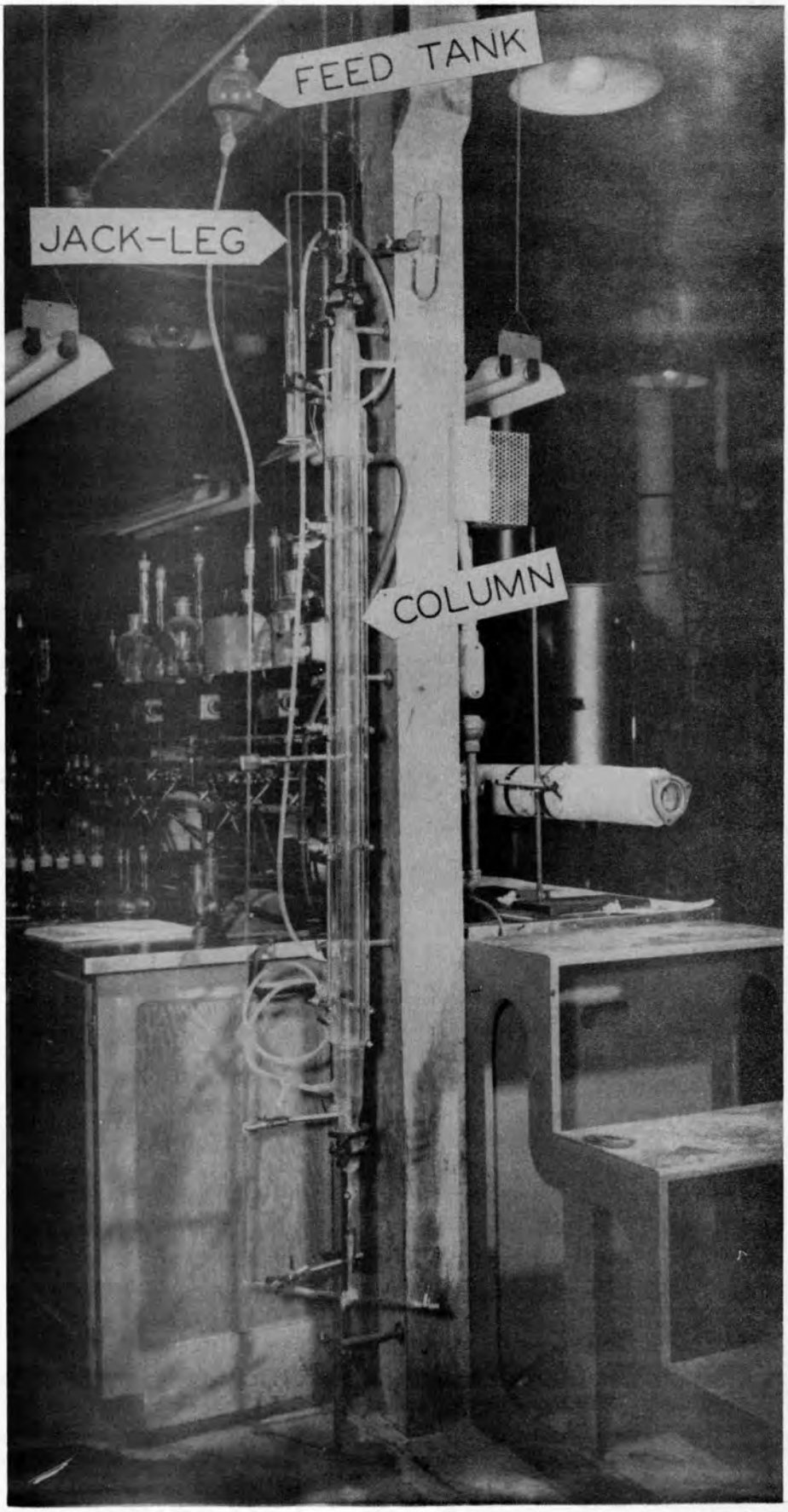

FIG. 2 EXTRACTION COLUMN AND APPARATUS FOR DISPERSING SOLVENTS LIGHTER THAN WATER

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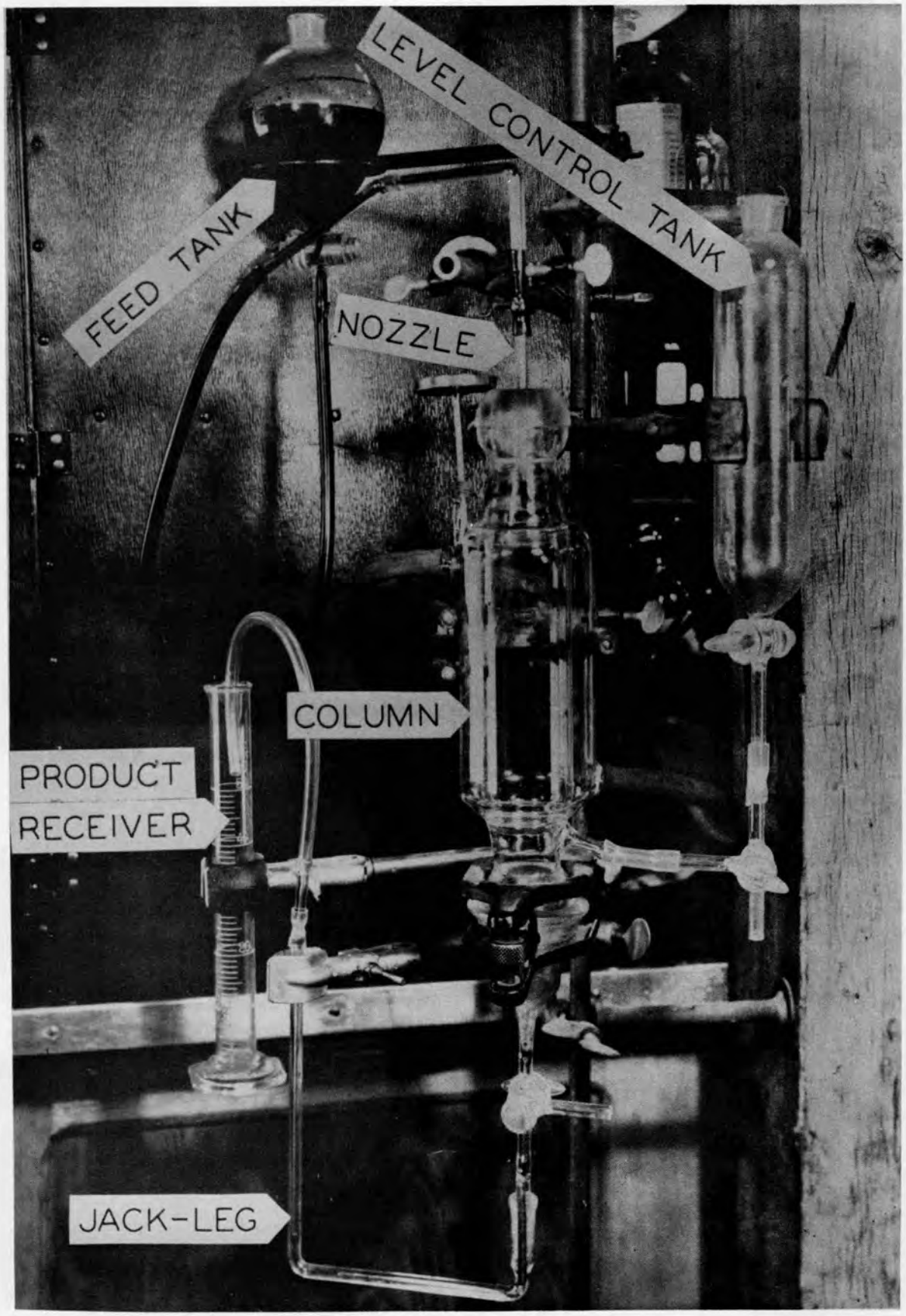

FIG. 3 EXTRACTION COLUMN AND APPARATUS FOR DETERMINING END-EFFECTS

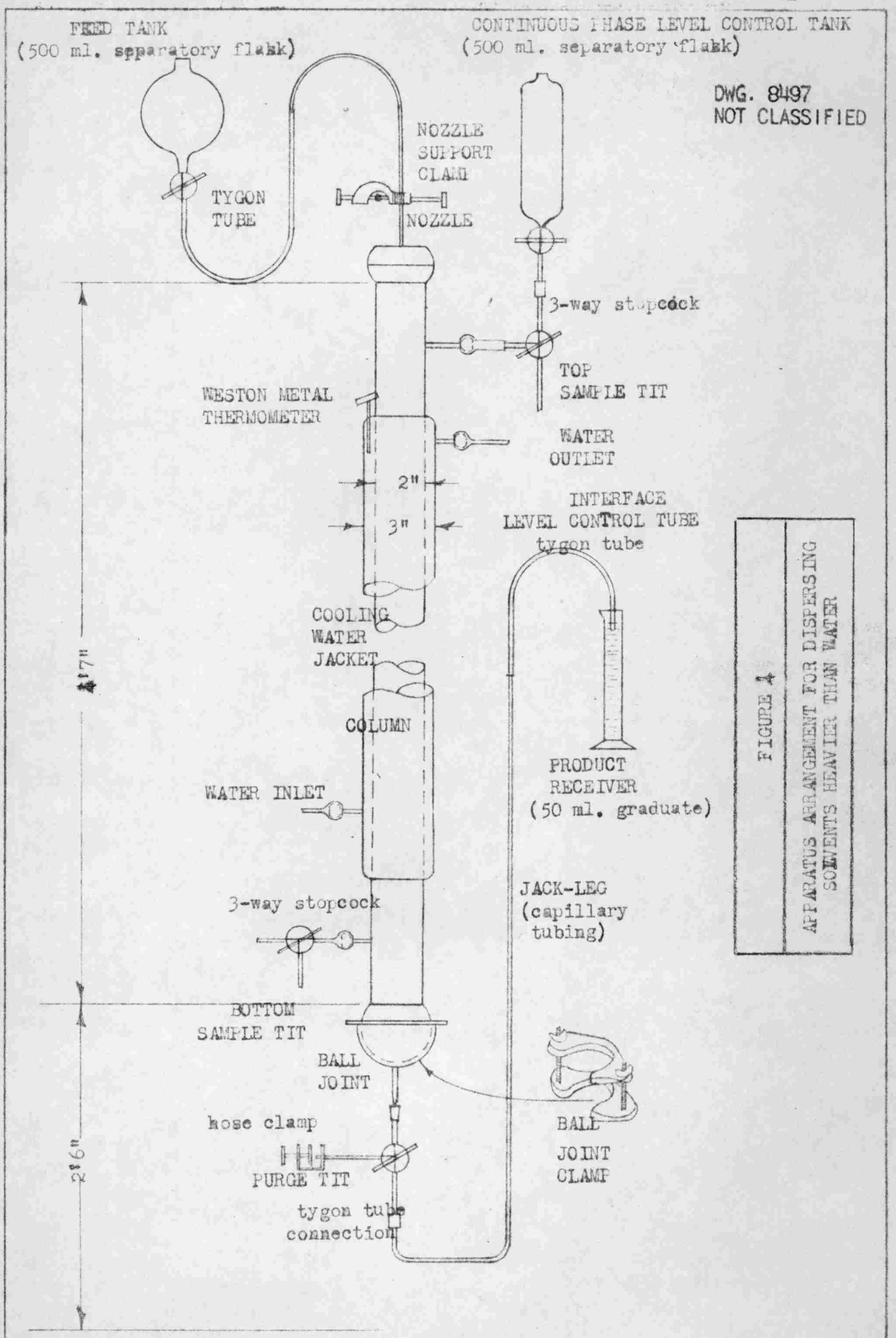

FIGURE 1

APPARATUS ARRANGEMENT FOR DISPERSING SOLVENTS HEAVIER THAN WATER

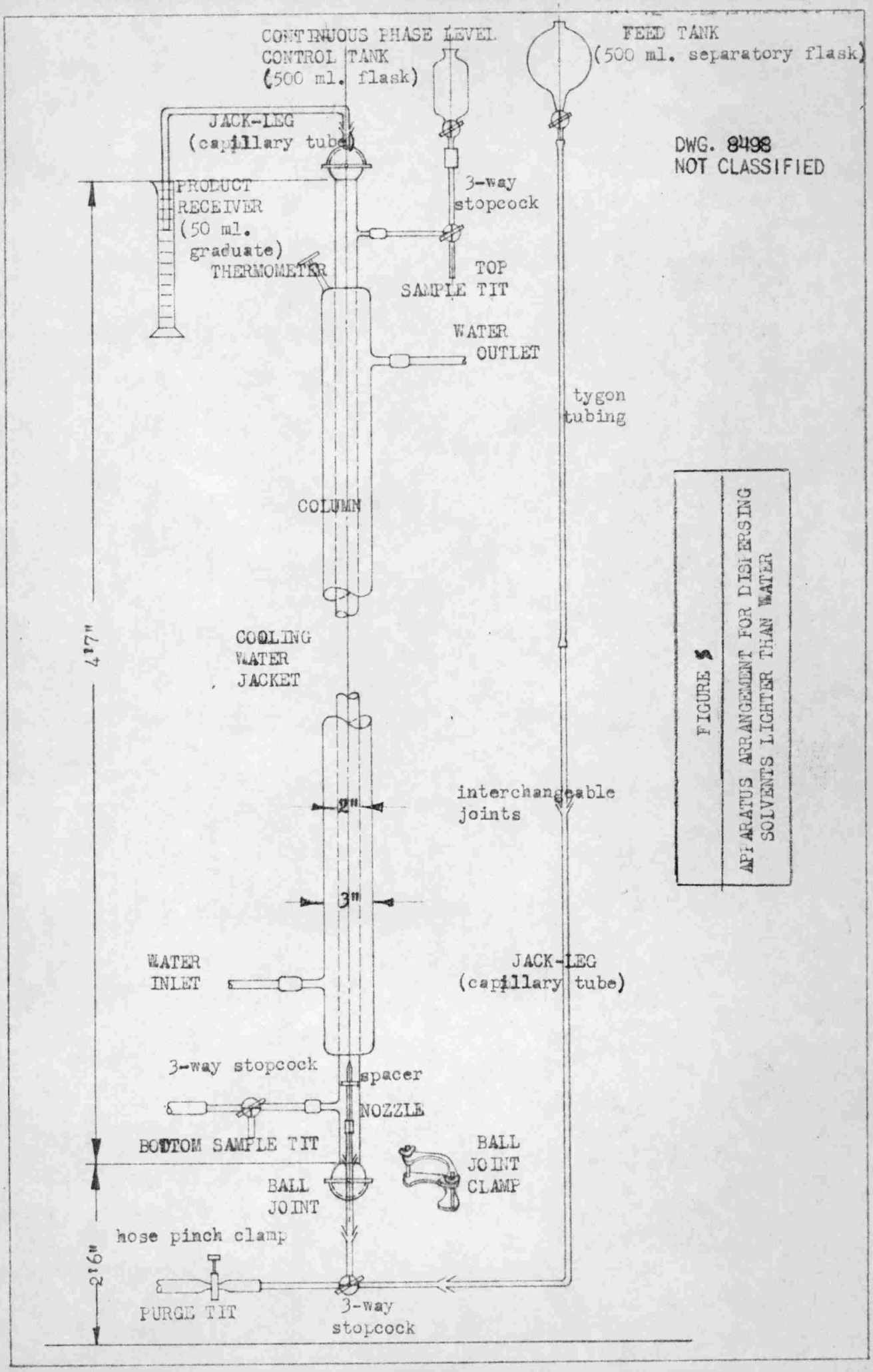

FIGURE 5

APPARATUS ARRANGEMENT FOR DISPERSING SOLVENTS LIGHTER THAN WATER

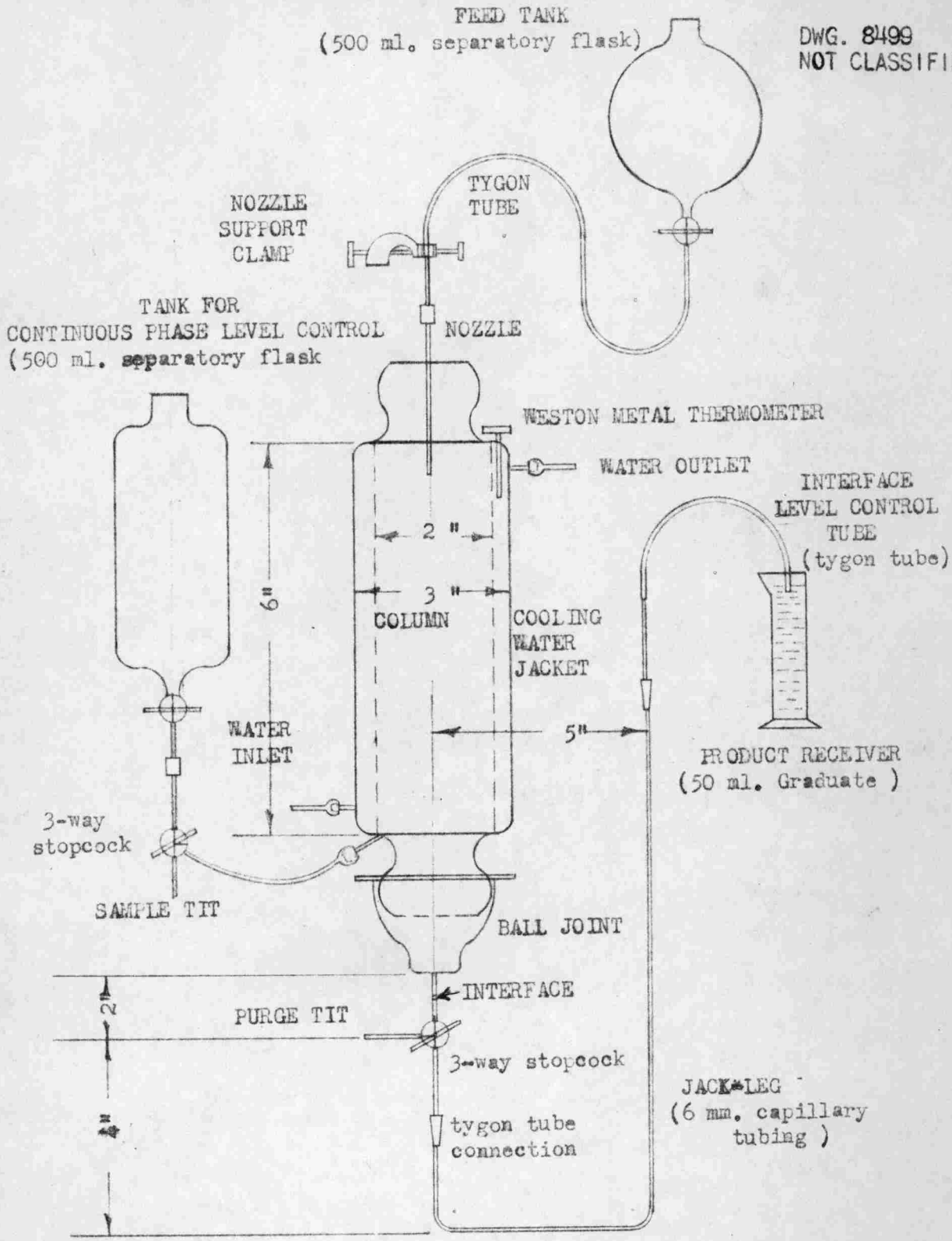

FIGURE 6

APPARATUS ARRANGEMENT FOR DETERMINING END-EFFECTS WHEN DISPERSING SOLVENTS HEAVIER THAN WATER

3 inches from the top and bottom of the inner glass tube in order to provide for continuous phase sampling and liquid level adjustment.

When dispersing solvents which are heavier than the continuous phase the arrangement of auxiliary equipment shown in Figures 1 and 4 was used. The column was left open at the top and the nozzle inserted through this opening. The nozzle was positioned by a clamp attached to the bar which supports the column. A connection was made between the 500 ml. separatory funnel used as a feed tank and the nozzle by means of tygon tubing. The tygon tubing being flexible, enables the feed tank, which is supported in a ring fastened to the main bar support to be raised or lowered. Adjustments in flow were made by raising and lowering the feed tank in conjunction with adjustment of the stopcock on the bottom stem of the feed tank. The top sample tit was connected through a three-way stopcock to a 500 ml. separatory funnel in such a manner that continuous phase liquid can be added to the column to adjust the continuous phase level, or a sample be removed.

A female ball joint fitting was clamped to the bottom male joint and held in place by a ball joint clamp as shown in Figure 4. A 6 mm. glass tube extended from this female ball joint and was connected by an interchangable joint to a three-way stopcock. In the first few runs, the product was removed by cracking the three-way valve so that the

continuous phase purged the dispersed solvent from the bottom of the column at a fixed rate. The mixture was caught in a 500 ml. separatory funnel. Although Sherwood, Evans and Longcor were successful in using this method of product removal, in the present case this procedure resulted in the purge tit acting as a very efficient secondary extractor and gave results which could not be reproduced. Hence, the bottom tit of the three-way valve was connected to a jack-leg of 6 mm. glass capillary tubing. A section of 6 mm. tygon tubing was fastened to the end of the jack-leg and inserted into the 50 ml. graduate used as a product receiver. By raising or lowering the tygon tube the hydrostatic balance was adjusted so the interface level was held constant in the tube extending from the bottom female ball joint. The dispersed phase filled the jack-leg and was carried over into the product receiver by displacement.

Two tits were provided for purging the continuous phase from the column after each run. The main bulk of the continuous phase was purged through the three-way stopcock attached to the bottom sample tit. The liquid remaining in the column below this tit is purged through the purge tit on the three-way stopcock on the jack-leg line.

When the dispersed phase is lighter than the continuous, the droplets rise through the continuous liquid and hence, the previous arrangement of auxiliary equipment was reversed. The feed tank was supported above the top of the column by a ring as shown in Figure 5.

It was connected by means of tygon tubing to a 6 mm. glass tube which ran parallel with the column to a level below the bottom ball joint. This L shaped glass tube was connected through an interchangeable joint to the side arm of a three-way stopcock which in turn was connected by its vertical arm to the stem from the bottom of the female ball joint fitting. The remaining arm of the three-way stopcock served as a purge tit.

A special female ball joint for supporting the nozzle was used at the bottom of the column. Instead of the stem being flush with the inside, it extended on up into the base of the column and had an interchangeable joint on top. The various nozzles employed were attached to the interchangeable joint.

The two sample tits and continuous phase liquid level control tank were arranged just as in the previous case for the heavy dispersed liquids. The female ball joint which was used on the bottom of the column previously was attached to the male ball joint fitting at the top and a U shaped 6 mm. glass tube connected to it by means of an interchangeable joint. This U shaped tube served as a jack-leg for product removal and dipped over into a 50 ml. graduate. An interface level was maintained in the stem from the top ball joint by adjusting the liquid level through the continuous phase level control tank.

It was possible to use the same column for column height experiments. By attaching glass tube extensions to the nozzles they could be moved any distance desired down or up the column. Rubber

spacers were used to center the extended nozzles in the center of the column. In general the liquid level was varied along with the height when the dispersed phase was heavier than the continuous. By lowering the liquid level the back pressure on the nozzle was reduced and better flow control was obtainable.

The short column shown in Figures 3 and 6 is identical in every detail to the long column just described except that the top sample tit was eliminated and the column length was 6 inches instead of 4 feet 7 inches. The short column was used for evaluating end-effects. The mode of operation was identical with that just described.

Additional auxiliary equipment was used for analyzing the solutions and making time and drop size measurements. The time required for the drops to fall the height of the column was determined by clocking the drops with a stopwatch. The overall experiment time was measured using an electric timer graduated in tenths of a second. Column heights were determined by measurement with a steel tape and nozzle dimensions determined by a micrometer.

CHAPTER VII

EXPERIMENTAL PROCEDURE

The experimental investigation covered both stripping and enriching operations for dispersing one liquid as drops in another. All chemicals used were C. P. grade to eliminate errors from impurities. For the same reason demineralized rather than tap water was employed as a continuous phase.

When the dispersed phase was heavier than the continuous, the apparatus was set up as described in Figures 1 and 4. The dispersed phase, containing the desired concentration of acetic acid was placed in the stoppered feed tank above the column. The three-way stopcock in the bottom stem was turned to the closed position and the column filled with the continuous phase. The nozzle to be used was then measured and attached to the feed tank by tygon tubing.

The nozzle was centered in the top of the column. By adjusting the height of the feed tank and cracking the stopcock below the feed tank a flow of approximately two drops per second was obtained. A steady flow could best be obtained by filling the feed tank only to the midpoint. This reduced fluctuations in height due to feed removal. Adjustments in flow were made by raising or lowering the feed tank.

The flow of dispersed phase into the bottom tit displaced the slight amount of continuous phase present at start-up and formed an interface. By cracking the three-way stopcock at intervals, the

jack-leg was completely filled with dispersed phase. Once the jack-leg had become full, the stopcock was turned to the open position and a hydrodynamic pressure balance and constant level interface obtained by raising or lowering the tygon tube on the jack-leg.

After 5 to 10 mls. of the dispersed phase had been purged through the jack-leg, the tygon tube was placed in the mouth of the product receiver and the electric timer started. The distance between the nozzle and interface was measured and the time of fall clocked by a stopwatch to determine the drop velocity. The drop rate was checked frequently and adjustments made to hold the rate constant.

When the level in the product receiver reached 50 mls., the three-way stopcock was closed, the timer stopped, and the feed tank stopcock closed. By means of the number of drops formed per second, the total time, and the total dispersed phase throughput, it was possible to compute the volume per drop. The diameter of the drops could then be computed by assuming their shape to be that of a perfect sphere.

The amount of acetic acid transferred was determined by titrating the samples with sodium hydroxide using thymol blue as an indicator. Samples were removed from the top and bottom of the column in 50 ml. volumetric flasks and transferred to 250 ml. erlenmeyers. Similarly a 50 ml. sample was removed from the feed

tank and transferred to a flask along with sufficient demineralized water to extract the acetic acid for titration. The entire contents of the product receiver was transferred to another flask along with demineralized water to aid the titration. From these chemical analyses and the sample volumes, it was possible to determine the amount of acetic acid transferred.

In the majority of experiments, water formed the continuous phase and an organic solvent containing acetic acid was dispersed. It was difficult to obtain a material balance based on the continuous phase due to the volume of dispersed phase purged through the jack-leg during the period of flow adjustment. Hence, the amount extracted was obtained by a material balance around the dispersed phase. Since demineralized water was expendable the continuous phase could be thrown away after each run and hence the initial continuous phase concentration was usually zero. When the column was filled to a height of 5 feet the continuous phase volume was 2500 mls. Thus the equilibrium concentration of the continuous phase could usually be considered as zero throughout the run and no error was entailed in basing the extraction on the dispersed phase analysis.

The method of operation when the dispersed phase was lighter than the continuous was essentially the same as that just described. The feed line in this case was filled almost up to the nozzle to prevent water from backing down the feed line. Usually a slight column of air separated the two, so extraction did not take place until the dispersed phase forced the air bubble out.

The end-effect column described in Figures 3 and 6 was operated in identical fashion to the large column. The procedure being just as above depending upon the relative densities of the two phases.

Capillary tubing ranging in inside diameter from 0.5 mm. to 1.5 mm. and standard tubing ranging in inside diameter from 5 mm. to 10 mm. were employed as nozzles. In some cases the tubing was drawn down to a fine point although usually a flat end was used. Thus care had to be exercised to be sure that the drop did not wet the horizontal nozzle surface when using flat-faced capillary tubing as this led to larger drops.

The room temperature was controlled by means of a wall thermostat and was held in the range of 20 to 30° C. The continuous phase temperature which was dependent upon the cooling water temperature followed the normal plant water temperature. This ranged from 12 to 20° C. Room temperature and water temperature were recorded for each run.

Samples were taken of all the solvents and the demineralized water. The specific gravity, viscosity, and interfacial tension were determined under approximately the same temperature conditions as those in the experiments. In addition, samples were taken for each concentration of feed and continuous phase in Series J runs, in which runs wetting agents were employed to lower interfacial tension. The density was determined by a Westphal balance, the viscosity by an Ostwald viscometer, and the interfacial tension by a DuNouy Interfacial Tensiometer.

CHAPTER VIII

EXPERIMENTAL RESULTS

Extraction rates were determined experimentally in a falling drop tower for water-acetic acid-solvent systems. The methods of calculation and experimental results are tabulated in Appendix G.

The experimental results can be broken down into two phases. In the first phase the basic system water-acetic acid-carbon tetrachloride was employed. This phase of the investigation consisted of varying one chemical property or condition at a time. Carbon tetrachloride containing 0.01 lb. moles/cu. ft. of acetic acid was dispersed in demineralized water in these runs. In the second phase, other solvents were substituted for carbon tetrachloride. Material balances were based on the dispersed phase in all runs.

The percent extraction and overall extraction coefficient based on the dispersed phase were determined in phase one for a range of drop diameters. This group of runs, Series A, is tabulated in Table XXIV. A normalizing technique as discussed later was applied in calculating the extraction coefficients. The fraction of extraction is plotted against drop diameter in Figure 7. In Figure 8 the drop velocity is plotted versus drop diameter. The overall extraction coefficients were computed from data taken off of Figures 7 and 8. The resulting coefficients are plotted as a function of drop diameter in Figure 9.

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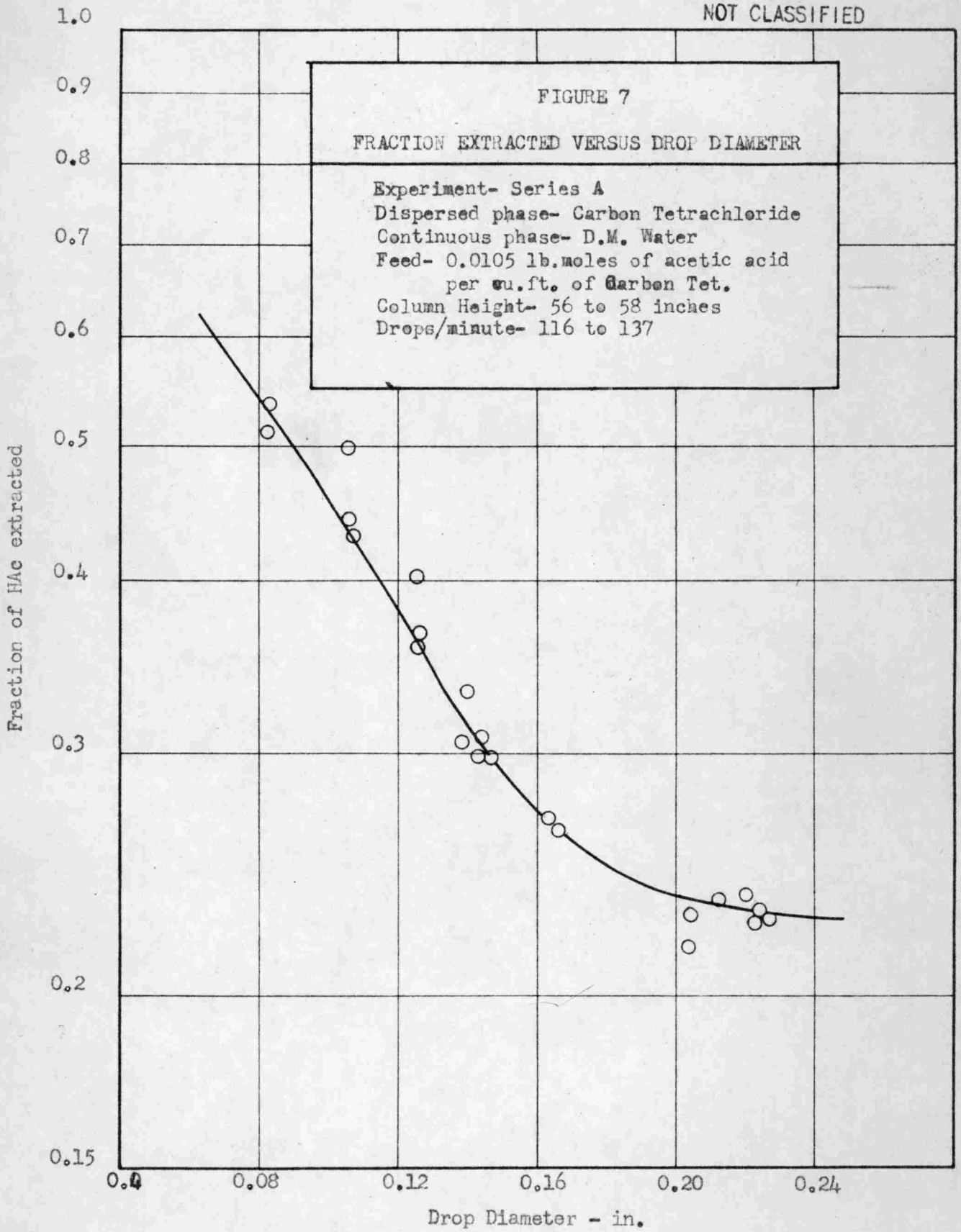

FIGURE 7

FRACTION EXTRACTED VERSUS DROP DIAMETER

Experiment- Series A
Dispersed phase- Carbon Tetrachloride
Continuous phase- D.M. Water
Feed- 0.0105 lb.moles of acetic acid per cu.ft. of Carbon Tet.
Column Height- 56 to 58 inches
Drops/minute- 116 to 137

DWG. 8501
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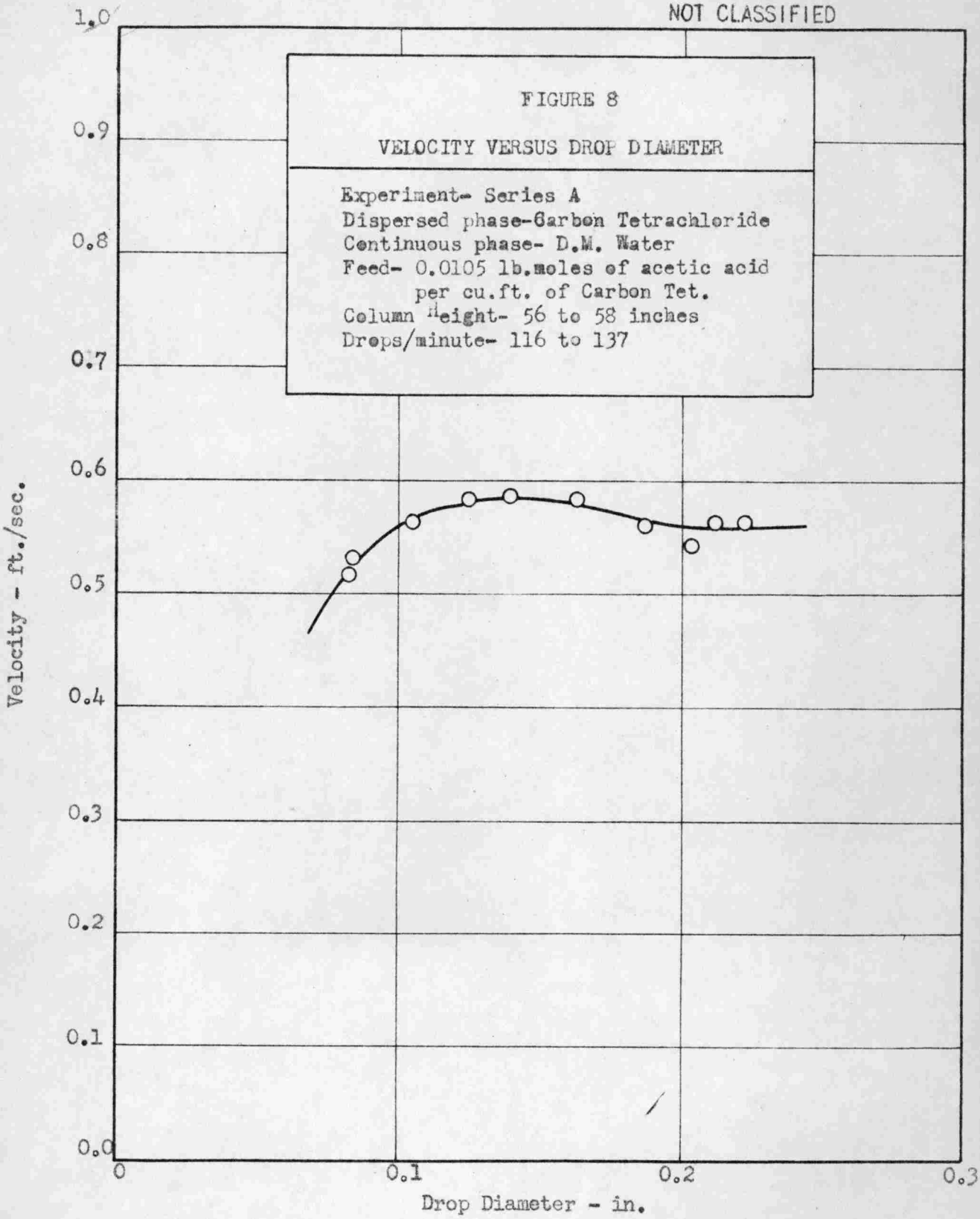

FIGURE 8

VELOCITY VERSUS DROP DIAMETER

Experiment- Series A
Dispersed phase-Carbon Tetrachloride
Continuous phase- D.M. Water
Feed- 0.0105 lb.moles of acetic acid per cu.ft. of Carbon Tet.
Column Height- 56 to 58 inches
Drops/minute- 116 to 137

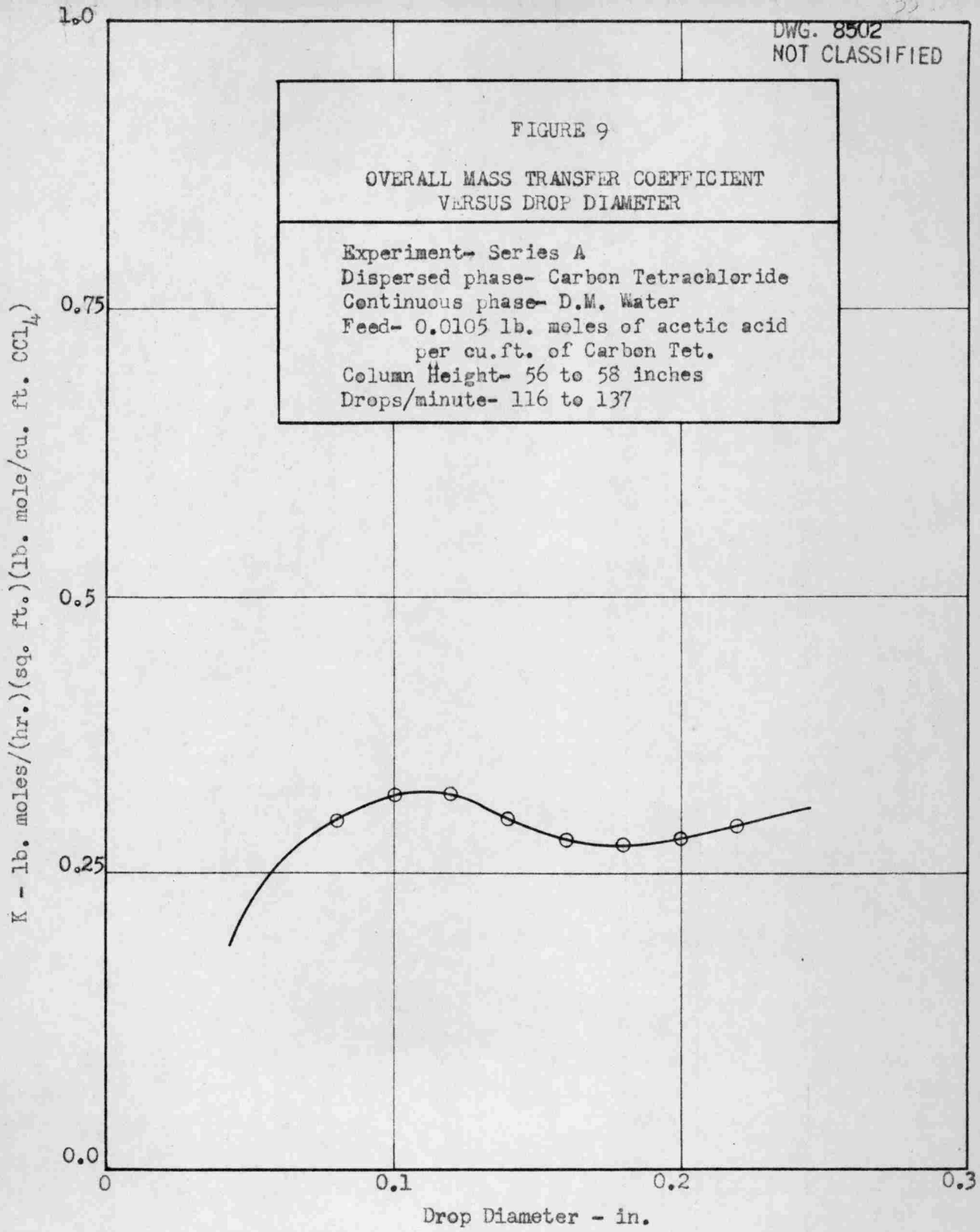
DWG. 8502
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FIGURE 9
OVERALL MASS TRANSFER COEFFICIENT
VERSUS DROP DIAMETER
Experiment- Series A
Dispersed phase- Carbon Tetrachloride
Continuous phase- D.M. Water
Feed- 0.0105 lb. moles of acetic acid
per cu.ft. of Carbon Tet.
Column Height- 56 to 58 inches
Drops/minute- 116 to 137
1.0
0.75
0.5
0.25
0.0
0
0.1
0.2
0.3
K - lb. moles/(hr.)(sq. ft.)(lb. mole/cu. ft. CCl_4)
Drop Diameter - in.

The room temperature, jacket water temperature and the rate of drop formation tended to vary slightly during this series of runs. Hence, in order to determine the significance of fluctuations in these conditions, runs were made in which only one of the above conditions was allowed to vary at a time. By measuring the rate of extraction it was possible to determine correction factors by which the experimental data could be normalized to the same temperature and drop rate.

Allowing only the drop flowrate to vary, the percent extraction was determined for a wide range of drop flowrates. The percent extraction is plotted as a function of the rate of drop formation in Figure 10 and as a function of the flow rate in Figure 11.

Similarly the dependence of the overall extraction coefficient on the temperature of the dispersed and continuous phases was determined allowing only one condition to vary at a time. The percent extraction is plotted versus dispersed phase temperature in Figure 12 and as a function of the continuous phase temperature in Figure 13. Because of the spread in the points in Figure 12 and 13 the data will give a straight line on semi-log and log-log as well as linear graph paper.

Various experimental techniques were employed to verify the hypothesis that the controlling mass transfer resistance lies in the dispersed phase. The introduction of a chemical reaction in one phase will frequently reduce the resistance to transfer of that phase. Hence, sodium hydroxide was added to the continuous phase and Series A runs were repeated using one selected drop diameter. The concentration of sodium

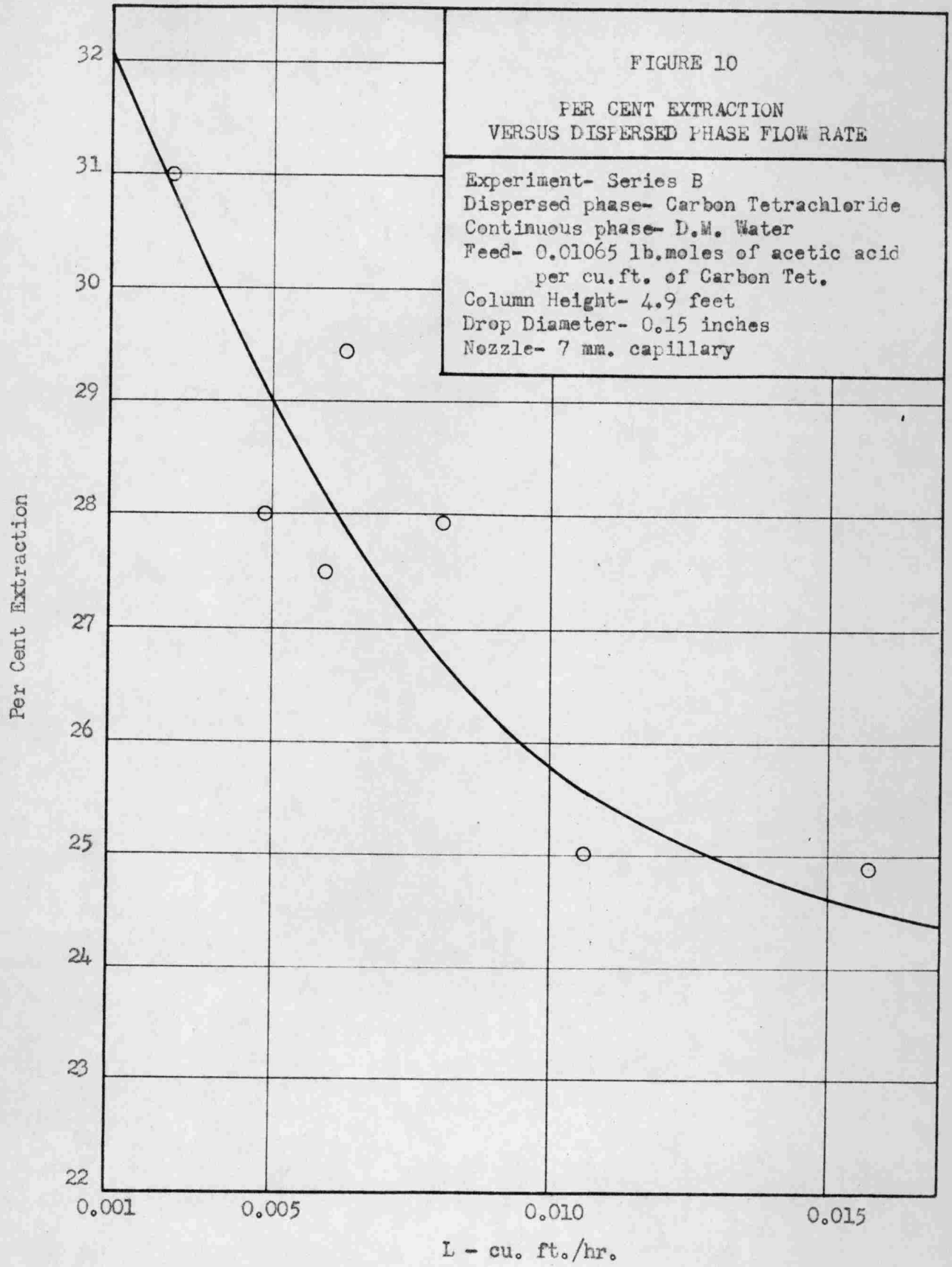
FIGURE 10
PER CENT EXTRACTION
VERSUS DISPERSED PHASE FLOW RATE
Experiment- Series B
Dispersed phase- Carbon Tetrachloride
Continuous phase- D.M. Water
Feed- 0.01065 lb.moles of acetic acid per cu.ft. of Carbon Tet.
Column Height- 4.9 feet
Drop Diameter- 0.15 inches
Nozzle- 7 mm. capillary
Per Cent Extraction
32
31
30
29
28
27
26
25
24
23
22
0.001
0.005
0.010
0.015
L - cu. ft./hr.

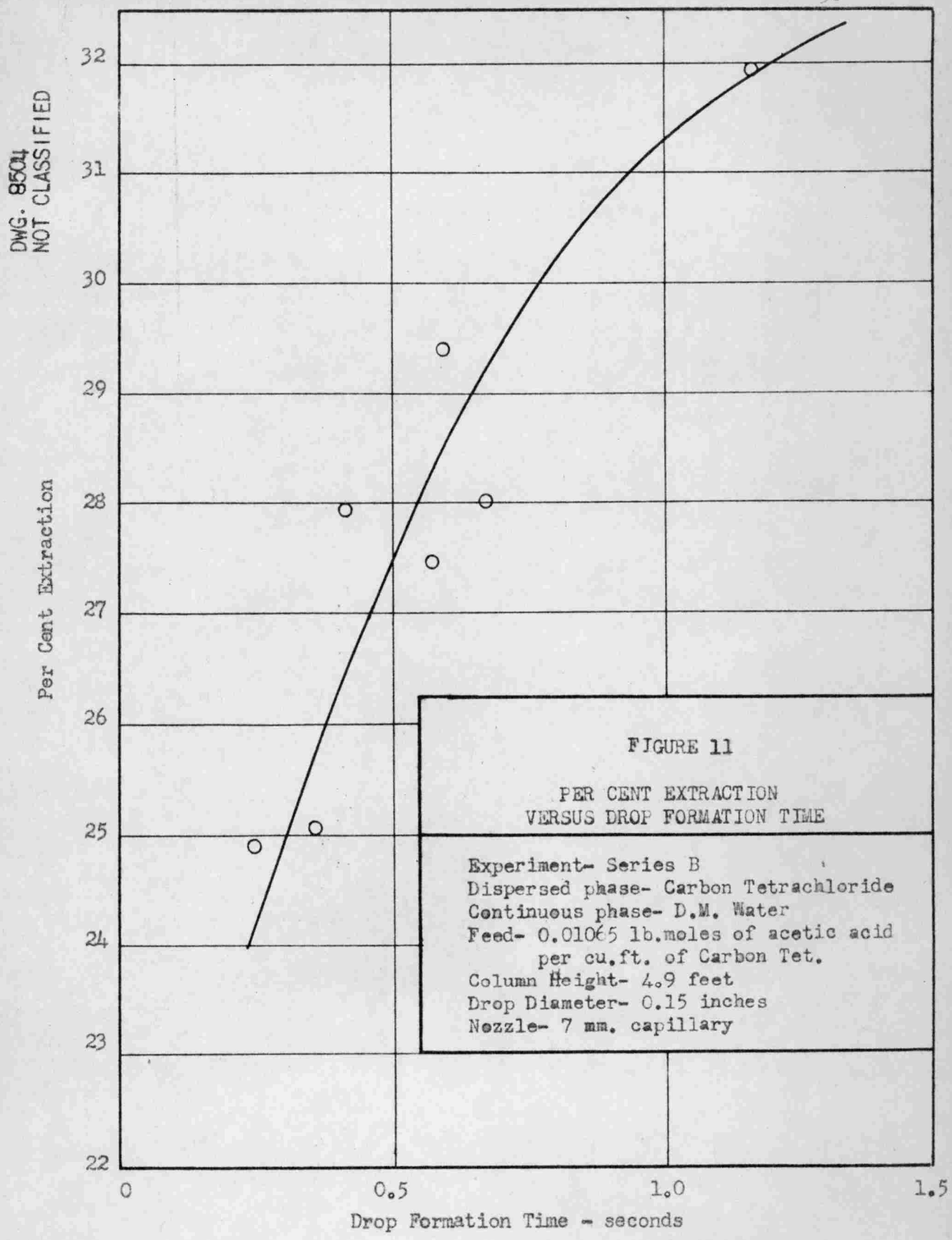

FIGURE 11

PER CENT EXTRACTION
VERSUS DROP FORMATION TIME

Experiment- Series B
Dispersed phase- Carbon Tetrachloride
Continuous phase- D.M. Water
Feed- 0.01065 lb.moles of acetic acid per cu.ft. of Carbon Tet.
Column Height- 4.9 feet
Drop Diameter- 0.15 inches
Nozzle- 7 mm. capillary

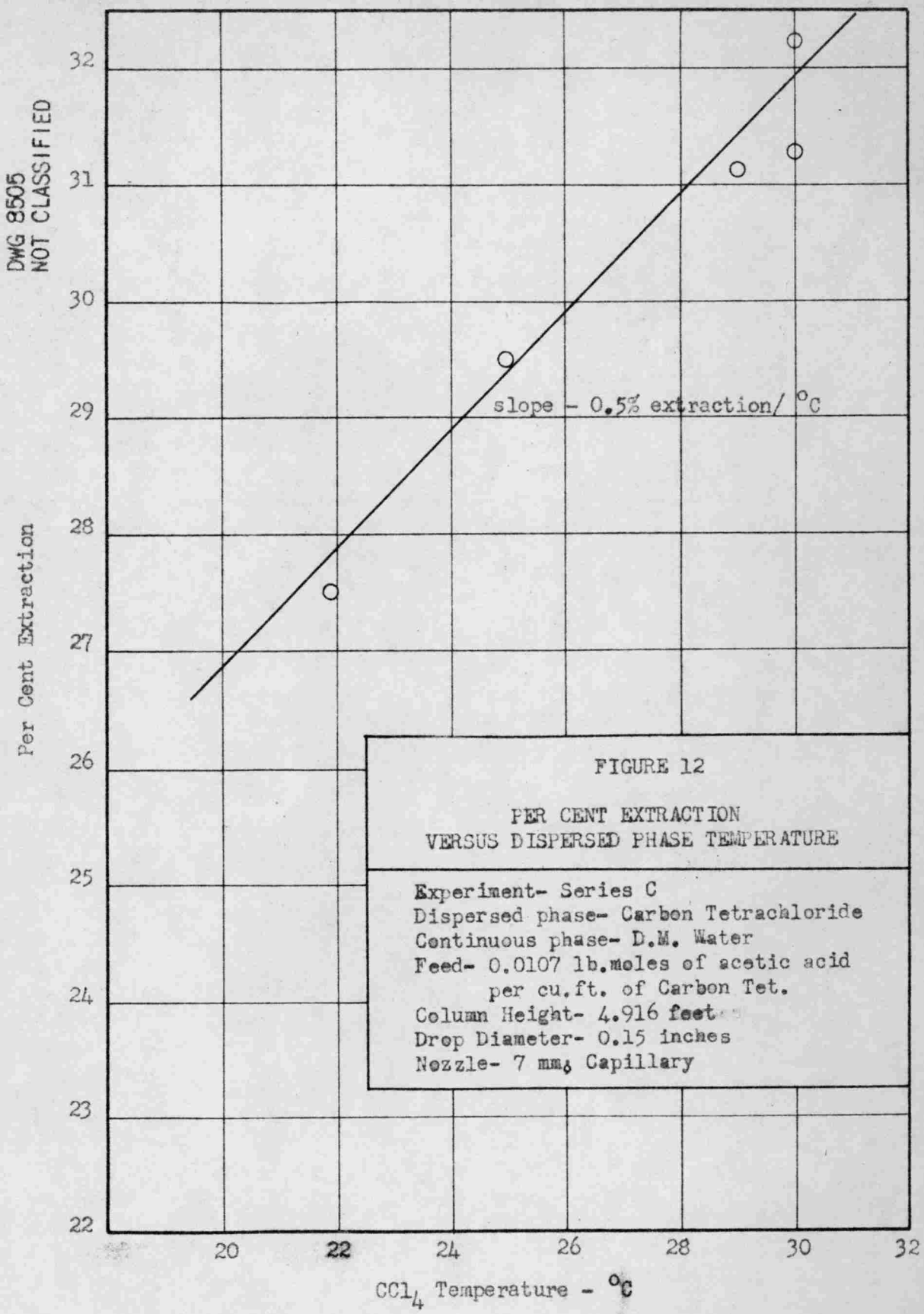

FIGURE 12

PER CENT EXTRACTION
VERSUS DISPERSED PHASE TEMPERATURE

Experiment- Series C
Dispersed phase- Carbon Tetrachloride
Continuous phase- D.M. Water
Feed- 0.0107 lb.moles of acetic acid per cu.ft. of Carbon Tet.
Column Height- 4.916 feet
Drop Diameter- 0.15 inches
Nozzle- 7 mm. Capillary

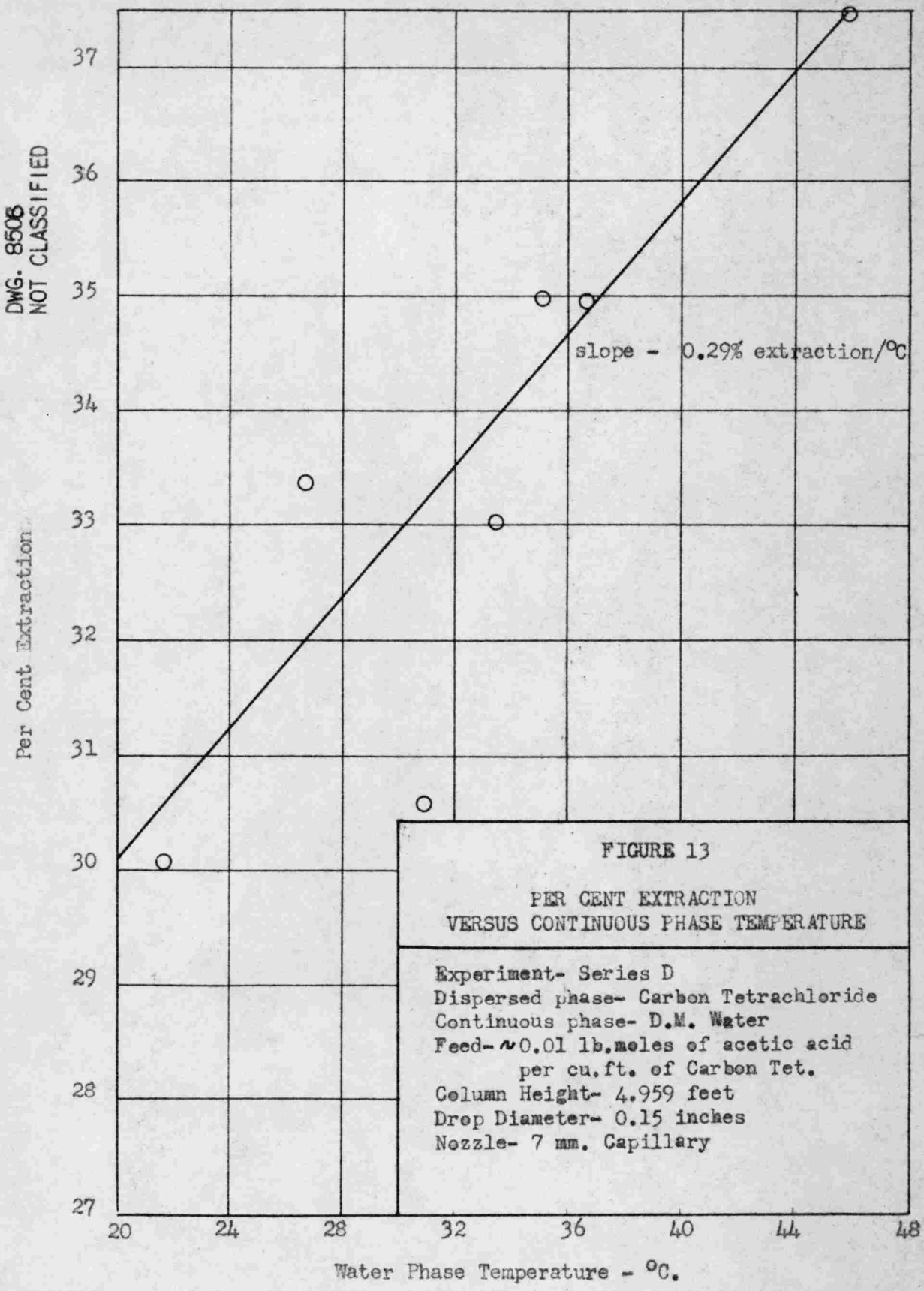

FIGURE 13

PER CENT EXTRACTION
VERSUS CONTINUOUS PHASE TEMPERATURE

Experiment- Series D
Dispersed phase- Carbon Tetrachloride
Continuous phase- D.M. Water
Feed- ~0.01 lb.moles of acetic acid per cu.ft. of Carbon Tet.
Column Height- 4.959 feet
Drop Diameter- 0.15 inches
Nozzle- 7 mm. Capillary

hydroxide was varied over a wide range to evaluate the possible affect of the concentration of the caustic on the percent extraction. The resulting overall extraction coefficients and percent extraction are given in Series H in Appendix G. The caustic had no effect upon the rate of extraction. This in part substantiates the hypothesis regarding the controlling resistance.

In order to further verify this hypothesis, an additional set of runs was made using a continuous water phase under dynamic or flow conditions. Using a specific drop diameter, drop rate temperature and column height, the velocity of the continuous was varied and the percent extraction determined. The Reynolds number relative to the tube diameter ranged from 0 to 61, while that relative to the drop diameter ranged from 630 to 690. It was impossible to get out of the viscous flow regime without destroying the single drop continuity. Hence, no conclusions could be reached.

In order to simplify the mathematical analysis of the data, systems were selected which had distribution ratios which varied only slightly with solute concentration. As a check, the concentration of acetic acid in the dispersed carbon tetrachloride was varied and the fraction of extraction determined for a specific drop diameter, drop rate, temperature and column height in Series F. Although the percent extraction and overall mass transfer coefficient both increased with decreasing solute feed concentration, the change was not due to a variation in distribution ratio and was in general insignificant.

The first chemical property to be varied in the first phase of investigation was the interfacial tension. By adding wetting agents (Tergitol #4 and #7) to the continuous phase in concentrations ranging from 0.01% by volume to 0.3%, it was possible to vary the interfacial tension for the water-carbon tetrachloride system from 33 to 0 dynes./cm. The same drop diameter, drop rate, temperature and column height were used in all runs. As the interfacial tension was lowered the drop size changed from 0.15 inches to 0.06 inches, while the change in the overall extraction coefficient was only that which would result due to drop size change.

This result indicates that interfacial tension does not affect the rate of extraction other than through its influence on drop size. However, this conclusion is in doubt because of the possible interface blocking which could result from the presence of the wetting agent. For this reason the use of additives in evaluating the influence of the chemical properties on the rate of extraction was abandoned. In order to determine further the influence of the chemical properties, results were obtained for other water-acetic acid-solvent systems. By investigating a number of systems sufficient results could be obtained to use the methods of determinants in mathematically analyzing the data. In order to correlate the data it is necessary to separate the fraction of extraction during free-fall of the dispersed drop from the fraction of extraction during drop formation. This was done by measuring the fraction of extraction as a function of column height. By graphical means the end-effects were eliminated.

In order to restrict the program to one which could be accomplished in a reasonable length of time the investigation of other solvent systems was made for a selected drop diameter of 0.15 inches and the extraction during free-fall separated from that due to drop formation for each solvent system. In all of these experiments the acetic acid was dissolved in the solvents and they in turn were dispersed in a continuous water phase.

The rate of extraction as a function of column height was determined at a drop diameter of 0.15 inches for dispersing the solvents methyl isobutyl ketone, benzene, toluene, chloroform, carbon tetrachloride and isopropyl ether. As is pointed out earlier the plot ln (C_f/C_r) versus column height should be a straight line if K is independent of acetic acid concentration. In Figure 14 $\ln(C_f/C_r)$ is plotted versus column height for the above solvents. By extrapolation to zero column height the amount of extraction occurring during drop formation is determined.

The overall extraction coefficient (including end-effects) was determined as a function of drop diameter for these same solvents systems. The results are plotted in Figure 15.

To determine whether the rate of drop formation influenced the free-fall result, a series of experiments (Series X) were run using a very short column height. The drop formation time only was varied and the fraction of extraction determined. The computed results are plotted as percent extraction versus drop formation time in Figure 16 along with Series B

FIGURE 14

RECIPROCAL FRACTION UNEXTRACTED VERSUS COLUMN HEIGHT

Curve	Series	Feed Conc. lb. moles/cu.ft.	Drops/min.	Drop Dia. in.	Solvent dispersed
⊗	E	0.01	110-150	0.150-0.164	CCl_4
○	L	0.01	120-150	0.138-0.149	M.I.K.
◎	P	0.01	111-125	0.152-0.158	Benzene
□	R	0.01	117-124	0.147-0.157	Toluene
△	V	0.01	120-136	0.140-0.142	$ChCl_3$

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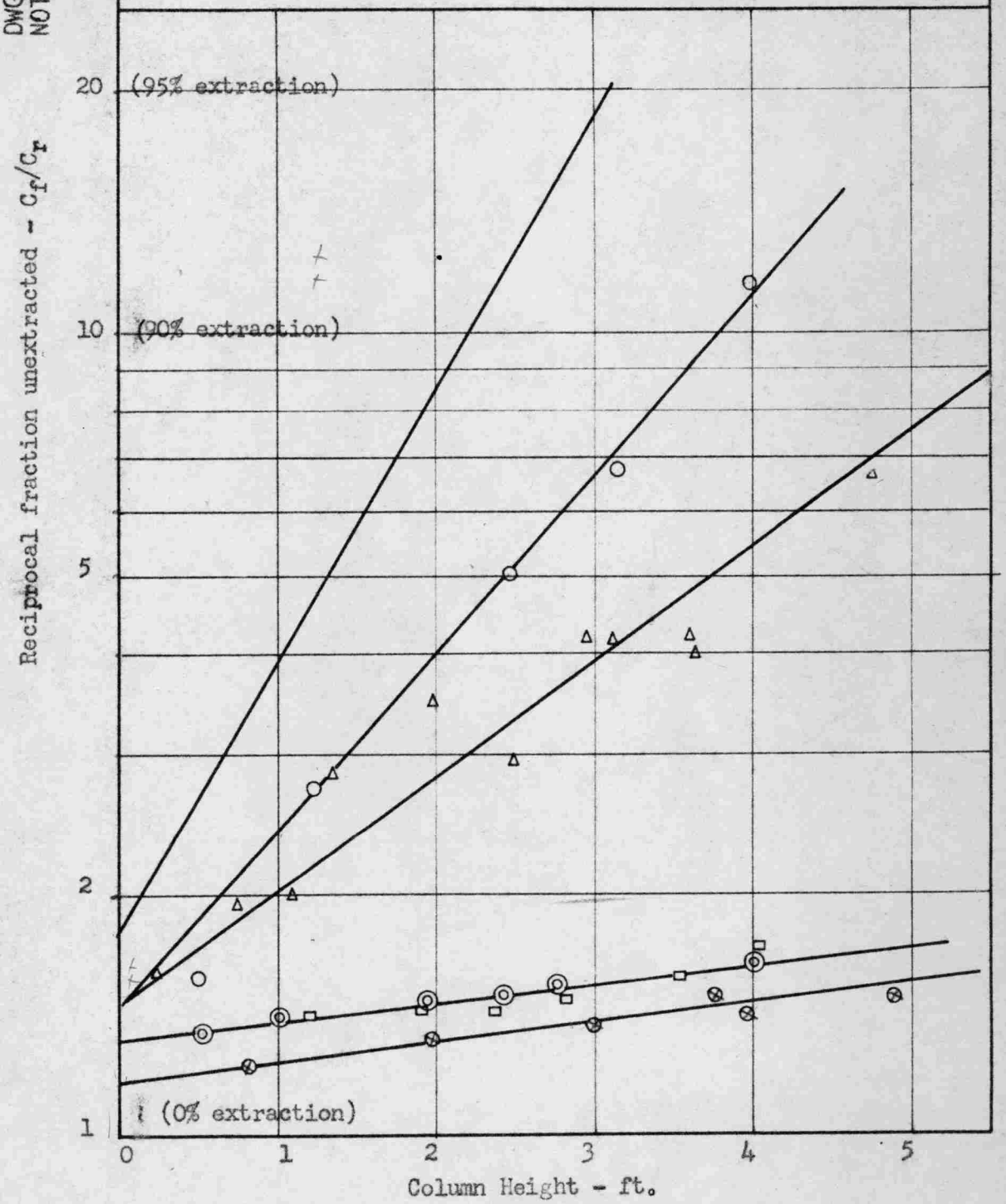


FIGURE 15

OVERALL MASS TRANSFER COEFFICIENTS VERSUS DROP DIAMETER

Curve	Series	Feed Conc. Lb. moles/cu.ft.	Drops/min.	Col. Ht. ft.	Solvent Dispersed
○	A	0.01	116-137	4.82	CCl_4
○	k	0.01	100-137	4.08	M.I.K.
⊙	O	0.01	104-127	4.0	Benzene
⊗	M	0.01	126-136	4.0	H_2O (HAc)
◎	N	0.01	130-153	4.0	H_2O
□	Q	0.01	102-143	4.0	Toluene
△	U	0.01	114-136	4.82	$ChCl_3$

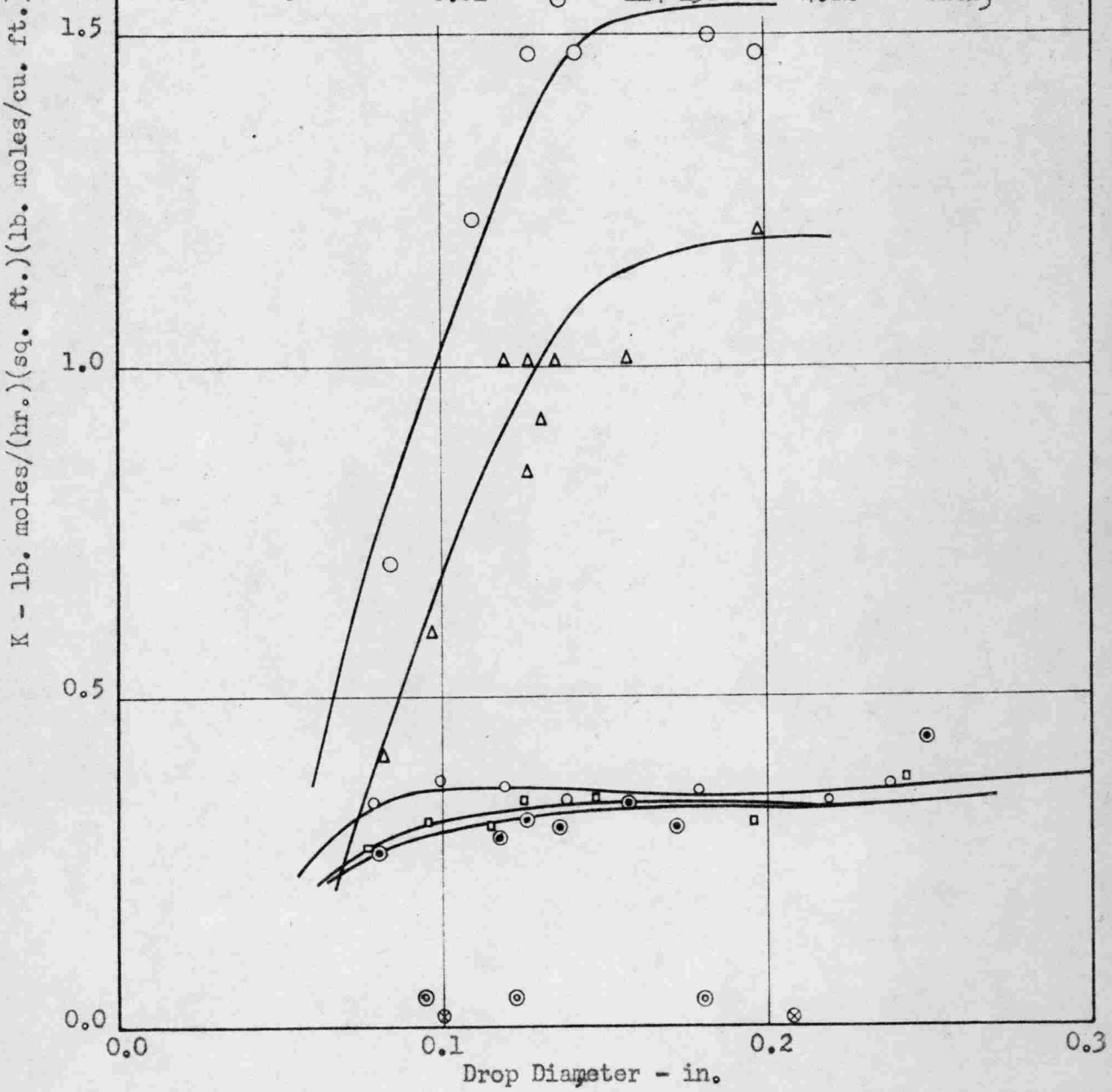
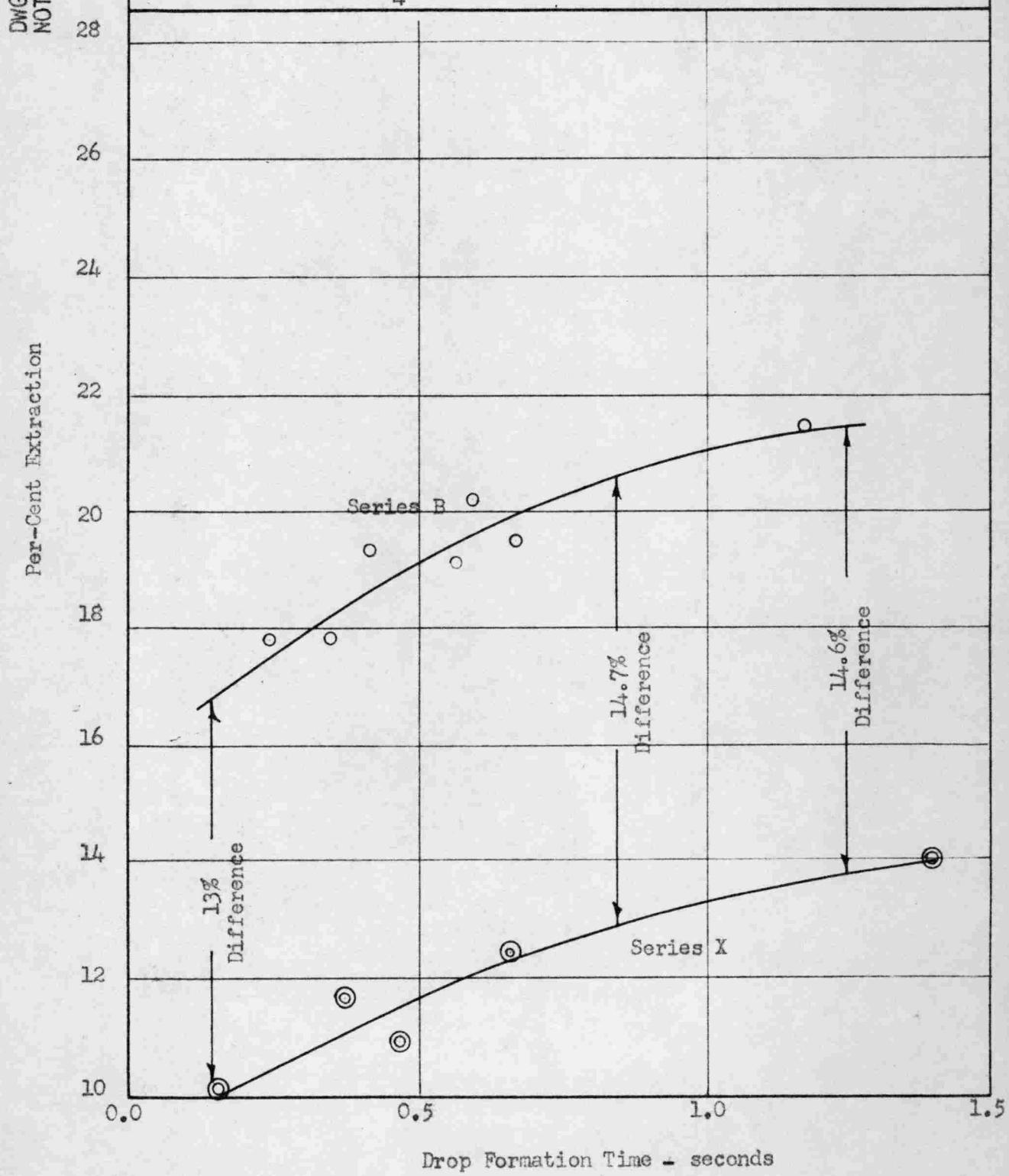


FIGURE 16

PER CENT EXTRACTION VERSUS DROP RATE

Experiment	Disp. Phase	Feed Conc. Lb.moles/cu.ft.	Col.Ht. ft.	Drop Dia. in.
Series B	CCl_4	0.01065	4.9	0.15
Series X	CCl_4	0.01037	0.43	0.14



in which drop formation time was varied using a long column. The difference between these two curves represents the percent extraction during free-fall. From Figure 16 it is apparent the free-fall extraction is independent of drop formation time.

The rate of extraction when the direction of transfer is reversed was measured in Series I for the basic water-acetic acid-carbon tetrachloride system. Acetic acid was dissolved in the continuous water phase and the rate of transfer into the dispersed phase determined for a range of drop diameters holding the remaining variables fixed. The rate of extraction reached equilibrium at some undetermined column height; hence, this series of runs was repeated using a shorter column in Series YB. The extraction coefficient in the reverse direction was also measured as a function of column height for a fixed drop diameter of 0.15 inches in order to obtain the free-fall result.

Finally, in order to determine if possible, the effect of the chemical properties of water on the rate of extraction, the relative phases in the carbon tetrachloride-acetic acid-water system were reversed. In the Series N experiments water was dispersed and transfer of acetic acid took place from the continuous carbon tetrachloride phase to the water. In series M the direction of transfer was reversed and water and acetic acid were used as a dispersed feed solution. In both of these latter experiments the rate of extraction was much lower than expected.

Errors

Errors may have been introduced into the results either by inaccuracies in physical measurements or by inaccurate chemical analyses. In some cases the nature of the liquids involved were such that accurate measurements could not be made.

Velocity, flowrate and interfacial area information is dependent upon measurements of time and volume. In clocking the overall experiment time and in measuring the product volume, the error was less than 1%. The accuracy with which the time of fall or contact was determined was about 95%. The largest time error was the measurement of the drop formation time. This error was from 5 to 10% due to fluctuations in the drop formation time rather than chronographic faults.

The analytical errors for most determinations were less than 5%. The thymol blue end-point in the case of some titrations was difficult to determine.

The major sources of error were in operating techniques and chemical behavior. For example: isopropyl ether showed a strong tendency to wet glass and as a result many of the drops did not pass up the column but filmed out on the walls. The error so introduced may have been as large as 30%.

The final accuracy of the overall extraction coefficient is probably 75% because of the accumulative nature of the above errors. This is a reasonable result, however, in view of the many complications in interpreting the observations.

CHAPTER IX

CORRELATIONS AND DISCUSSION

From the tabular and graphical results in Chapter VIII it was possible to determine the controlling resistances in a falling drop liquid liquid extraction column. The dependence of the extraction rate upon the variables of the system was also evaluated.

In the mathematical analysis of the experimental data it is necessary to evaluate the relative magnitudes of the resistance to transfer of both the continuous and dispersed phases. For these experiments, the hypothesis is made that the controlling resistance is in the dispersed phase. This is based on the following suppositions and observations:

1. As the falling drop descends through the tower it comes into contact with a new continuous phase surface. This prevents a concentration gradient from forming in the continuous phase adjacent to the drop.

2. The distribution ratio (K) of the water-acetic acid-solvent systems are all greater than 1 and in many cases are greater than 20. Hence, the magnitude of the resistance $1/(k_c K)$ is much smaller than $1/k_d$. Therefore: $K_o = k_d$.

3. In the Series H experiments the addition of sodium hydroxide resulted in no increase in the rate of extraction.

4. In the Series G experiments, changing the continuous phase from a stagnant to a moving liquid did not increase the rate of extraction. The results of this series of runs are however not too significant since the flow of the continuous phase was in the viscous region.

When the experimental program was initiated it was felt the interfacial tension would play an important role in determining the rate of extraction. The experimental results did not sustain this belief.

1. The overall extraction coefficients obtained were not proportional to the interfacial tension.

2. In the Series J runs the interfacial tension was varied over wide limits without producing any change in the rate of extraction other than that which would be expected because of changes in drop diameter. The results are however not conclusive since the wetting agent used to lower the interfacial tension does so by accumulation at the interface and therefore may block the passage of acetic acid molecules and counteract the effect of lowered interfacial tension.

The experimental results indicated that more extraction takes place during drop formation or as end-effects than would be expected as a result of pure diffusion. It is therefore necessary to eliminate the end-effects and determine the rate of extraction when the interfacial area is in static condition as during free-fall of the drop. Fortunately it is possible to separate the rate of extraction during free-fall by graphical means using Figure 14.

Using the fraction unextracted during free-fall and the dimensions of the drop it is possible to compute overall extraction coefficients for free-fall conditions. The data can also be interpreted in terms of eddy diffusion coefficients D_e which are computed from equations developed in appendix E. By comparing the eddy diffusion coefficients (D_e) with the molecular diffusion coefficients (D_i) the mechanism of extraction is partially revealed.

A comparison of the relative chemical properties of the different dispersed solvents is shown in Table I. In Table II the results of the physical properties are compared to the eddy diffusion coefficient and to the overall extraction coefficient obtained by methods shown in

appendix E and F. As was stated before, the data are expressed for convenience on the basis of the key variable, drop diameter, rather than the Reynolds number.

In order to evaluate the relative importance of each of the chemical and physical variables on the rate of extraction, the mathematical methods of determinant analysis were applied to the data in Tables I and II. The results of this analysis are given in Appendix F. In order to obtain a correlation which would fit the six solvents it was necessary to introduce the variables - drop diameter, viscosity, density, velocity, interfacial tension and molecular diffusivity. The resulting correlation could not however be broken down into dimensionless groups.

It was apparent from the analysis of the data of Series YB and Series A, that either, the basic hypothesis regarding the controlling resistance was in error, or else there were unknown chemical affects controlling the rate of extraction. The overall extraction coefficient for free-fall conditions in Series YB was 1.6 ft./hr. at a drop diameter of 0.15 inches. This is approximately 6 times the result of 0.25 ft./hr. obtained for a drop diameter of 0.15 inches in Series A. The only difference between the two series of runs is that in YB the direction of transfer is from the continuous phase to the dispersed (carbon tetrachloride), while in A it is from the dispersed phase (carbon tetrachloride) to the continuous water phase. If the controlling resistance is in the dispersed phase then the rate of transfer should be the same in both directions.

TABLE I

CHEMICAL PROPERTIES OF THE DISPERSED SOLVENTS

	Viscosity-μ c.p.	Density-ρ grs./cc	Kimematic Viscosity-ν x 10^2 cm.^{2} / sec.	Interfacial Tension* dynes/cm.
Chloroform	0.57	1.473	0.387	25
Methyl-Isobutyl Ketone	0.559	0.796	0.703	8
Benzene	0.61	0.873	0.699	22
Toluene	0.561	0.860	0.653	23
Carbon Tetrachloride	0.941	1.586	0.592	28
Isopropyl Ether	0.349	0.721	0.484	15

Acetic acid concentration

* 0.01 lb.moles/cu.ft. of solvent.

TABLE II

COMPARISON OF RESULTS FOR THE SAME DROP DIAMETER
FREE OF END-EFFECTS

	Drop Diameter-d Inches	Velocity-V ft./sec.	$D_i^{*} \times 10^6$ In.^{2}/ sec.	$D_e^{**} \times 10^6$ In.^{2}/ sec.	K_o freefall ft./hr.
Chloroform	0.141	0.54	3.37	56.5	1.265
Methyl Isobutyl Ketone	0.140	0.37	3.424	69.7	1.32
Benzene	0.155	0.32	3.472	1.92	0.142
Toluene	0.153	0.33	3.78	1.885	0.1445
Carbon Tetrachloride	0.15	0.58	2.04	3.67	0.248
Isopropyl Ether	0.144	0.45	5.48	590	2.60

* Computed from the data of Wilke.

** Based on diffusion in spherical coordinates.

The difference in the rate of transfer in the above can only be due to a property such as interfacial tension or to chemical affects. The interfacial tension does not appear to control the rate of transfer. Hence, this resistance is probably a chemical affect. This can be further substantiated by examining Figures 14 and 15. In Figure 15 the curve representing the change of K_o with drop diameter has the same general shape for each dispersed solvent. Hence, the interference to transfer in the carbon tetrachloride must be independent of both drop diameter and velocity. This automatically rules out all affects but chemical.

It is observed in Figure 15 that the lowest rates of transfer are for non-polar organic solvents. The main chemical affect peculiar to these solvents is that of association. A comparison was therefore made in Table III between the solution properties of the various solvents and water. One trend is apparent from this tabulation. That is, the overall extraction coefficient increases as the degree of association decreases. The number of single unassociated molecules can be computed from the degree of association by the expression $n/(2-n)$ where n is the degree of association. In carbon tetrachloride there is only 1 single acetic acid molecule for every two double molecules when the acetic acid concentration is 0.01 lb. moles/cu.ft.. Water has a strong tendency to dissociate acetic acid at low concentrations and only the single molecules are compatible with it. Thus when (acetic acid) is being transferred from carbon tetrachloride to water, the

TABLE III

SOLVENT - ACETIC ACID-WATER SOLUTION PROPERTIES

	Solubility grs./100 cc H_2O	$K = \frac{C_w}{C_s}$*	Degree of Association n*
Water		75	1.00
Chloroform	0.621	6	1.58
Methyl Isobutyl Ketone	2.0	2.09	1.0
Benzene	0.073	18.5	1.79
Toluene	0.047	20	1.71
Carbon Tetrachloride	0.08	19.2	1.67
Isopropyl Ether	0.90	4.76	1.00
Carbon Tetrachloride**	0.08	3.2	1.56

* Acetic acid concentration 0.01 lb.moles/cu.ft. of solvent.

** Propionic acid concentration 0.01 lb.moles/cu.ft.

single molecules only are able to diffuse into water and the double molecules build up in the film. Acetic acid exists in water at a concentration of 0.01 lb. moles/cu.ft. entirely as single molecules. Therefore, they all are involved with the transfer of acetic acid from the water to the dispersed carbon tetrachloride.

These results do not nulify the hypothesis that the major resistance is in the dispersed phase. However, there are essentially two parallel paths of diffusion. The first is that of the single molecules while the second is that of the double molecules. The lower molecular diffusivity of the double molecules together with the time required for the dissociation reaction make the second or double molecule transfer path subordinate to that of the single molecules. As a result, the rate of extraction can be based on the concentration of single molecules in computing overall mass transfer coefficients or else the coefficient corrected by a factor.

In order to evaluate the affects of the distribution ratio (K), the carbon tetrachloride experiments in Series A were repeated in Series Z, substituting propionic acid for acetic acid. Propionic acid like acetic acid is a carboxylic acid and therefore has similar properties. The distribution ratio of propionic acid is over 5 times smaller than that of acetic acid in the water-carbon tetrachloride system, hence, there should be a substantial change in the rate of extraction if the distribution ratio is an important variable. The results using propionic acid were almost identical to those using acetic acid. The slight increase in overall extraction coefficient

from 0.25 ft./hr. for acetic acid to 0.35 ft./hr. for propionic acid, under identical conditions, is due most likely to the lower degree of association of propionic acid and not to the distribution ratio.

In order to analyze the results and break down the measured extraction rates in terms of the variables, the hydrodynamics was carefully investigated. A comparison was made between the observed velocity versus diameter pattern and that predicted from known hydrodynamic relations for spheres. Due to the deformation of the liquid drops as observed through the glass column and measured in the drop deformation study, the results deviated from the predicted values in the turbulent flow regime. The break between the transitional flow region and the turbulent flow regime occurred at a Reynolds number of 300 to 350. This is equivalent to a drop diameter of 0.08 - 0.10 inches which is very near the lower limit of the experimental investigation.

Relations were developed for correlating the rate of extraction under the turbulent flow regime and the transitional regime based on the hypothesis that all resistance to transfer is in the dispersed phase. These developments are outlined in Appendix E and F. In the turbulent flow regime the concept of a fictive film is introduced into the correlation. This film can be related to the properties of the dispersed solvent. By dimensional analysis the overall extraction coefficient can be expressed as:

$$\frac{K_o d}{D_i} = \phi \left(\frac{dV \rho_d}{\mu_d}\right)^a \left(\frac{\mu_d}{\rho_d D_i}\right)^b \left(\frac{dV^2 \rho_c}{I.T.}\right)^c$$

In the transitional regime of flow the properties of the solvents are of less importance and the rate of transfer is dependent upon the rate of molecular diffusion. From the diffusion equation for the unsteady state, an expression can be developed for determining the fraction unextracted. Since the drops behave as perfect spheres in this regime the diffusion expression can be solved in spherical coordinates to obtain:

$$K_o = \frac{L}{A} \ln \frac{\pi^2}{6} \frac{1}{\sum_{n=1}^{\infty} \frac{1}{n^2} e^{-\left(\frac{n\pi}{r_o}\right)^2 D_i \theta}}$$

To evaluate the validity of these concepts, the data obtained for the dispersion of carbon tetrachloride as drops in a continuous water phase was carefully broken down. The results of Series A and W were plotted as fraction extracted versus drop diameter in Figure 17. By graphical interpolation it is possible to derive the curve for free-fall conditions and eliminate the end effects. From these data the reciprocal fraction unextracted were computed and compared with that predicted by the law of diffusion for spheres. The results are plotted in Figure 18 as a function of drop diameter. The overall extraction coefficient was then computed and a comparison made in Figure 19 between the overall extraction coefficient including end-effects, the

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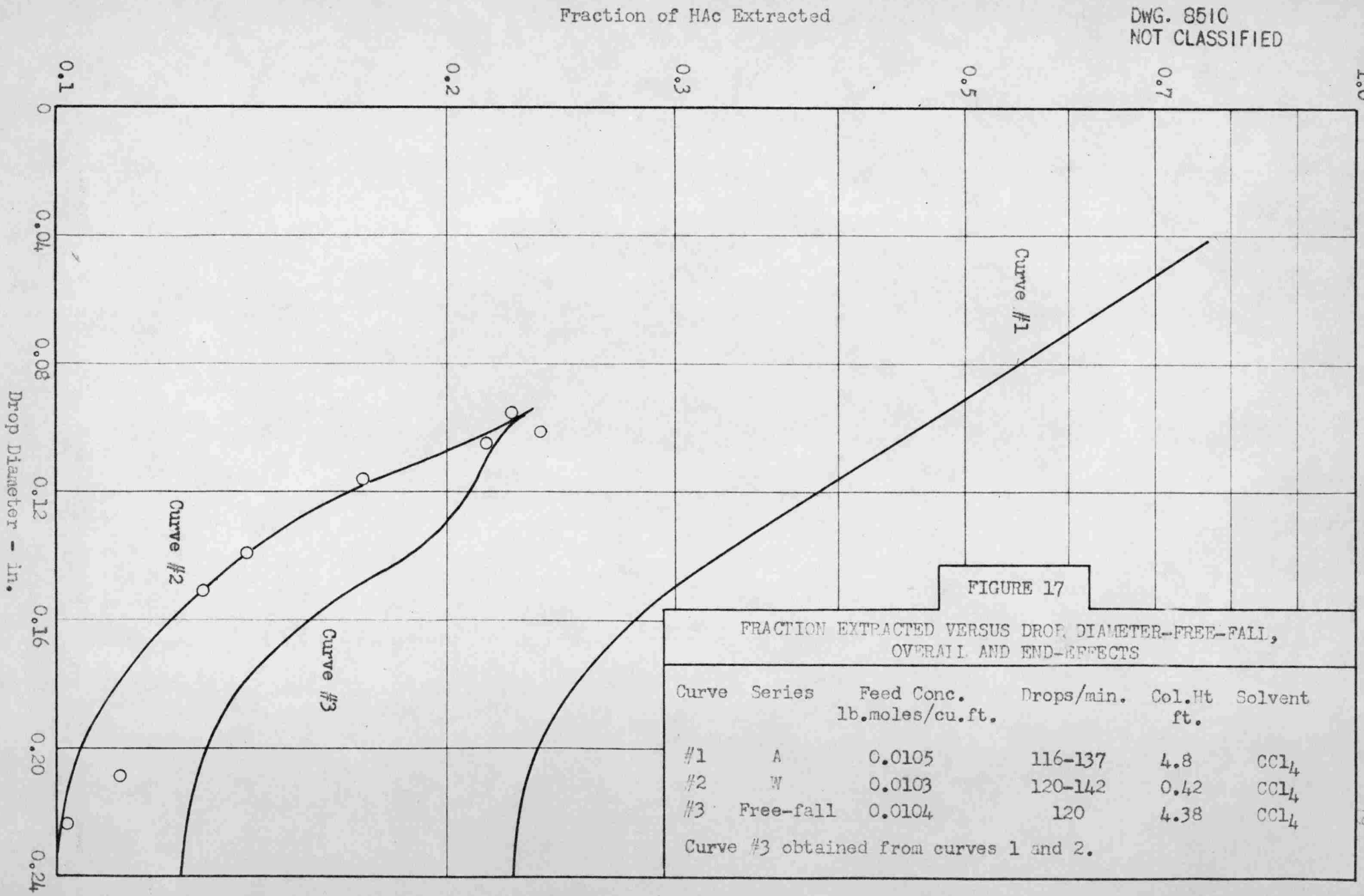

FIGURE 17

FRACTION EXTRACTED VERSUS DROP DIAMETER-FREE-FALL, OVERALL AND END-EFFECTS

Curve	Series	Feed Conc. lb.moles/cu.ft.	Drops/min.	Col.Ht ft.	Solvent
#1	A	0.0105	116-137	4.8	CCl_4
#2	W	0.0103	120-142	0.42	CCl_4
#3	Free-fall	0.0104	120	4.38	CCl_4

Curve #3 obtained from curves 1 and 2.

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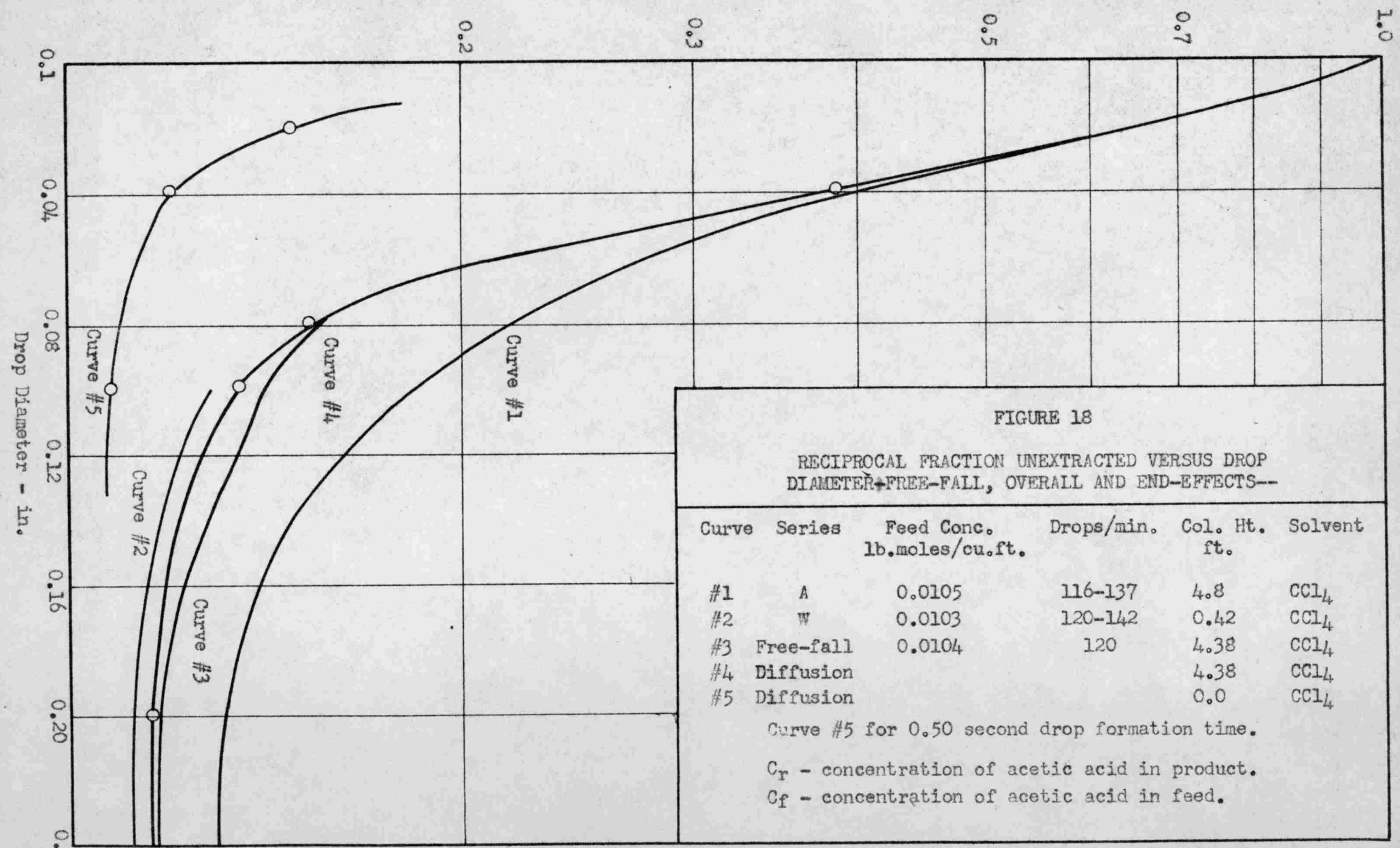

FIGURE 18

RECIPROCAL FRACTION UNEXTRACTED VERSUS DROP DIAMETER-FREE-FALL, OVERALL AND END-EFFECTS--

Curve	Series	Feed Conc. lb.moles/cu.ft.	Drops/min.	Col. Ht. ft.	Solvent
#1	A	0.0105	116-137	4.8	CCl_4
#2	W	0.0103	120-142	0.42	CCl_4
#3	Free-fall	0.0104	120	4.38	CCl_4
#4	Diffusion			4.38	CCl_4
#5	Diffusion			0.0	CCl_4

Curve #5 for 0.50 second drop formation time.

C_r - concentration of acetic acid in product.

C_f - concentration of acetic acid in feed.

FIGURE 19

MASS TRANSFER COEFFICIENT VERSUS DROP DIAMETER-FREE-FALL, OVERALL AND END-EFFECTS-

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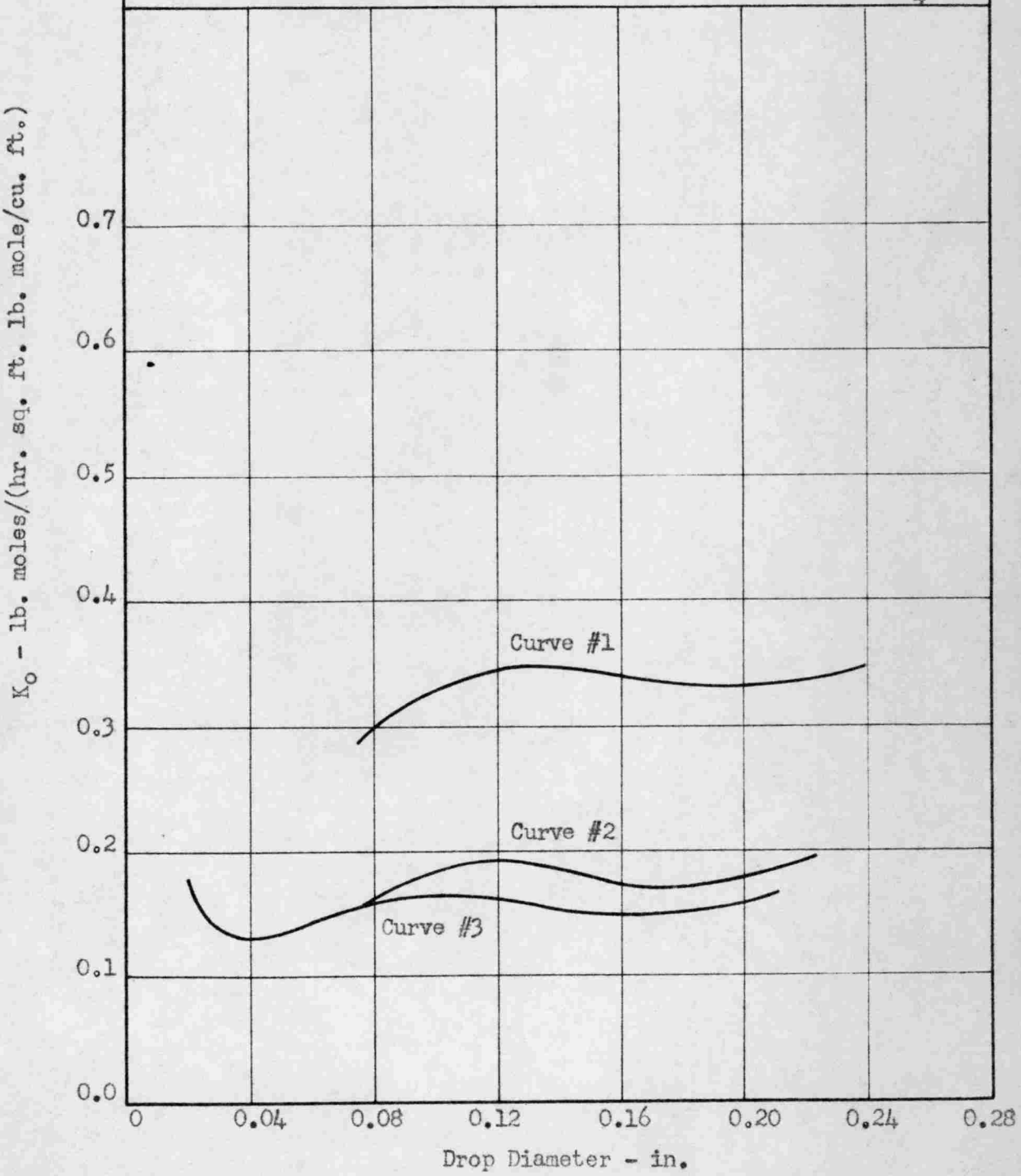
Curve	Series	Feed Conc. lb.moles/cu.ft.	Drops/min.	Col. Ht. ft.	Solvent
#1	A	0.0105	116-137	4.8	CCl_4
#2	Free-fall	0.0104	120	4.38	CCl_4
#3	Diffusion			4.38	CCl_4



free-fall extraction coefficient and that which would be predicted from the law of diffusion.

As would be expected, the rate of extraction is greater in the turbulent flow regime than that predicted for true molecular diffusion. This indicates a decrease in resistance which results probably from turbulence within the drop. Below a Reynolds number of 300 to 350, where the flow changes from turbulent to transitional, the overall extraction coefficient decreases and approaches that predicted for unsteady state molecular diffusion in spheres.

This same trend in the overall extraction coefficient is exhibited by all the systems, as shown in Figure 15, below a Reynolds number of 300 - 350.

In order to determine whether a viscous vortex is causing the increased transfer in the turbulent regime, the reciprocal fraction unextracted obtained experimentally was compared with that predicted for a viscous doughnut shaped vortex within the drop. The methods of analysis are outlined in Appendix E and a comparison of the results for the free-fall condition between the various solvents at a diameter of 0.15 inches is shown in Table IV. It is apparent from this tabulation that the rate of extraction in the turbulent flow regime is much greater than can be accounted for by a viscous vortex.

TABLE IV

COMPARISON OF EXPERIMENTAL RESULTS WITH THOSE PREDICTED BY THE LAWS OF DIFFUSION

	Experimental		Sphere	Cylinder
	C_r / C_f	$\ln(C_f/C_r)$	C_r/C_f	C_r/C_f
Chloroform	0.268	1.324	0.78	0.705
Methyl Isobutyl Ketone	0.132	2.027	0.73	0.64
Benzene	0.798	0.2272	0.73	0.645
Toluene	0.798	0.2272	0.715	0.605
Carbon Tetrachloride	0.797	0.2271	0.84	0.775
Isopropyl Ether	0.04	3.2		

In order to derive a relationship between the film coefficient and the properties of the solvents for the turbulent flow regime, it is necessary to discard our previous evaluation based on determinants and approach the problem through dimensional analysis and through analogies to momentum and heat transfer. Using the dimensional analysis solution presented in Appendix F, a comparison was made between the dimensionless groups for each of the solvents at a drop diameter of 0.15 inches. In computing the Nusselt equivalent number (Kd/D_i), the experimentally measured values of K_o were used. These were corrected later for concentration based on the concept of single molecule transfer in non-polar organic liquids.

To complete the correlation it is necessary to evaluate the coefficient of proportionality and the powers of our dimensionless groups. The power of the Schmidt number $\frac{\nu}{D_i}$ can be determined by comparing the results for isopropyl ether with those for carbon tetrachloride in which the direction of transfer is from the water to the dispersed carbon tetrachloride. The properties of isopropyl ether and carbon tetrachloride yield the same value for both the Reynolds number ($dV\rho/\mu$) and Weber Capillary group ($dV^2\rho/I.T.$) when evaluated for the same experimental conditions. Therefore, the power of the Schmidt number can be evaluated by comparing the Schmidt (ν/D_i) and Nusselt Equivalent (Kd/D_i) numbers for the two solvents. This power is then found to be 0.44.

By comparing the expression derived by dimensional analysis above to the analogous expression for turbulent flow in pipes and to the expression for mass transfer in fictive films, the Reynolds number can be assigned an exponent of 0.8. The exponent of the Weber number can then be determined by evaluating the powers to which d or V must appear to correspond to the experimental results.

From Figure 19 it is apparent the shape of the curve K_o for free-fall is similar to that for K_o overall (including end-effects). Therefore, the curves presented in Figure 15 can be used to determine the dependence of K_o on drop diameter. As the drop diameter changes certainly neither the chemical properties nor the degree of association changes. However, the velocity does vary. The shape of all the curves in Figure 15 are expressed in the turbulent flow regime, by the function $d^{0.6}\, V^{2.4} = (dV^2)^{0.8}(dV)^{0.8}\, \frac{1}{d}$. Thus if the exponent of the Reynolds number is 0.8 the Weber capillary group must have an exponent of 0.8 also, in order to fit the experimental result. By assuming the highest accuracy for the data from the experiments in which transfer is from the continuous water phase to the dispersed carbon tetrachloride the proportionality constant is evaluated as 0.03. The equation correlating extraction for the turbulent flow regime is therefore given by:

$$\frac{K_o d}{D_i} = 0.03 \left(\frac{dV\rho_d}{\mu_d}\right)^{0.8} \left(\frac{\mu_d}{\rho\, D_i}\right)^{0.44} \left(\frac{dV^2 \rho_c}{I.T.}\right)^{0.8}$$

In order to determine the accuracy of this expression, the Nusselt equivalent number was computed for all the experimental solvent systems using the above equation. The results are compared with the experimental values in Table VB. In addition a comparison is made between the computed results and the experimental values obtained when the concentration is based on only the single unassociated molecules in non-polar organic liquids.

The equation for the overall extraction coefficient developed above from experimental data fits the data reasonably well considering the number of corrections and interpolations that have been made. The hypothesis, that only single molecules are accepted by the water when transfer is from a dispersed non-polar organic liquid, appears to be fairly valid from the comparison in Table VB. Deviations from this hypothesis in the case of non-polar liquids may be a result of the first order reaction of double molecule dissociation in non-polar organic liquid dispersed phase fictive films. Since the curves in Figure 14 are straight lines, a reaction of dissociation if governing must be a first order reaction.

The principal deviation from the experimental correlation occurs when the water is dispersed and the transfer of acetic acid takes place from water to the continuous organic liquid phase as in Series M runs. The overall extraction coefficient experimentally obtained experimentally for this situation is 0.015 ft/hr. which is considerably less than that predicted. This can be accounted for by any one of the following conjectures:

TABLE VA

RELATIVE DIMENSIONLESS GROUPS

	Reynolds	Weber	Schmidt	Nusselt Equivalent
Chloroform	1520	3.9	178	178
Methyl Isobutyl Ketone	568	5.68	318	181
Benzene	437	1.72	390	21.3
Toluene	595	1.71	269	19.6
Carbon Tetrachloride*	1133	4.26	450	392
Carbon Tetrachloride**	1133	4.26	450	61.4
Isopropyl Ether	1030	4.60	137	229
Water ***	520	4.04	1200	11.1
Water ****	520	4.04	1200	4.15

* Direction of transfer from water to the dispersed CCl_4

** Direction of transfer from the disperse CCl_4 to the water.

*** Direction of transfer from continuous CCl_4 phase to dispersed H_2O

**** Direction of transfer from dispersed H_2O to continuous CCl_4 phase.

TABLE VB

COMPARISON OF EXPERIMENTAL AND PREDICTED RESULTS IN THE TURBULENT FLOW REGION

	Nusselt Equivalent Numbers			
	Experimental[1]	Predicted[1]	Predicted[2]	Experimental[2]
Chloroform	178	370	345	670
Methyl Isobutyl Ketone	181	193	244	181
Benzene	21.3	170	82.5	181
Toluene	19.6	190	92	115
Carbon Tetrachloride*	392	-392	392	392
Carbon Tetrachloride**	61.4	392	392	310
Isopropyl Ether	229	215	225	229
Water ***	11.1	277	268	56
Water ****	4.15	277	268	4.15

Experimental[1] - Actual value obtained for free-fall conditions without correcting for association.

Experimental[2] - Experimental[1] values corrected for concentrations based on single molecules.

Predicted[1] - Without the use of Webers group. Thus, based on analogy to turbulent heat or momentum transfer.

Predicted[2] - Solution of the equation for Dimensional analysis.

1. There were insufficient data to evaluate the effect of the continuous phase chemical properties on the extraction coefficient. Hence these were assumed to be insignificant. This may not have been a correct assumption.

2. Although acetic acid ionizes to the extent of only 1% when its concentration is 0.01 lb.moles/cu.ft. in the dispersed water phase, the reduction in concentration in the film would result in a higher degree of ionization. The ionic state is incompatible with the non-polar carbon tetrachloride, therefore, the rate of extraction would be reduced.

3. The ion-dipole attraction between acetic acid and water could possibly retard the rate of extraction.

4. Heretofore the continuous phase film coefficient has been disregarded. Thus when carbon tetrachloride containing 0.01 lb.moles of HA_c per cu. ft. the resistance of the continuous water phase is $\frac{1}{Kk_c} = 0.05 \frac{1}{k_c}$. However, when water is dispersed containing 90.01 lb.moles cu.ft. acetic acid per cu. ft. the resistance of the continuous carbon tetrachloride phase is $\frac{1}{K^{\perp}k_c} = 75 \frac{1}{k_c}$ and hence is not subordinated to the dispersed phase resistance.

5. An experimental value of K_o for overall conditions is being compared to free-fall K_o. If sufficient information were present to evaluate free-fall K_o the result might be more in agreement.

The dependence of the rate of extraction on the hydrodynamics of the disperse phase system is well established by the manner the overall extraction coefficients depends on the velocity pattern. The concept of transfer based on single molecules in non-polar organic liquids although new to the field of solvent extraction is supported by experiments in the field of gas absorption. Whitney and Vivian[24] found in the absorption of sulphur dioxide in water that a better correlation

is obtained for their data if they included only the undissociated sulphur dioxide in their transfer calculations. Hydrolysis plays a part in this mass transfer mechanism just as association plays a part in our mechanism of solvent extraction.

By means of these extraction rate correlations in the turbulent and transitional flow regimes it is possible to predict the rate of transfer during free-fall in such industrial equipment as spray towers and sieve plate columns. As has been shown, the end effects during drop formation are quite appreciable. However, no correlation is presented at this time because of the complexity of analyzing the end-effect condition. This is especially difficult because of the dynamic interfacial area.

In the correlation for the turbulent flow regime the exponents of the dimensionless group can vary over a small range and still fit the data equally well.

CHAPTER X

CONCLUSIONS

The rate of extraction has been investigated for liquid-liquid extraction in a single drop tower. Various organic solvents have been used as the dispersed phase and acetic acid transferred from them to a continuous demineralized water phase. From the results of the investigation it is possible to conclude:

1. It is possible to separate the affects of physical variables on the rate of extraction by varying one at a time and holding the remaining variables constant.

2. In order to correlate the affects of chemical variables on the rate of extraction, a comparison must be made between the results of similar systems rather than varying one variable at a time.

3. From the experimental results it appears that interfacial tension determines the rate of extraction only insofar as it influences the hydrodynamics and shape of the falling drop.

4. The rate of extraction occurring during drop formation is much larger than predicted. These end-effects are determined largely by drop size, nozzle dimensions, molecular diffusivity and drop formation time.

5. The hydrodynamics of the falling drops agree with known laws of hydrodynamics for perfect spheres in the transitional and viscous flow regimes.

6. The hydrodynamics of falling drops deviate from that of perfect spheres in the turbulent flow regime. The drops tend to deform with increasing diameter and have the shape of an ellipsoid of revolution about the minor axis. As a result of drop deformation, the velocity reaches a maximum value around a Reynolds number of 600 and no further increase occurs.

7. In the transitional flow regime, between a Reynolds number of 100 to 300, the rate of extraction is expressed by the diffusion equation in spherical coordinates when the major resistance is in the dispersed phase.

8. In the turbulent flow regime, above a Reynolds number of 300 to 350, the rate of extraction is given by the expression:

$$\frac{K_o d}{D_i} = 0.03 \left(\frac{dV\rho_d}{\mu_d}\right)^{0.8} \left(\frac{\mu_d}{\rho_d D_i}\right)^{0.44} \left(\frac{d\, V^2 \rho_c}{I.T.}\right)^{0.8}$$

9. When organic solvents were dispersed and acetic acid transferred from them to the continuous water phase the resistance to transfer is dependent upon the dispersed phase.

10. In dealing with non-polar organic liquids the degree of association must be taken into account and the rate of transfer can be approximated by assuming that only the single unassociated molecules diffuse across the film in the turbulent flow regime.

11. When water is dispersed and an organic solvent made the continuous phase the rate of extraction obtained experimentally is lower than that predicted. This may be due to analytical errors in the experiments, ion-dipole effects, lack of information on the affect of the continuous phase, or the continuous phase resistance may be important.

12. The extraction coefficient does not depend on the distribution ratio.

The results obtained are by no means inclusive or extensive enough to formulate a complete theory for mass transfer in a falling drop tower. It is therefore recommended that the following points are deserving of further investigation:

1. The effect of the properties of the continuous phase on the rate of extraction should be evaluated by dispersing water in other organic solvents. This would also aid in determining the cause for the deviations in Series M, in which water is dispersed in carbon tetrachloride and acetic acid transferred to the continuous phase.

2. By reversing the direction of transfer so that it is from the continuous to the dispersed phase, for dispersing an organic liquid such as methyl isobutyl ketone in water it will be possible to determine whether association is the factor which governs the rate of transfer. Since association does not take place in hexone the rate of extraction should be the same for either direction of transfer.

3. In future investigations of this nature analysis of results will be facilitated by the selection of solvents and solutes in which neither association nor solvation takes place.

A complete evaluation of the field of mass transfer in liquid-liquid extraction systems will be possible only after the following investigations:

1. The development of a better correlation for molecular diffusivity is needed in order to further the progress of investigations in diffusional operations.

2. The analysis of chemical problems, such as that of association which were encountered here would be facilitated by a better understanding of the chemistry of liquid-solute systems.

3. The kinetics and chemistry of surfaces such as a liquid-liquid interface, is in need of investigation.

4. Once the hydrodynamics, chemistry and mass transfer rate equations are determined the rate of extraction in industrial equipment such as packed columns can be determined concisely.

APPENDIX A

EQUILIBRIUM DATA

The logarithmic mean driving force is employed in computing the overall mass transfer coefficients. In order to calculate the driving force an exact knowledge of the equilibrium distribution ratio of the acetic acid solute, between the solvents is needed. Mutual solubility data are also necessary to estimate the amount of solute transfer by solvent solution in relation to that by diffusion of solute molecules along.

The distribution ratio was determined experimentally for the partition of acetic acid between the six solvents and demineralized water. The procedure consisted of adding 50 mls. of solvent and 10 or 50 mls. of demineralized water to a 125 ml. separatory funnel followed by varying amounts of acetic acid ranging from 5 mls. down to 0.05 mls. to each of the separate funnels. The phases were thoroughly agitated to bring the system to phase equilibrium and the funnels were then left sealed for several hours. The two phases were separated and the acetic acid concentration of each determined by titration with standard sodium hydroxide using thymol blue for an indicator.

The experimental data so obtained were calculated on the basis of concentrations in lb. moles/cu. ft. and compared with data for the same systems in equivalent units from the following references:

1. International Critical Tables
2. Transactions of the American Institute of Chemical Engineers.
3. Solubilities of Organic Chemicals, by Seidell
4. Industrial and Engineering Chemistry.

The data are tabulated for comparison and are also plotted in terms of distribution ratio versus concentration in each phase, and concentration of one phase versus equilibrium concentration of the other phase. The following systems are presented:

carbon tetrachloride-acetic acid-water

methyl isobutyl ketone-acetic acid-water

benzene-acetic acid-water

toluene-acetic acid-water

isopropyl ether-acetic acid-water

chloroform-acetic acid-water

carbon tetrachloride-propionic acid-water

The possible effect of wetting agents on the distribution ratio was investigated using Tergitol #4 made by Union Carbide and Carbon Chemicals Corporation. A concentration of wetting agent of 0.1% by volume was used with the system carbon tetrachloride-acetic acid-water. The results obtained were almost identical with those for the same system without wetting agent. Since the interfacial tension of the system with the wetting agent is considerably lower than that without, it is therefore concluded that interfacial tension does not play a part in determining the equilibrium distribution of a solute between two immiscible liquids.

The mutual solubility data were obtained entirely from the literature. When sufficient data were available a triangular plot has been made for the mutual solubility of all components. Otherwise the data are presented for the more pertinent conditions in Table XVIII. Two additional references contained mutual solubility information:

1. Synthetic Organic Chemicals, Union Carbide and Carbon Chemicals Corporation.

2. Chemical Engineering Handbook, Perry

The experimental results obtained for the distribution ratios agreed favorably with the results taken from the literature. The only deviations that occur were at acetic acid concentrations below 0.0001 lb. moles/cu. ft. The ionization of acetic acid at concentrations below this figure is high and could easily cause the deviation in the data. Association in organic liquids such as carbon tetrachloride, benzene, toluene and chloroform tends to also decrease at these extremely low concentrations.

The distribution ratio such as presented in the results is the ratio of the concentration of acetic acid in the water phase to that of solvent phase in equilibrium with it. The distribution ratio may either vary with concentration or remain constant depending upon the solvent involved. Thus in the systems using methyl isobutyl ketone and isopropyl ether, in both of which acetic acid does not associate at the concentrations studied, the distribution ratio remains constant. In contrast, the distribution ratio varies with concentration for the other four systems, using acetic acid as a solute. The degree of association

in these latter systems can be determined by examining the data through the equilibrium constant, which does not change with concentration.

Thus, for a system in which association takes place, equilibrium is given by $A_n \rightleftarrows nA_1$, where in one phase the molecular weight is n times that in the other phase. The degree of association is defined as n. The distribution ratio can be expressed as:

$$K = C_w/C_s$$

If association takes place in the solvent phase to the degree n, the equilibrium constant is given by:

$$K_c = C_w(1 - \alpha)^n/C_s$$

If we assume further that α, the degree of ionization, is zero, then it is possible to determine the degree of association through the relation:

$$n = \frac{\mathrm{Log}_e(C_{s1}/C_{s2})}{\mathrm{Log}_e(C_{w1}/C_{w2})}$$

Selection of equilibrium concentrations of water and solvent at two points fairly close together eliminates variations in n. Having determined the degree of association it is possible to estimate the number of single and double molecules in the solvent phase by means of:

$$2D_o + 1S = n(D_o + S)$$

where:

D_o - number of double molecules

S - number of single molecules.

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NOT CLASSIFIED

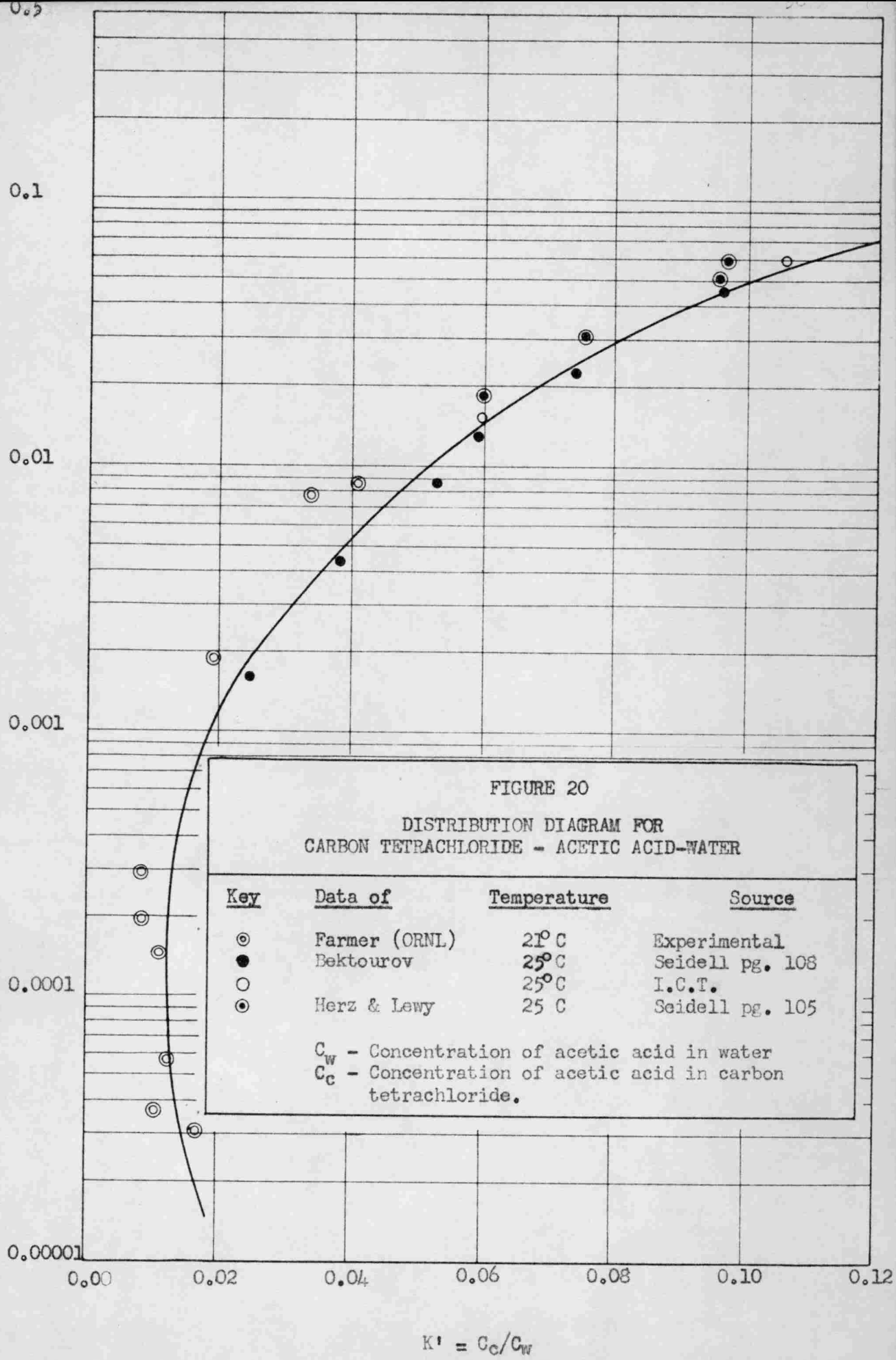

FIGURE 20

DISTRIBUTION DIAGRAM FOR
CARBON TETRACHLORIDE - ACETIC ACID-WATER

Key	Data of	Temperature	Source
⊙	Farmer (ORNL)	21° C	Experimental
●	Bektourov	25° C	Seidell pg. 108
○		25° C	I.C.T.
⊙	Herz & Lewy	25 C	Seidell pg. 105

C_w - Concentration of acetic acid in water
C_c - Concentration of acetic acid in carbon tetrachloride.

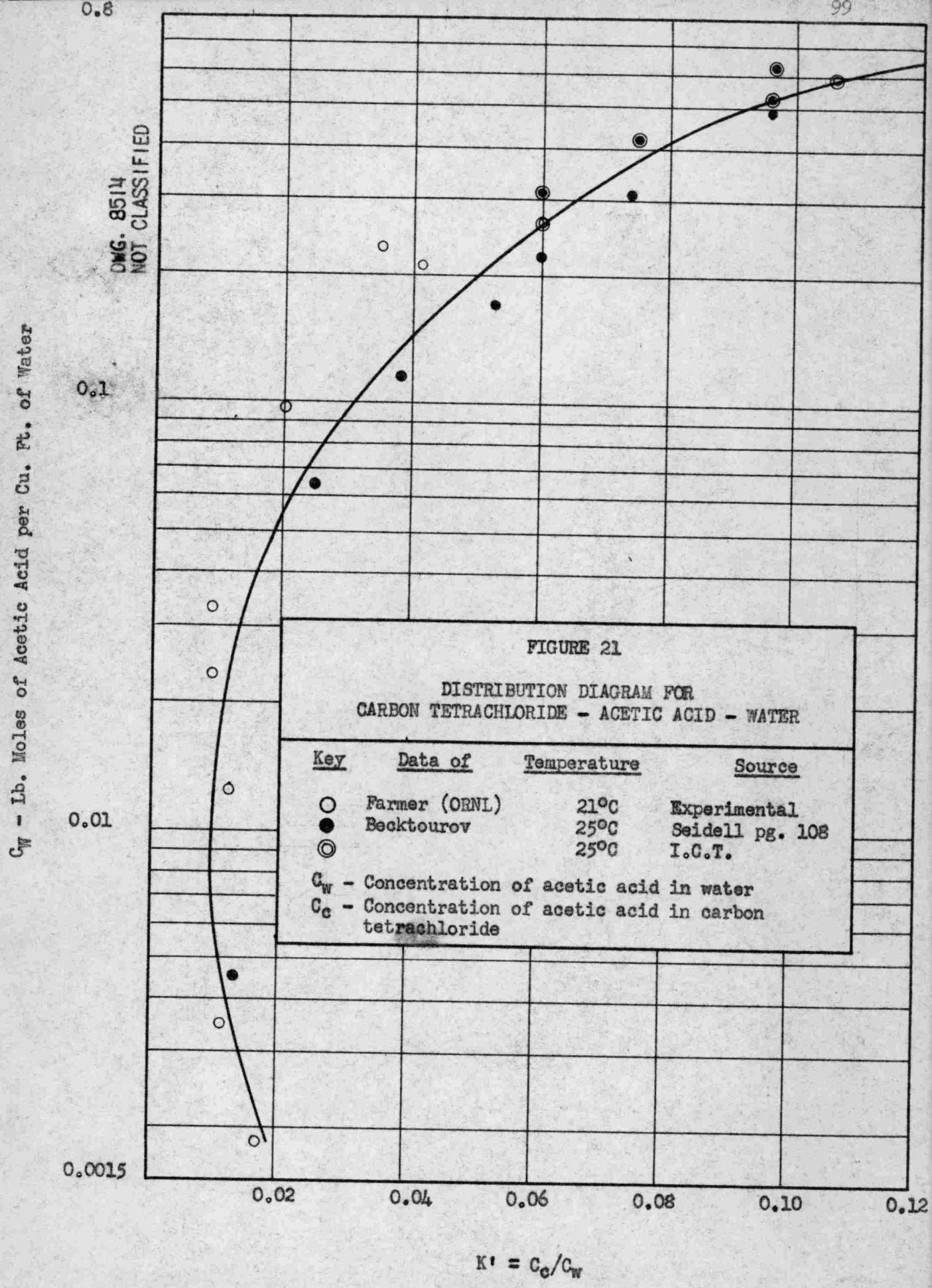

FIGURE 21

DISTRIBUTION DIAGRAM FOR
CARBON TETRACHLORIDE - ACETIC ACID - WATER

Key	Data of	Temperature	Source
○	Farmer (ORNL)	21°C	Experimental
●	Becktourov	25°C	Seidell pg. 108
◎		25°C	I.C.T.

C_W - Concentration of acetic acid in water
C_c - Concentration of acetic acid in carbon tetrachloride

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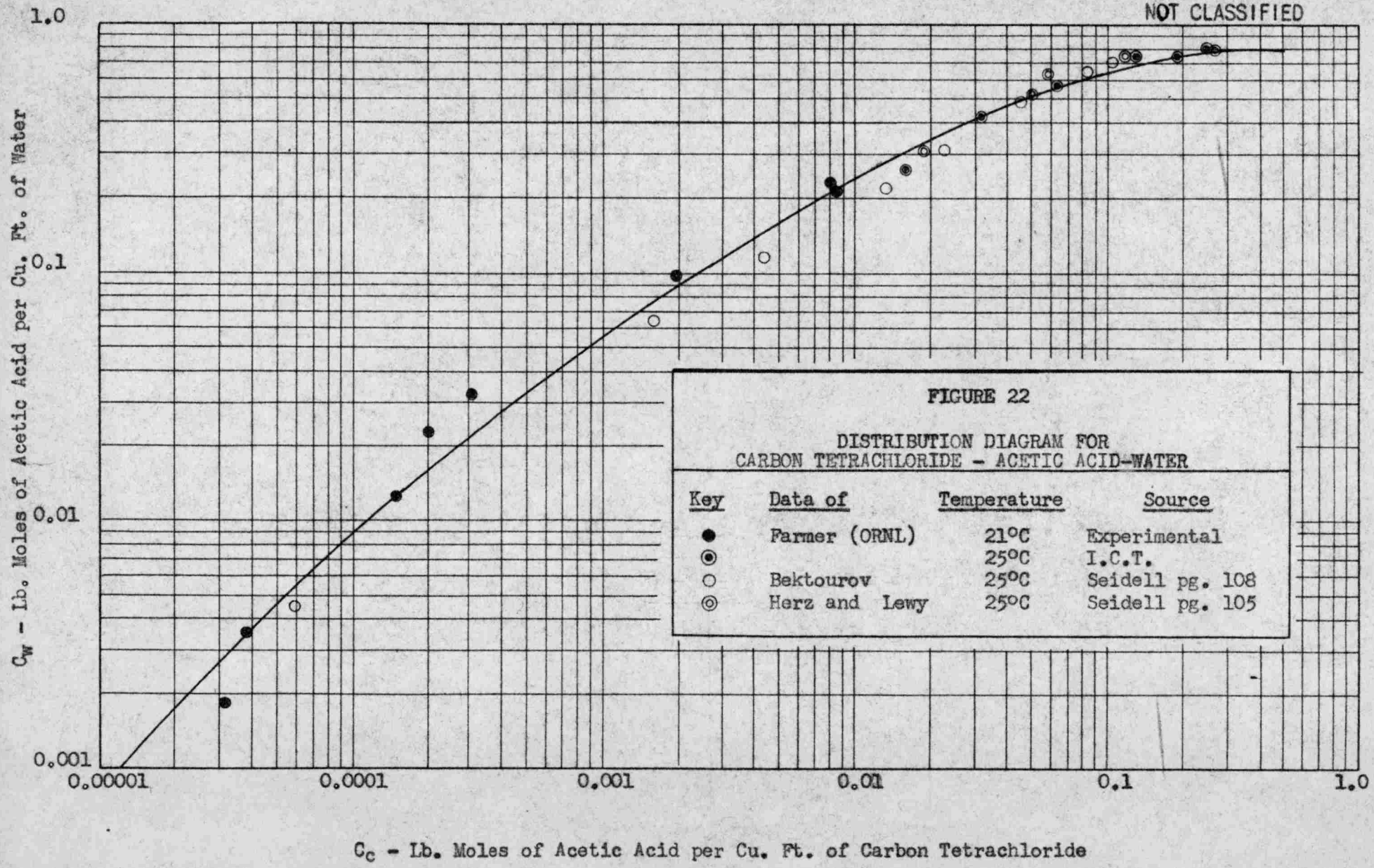

FIGURE 22

DISTRIBUTION DIAGRAM FOR
CARBON TETRACHLORIDE - ACETIC ACID-WATER

Key	Data of	Temperature	Source
●	Farmer (ORNL)	21°C	Experimental
◉		25°C	I.C.T.
○	Bektourov	25°C	Seidell pg. 108
◎	Herz and Lewy	25°C	Seidell pg. 105

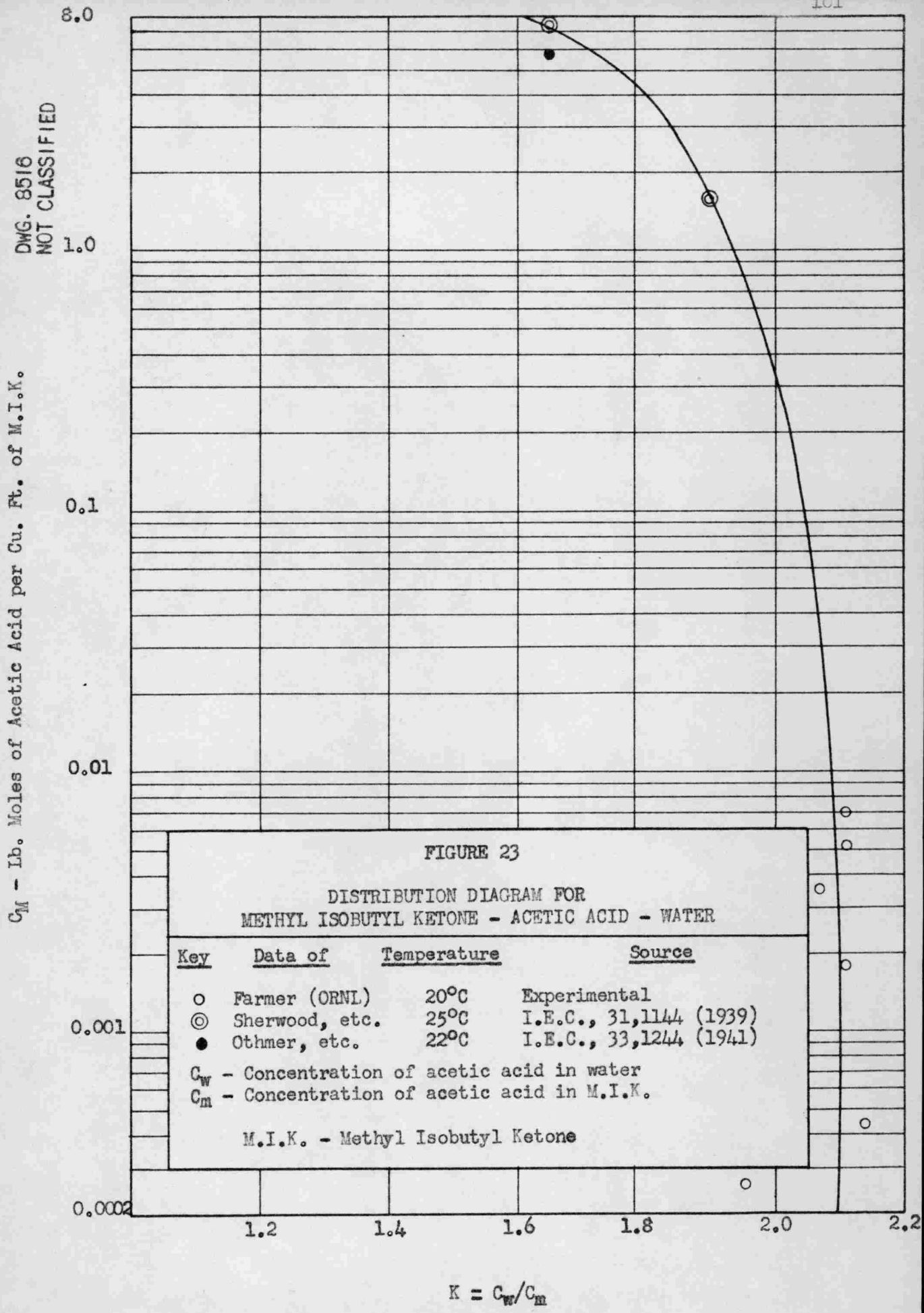

FIGURE 23

DISTRIBUTION DIAGRAM FOR
METHYL ISOBUTYL KETONE - ACETIC ACID - WATER

Key	Data of	Temperature	Source
○	Farmer (ORNL)	20°C	Experimental
◎	Sherwood, etc.	25°C	I.E.C., 31,1144 (1939)
●	Othmer, etc.	22°C	I.E.C., 33,1244 (1941)

C_w - Concentration of acetic acid in water
C_m - Concentration of acetic acid in M.I.K.

M.I.K. - Methyl Isobutyl Ketone

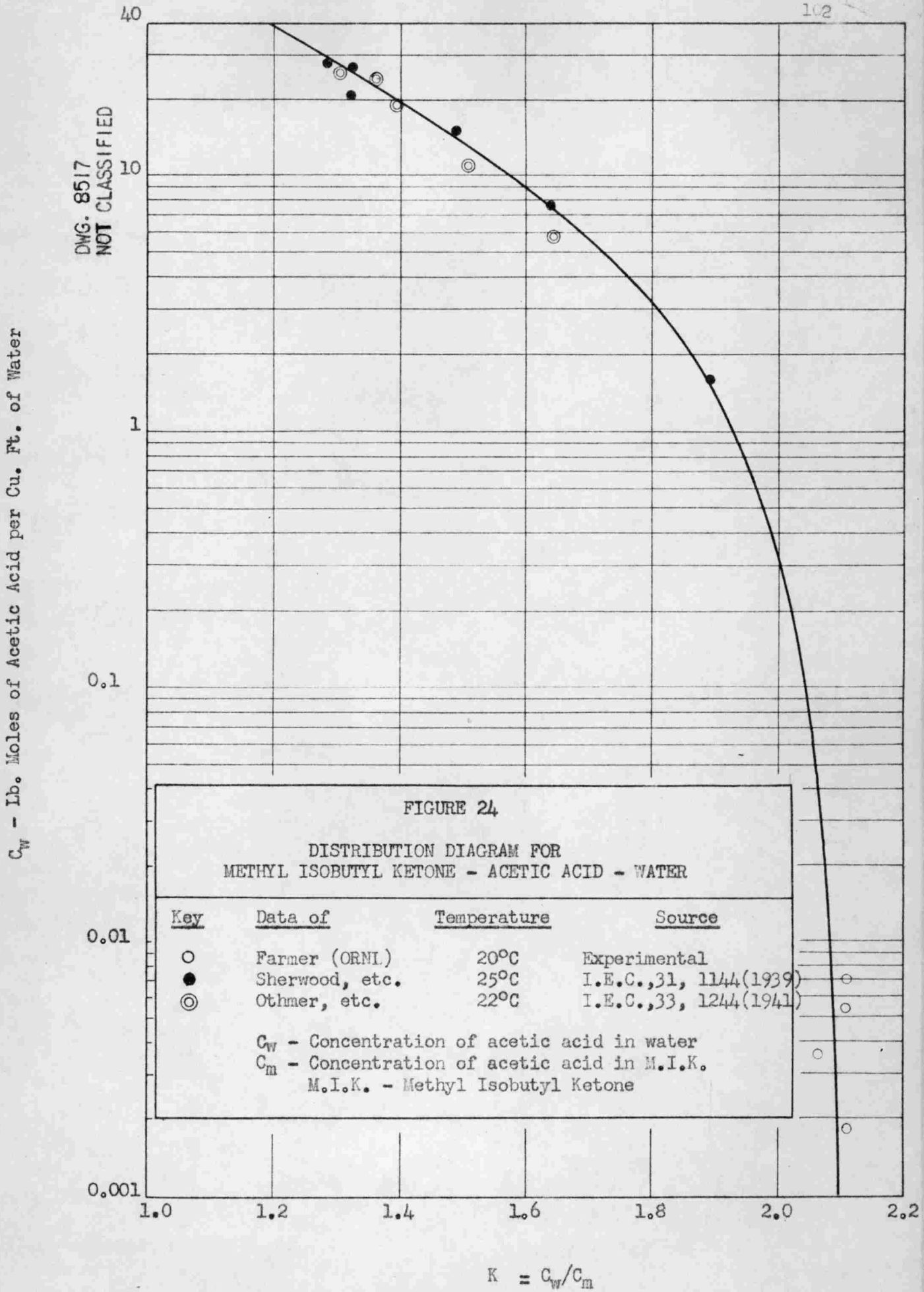

FIGURE 24

DISTRIBUTION DIAGRAM FOR
METHYL ISOBUTYL KETONE - ACETIC ACID - WATER

Key	Data of	Temperature	Source
○	Farmer (ORNL)	20°C	Experimental
●	Sherwood, etc.	25°C	I.E.C.,31, 1144(1939)
◎	Othmer, etc.	22°C	I.E.C.,33, 1244(1941)

C_w - Concentration of acetic acid in water
C_m - Concentration of acetic acid in M.I.K.
M.I.K. - Methyl Isobutyl Ketone

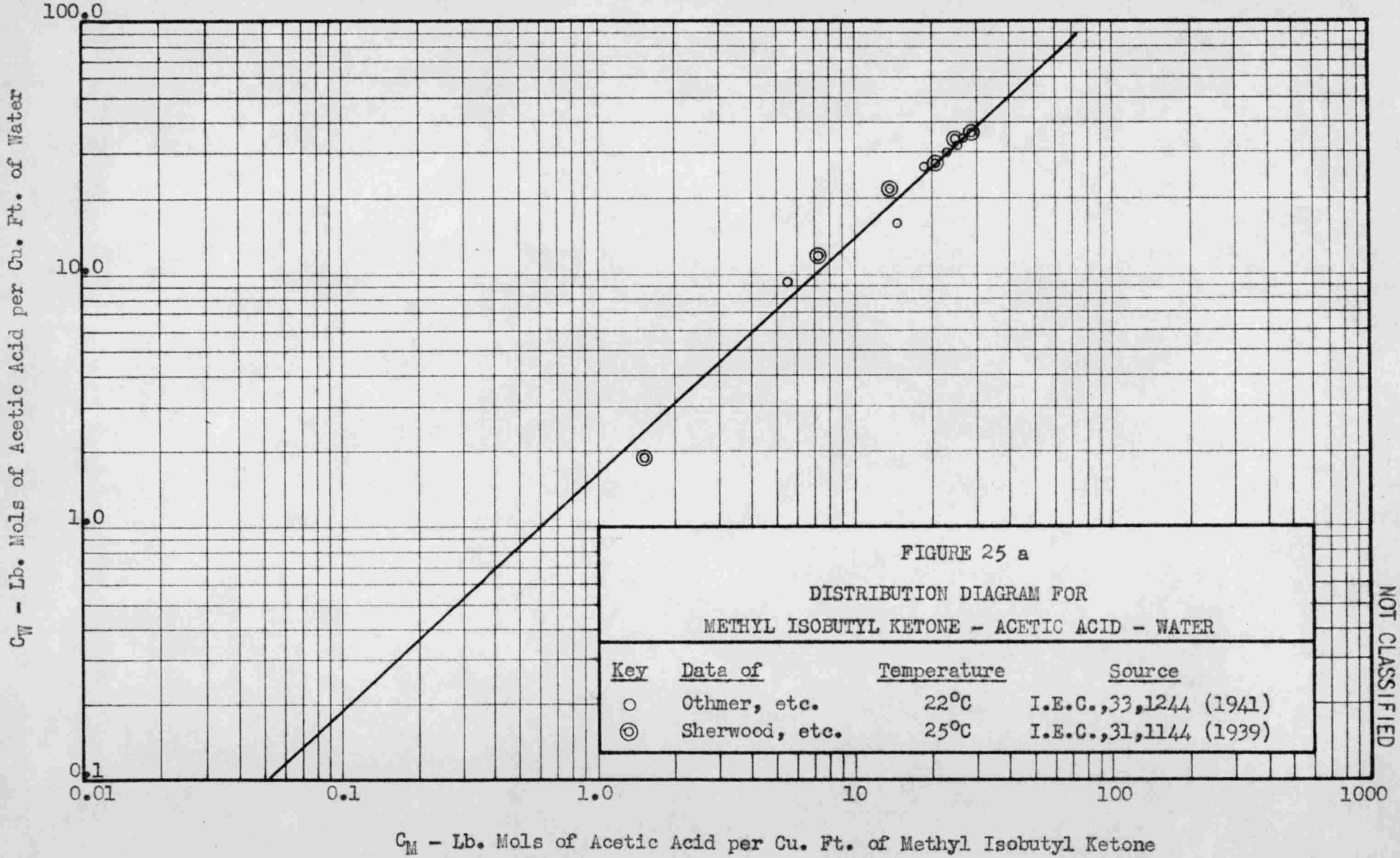

FIGURE 25 a

DISTRIBUTION DIAGRAM FOR

METHYL ISOBUTYL KETONE - ACETIC ACID - WATER

Key	Data of	Temperature	Source
○	Othmer, etc.	22°C	I.E.C.,33,1244 (1941)
◎	Sherwood, etc.	25°C	I.E.C.,31,1144 (1939)

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NOT CLASSIFIED

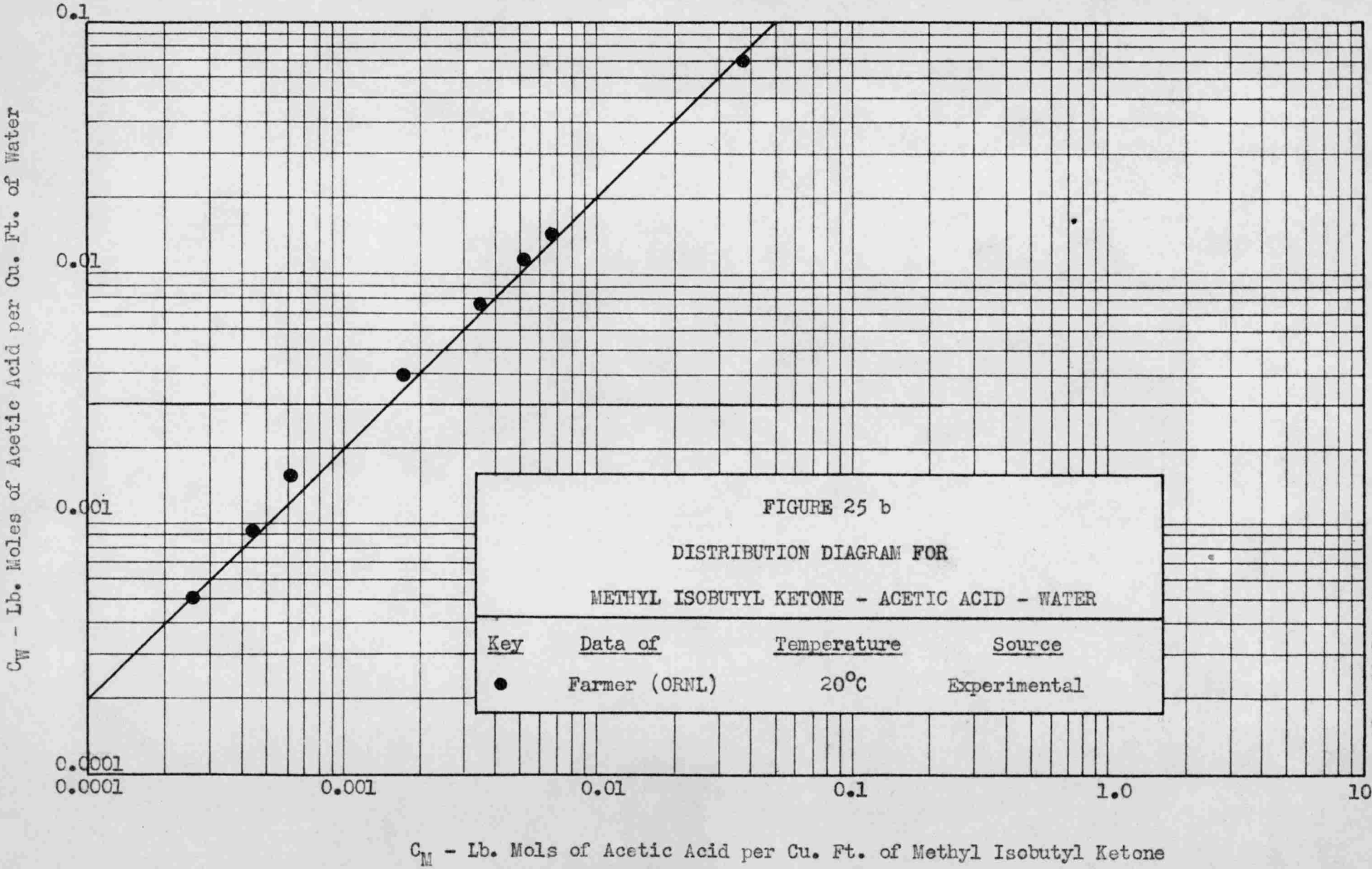


FIGURE 25 b

DISTRIBUTION DIAGRAM FOR

METHYL ISOBUTYL KETONE - ACETIC ACID - WATER

Key	Data of	Temperature	Source
●	Farmer (ORNL)	20°C	Experimental

DWG. 8519
NOT CLASSIFIED

DWG. 8520
NOT CLASSIFIED

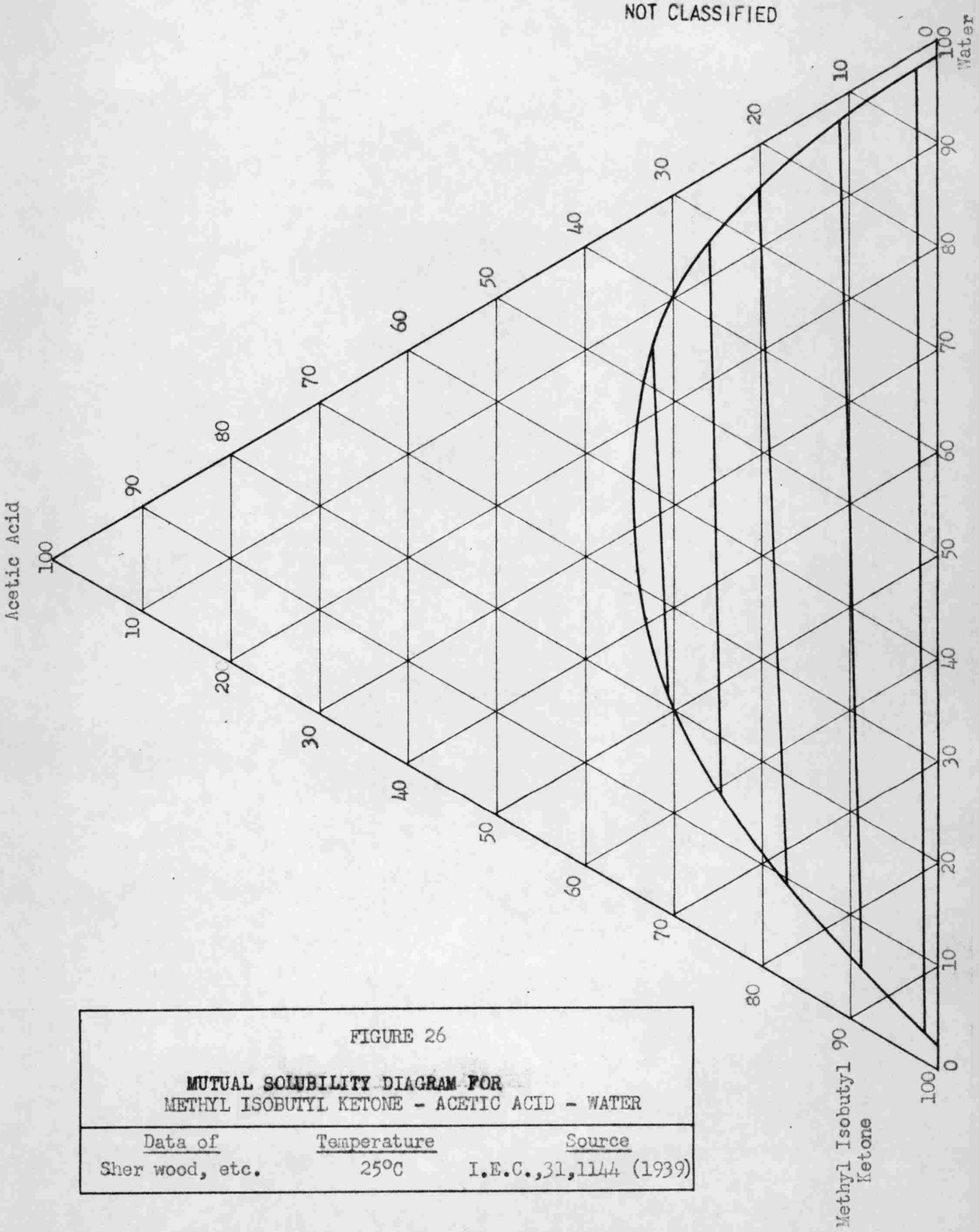


FIGURE 26

MUTUAL SOLUBILITY DIAGRAM FOR
METHYL ISOBUTYL KETONE - ACETIC ACID - WATER

Data of	Temperature	Source
Sher wood, etc.	25°C	I.E.C.,31,1144 (1939)

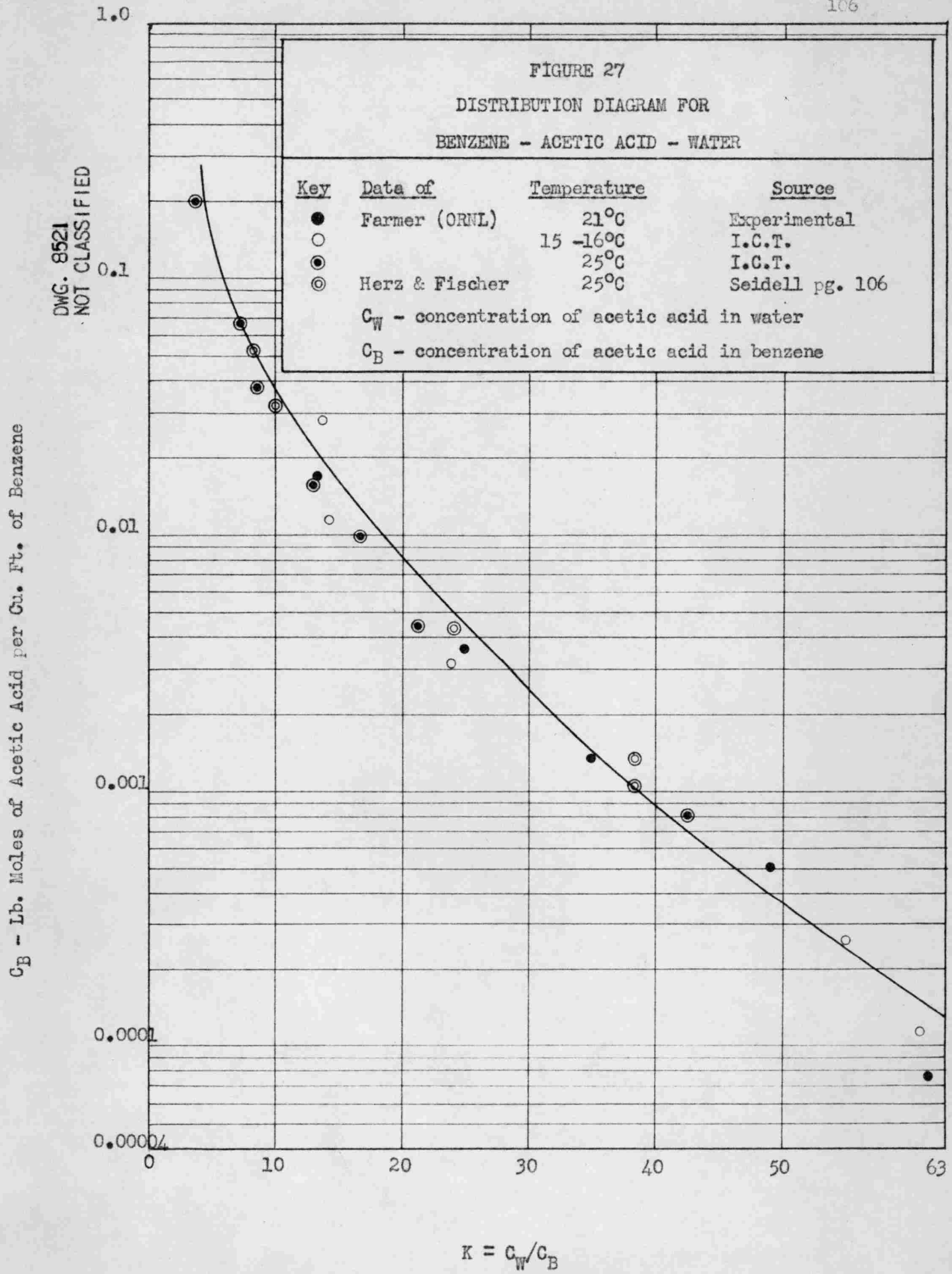


FIGURE 27
DISTRIBUTION DIAGRAM FOR
BENZENE - ACETIC ACID - WATER
Key
Data of
Temperature
Source
Farmer (ORNL)
21°C
Experimental
15 -16°C
I.C.T.
25°C
I.C.T.
Herz & Fischer
25°C
Seidell pg. 106
C_W - concentration of acetic acid in water
C_B - concentration of acetic acid in benzene
DWG. 8521
NOT CLASSIFIED
C_B - Lb. Moles of Acetic Acid per Cu. Ft. of Benzene
1.0
0.1
0.01
0.001
0.0001
0.00004
0
10
20
30
40
50
63
$K = C_W/C_B$

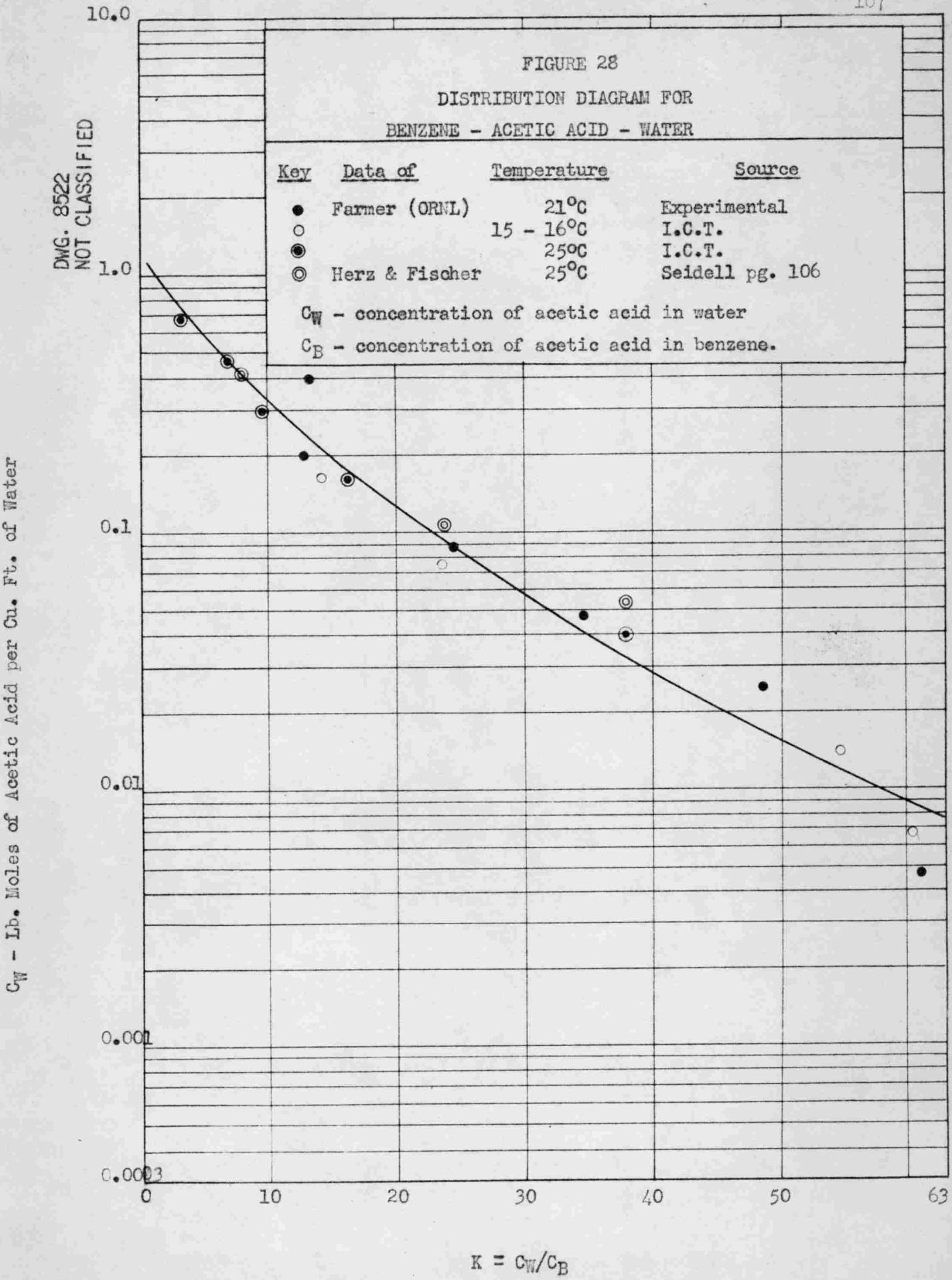


FIGURE 28
DISTRIBUTION DIAGRAM FOR
BENZENE - ACETIC ACID - WATER
Key Data of Temperature Source
Farmer (ORNL) 21°C Experimental
15 - 16°C I.C.T.
25°C I.C.T.
Herz & Fischer 25°C Seidell pg. 106
C_W - concentration of acetic acid in water
C_B - concentration of acetic acid in benzene.
DWG. 8522
NOT CLASSIFIED
10.0
1.0
0.1
0.01
0.001
0.0003
0
10
20
30
40
50
63
C_W - Lb. Moles of Acetic Acid per Cu. Ft. of Water
$K = C_W/C_B$

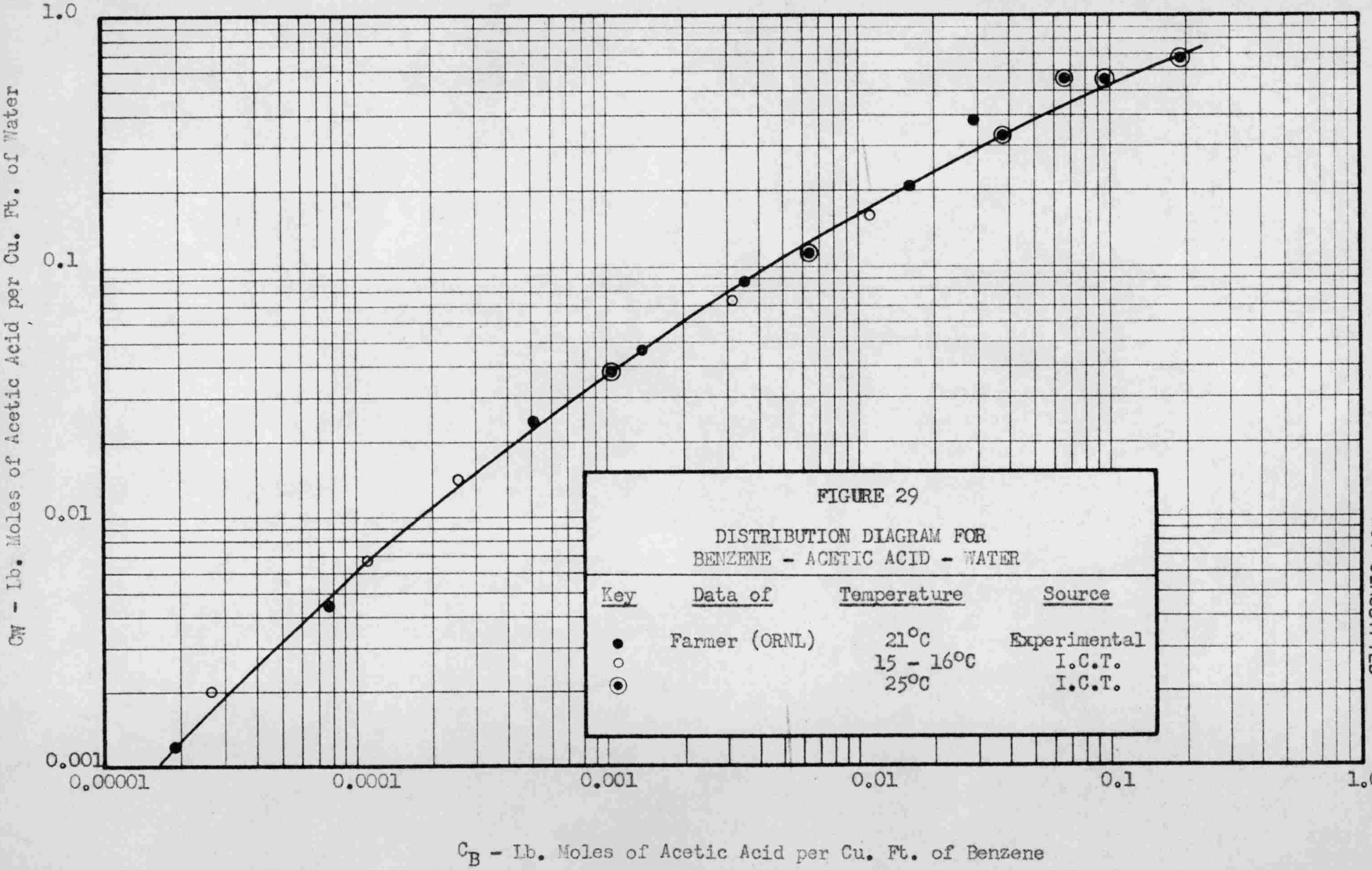
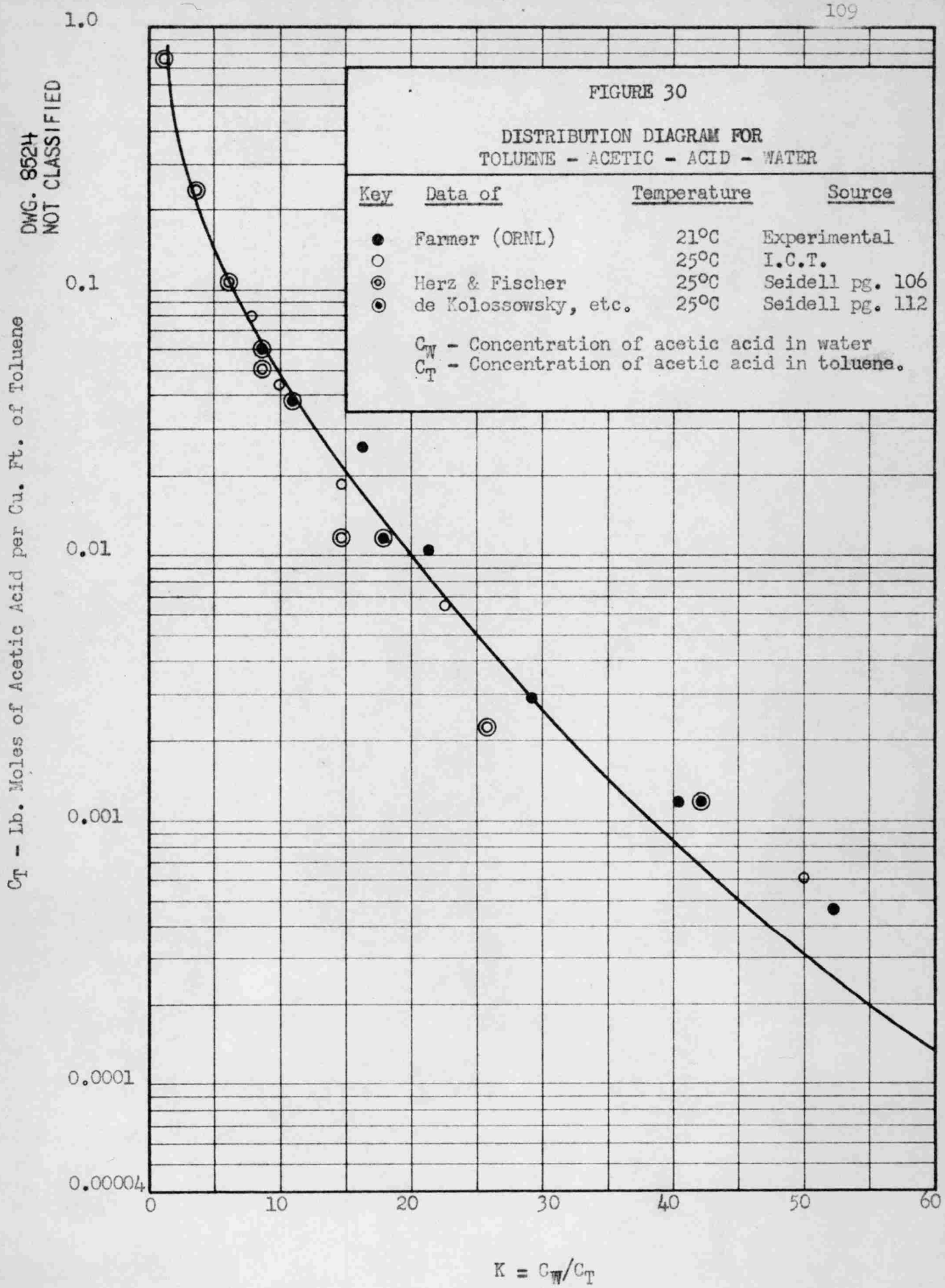


FIGURE 29

DISTRIBUTION DIAGRAM FOR
BENZENE - ACETIC ACID - WATER

DWG. 8523
NOT CLASSIFIED


DWG. 8524
NOT CLASSIFIED
FIGURE 30
DISTRIBUTION DIAGRAM FOR
TOLUENE - ACETIC - ACID - WATER
Key Data of Temperature Source
Farmer (ORNL) 21°C Experimental
25°C I.C.T.
Herz & Fischer 25°C Seidell pg. 106
de Kolossowsky, etc. 25°C Seidell pg. 112
C_W - Concentration of acetic acid in water
C_T - Concentration of acetic acid in toluene.
C_T - Lb. Moles of Acetic Acid per Cu. Ft. of Toluene
1.0
0.1
0.01
0.001
0.0001
0.00004
0
10
20
30
40
50
60
$K = C_W/C_T$

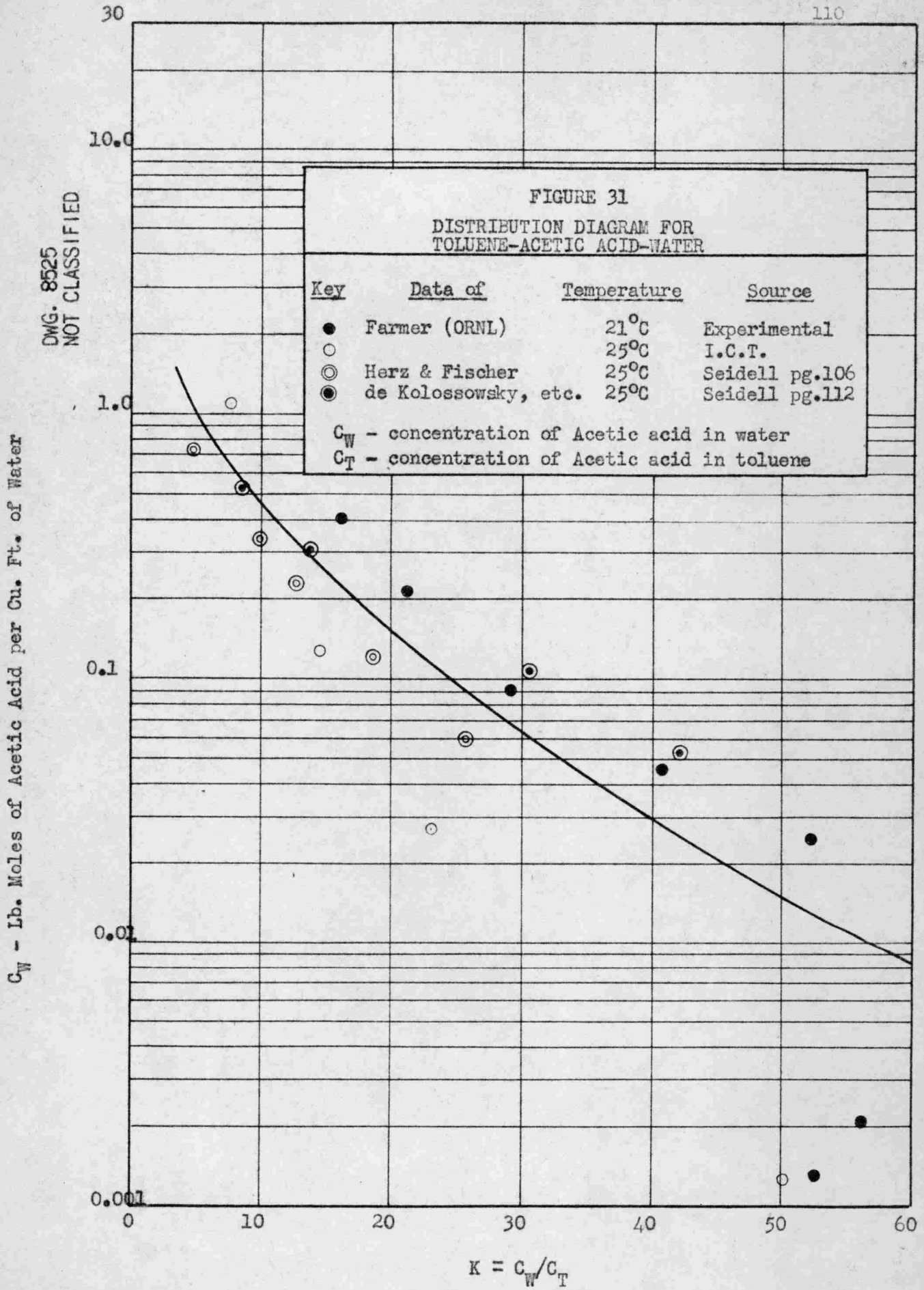


FIGURE 31

DISTRIBUTION DIAGRAM FOR TOLUENE-ACETIC ACID-WATER

Key	Data of	Temperature	Source
●	Farmer (ORNL)	21°C	Experimental
○		25°C	I.C.T.
◎	Herz & Fischer	25°C	Seidell pg.106
◉	de Kolossowsky, etc.	25°C	Seidell pg.112

C_W - concentration of Acetic acid in water
C_T - concentration of Acetic acid in toluene

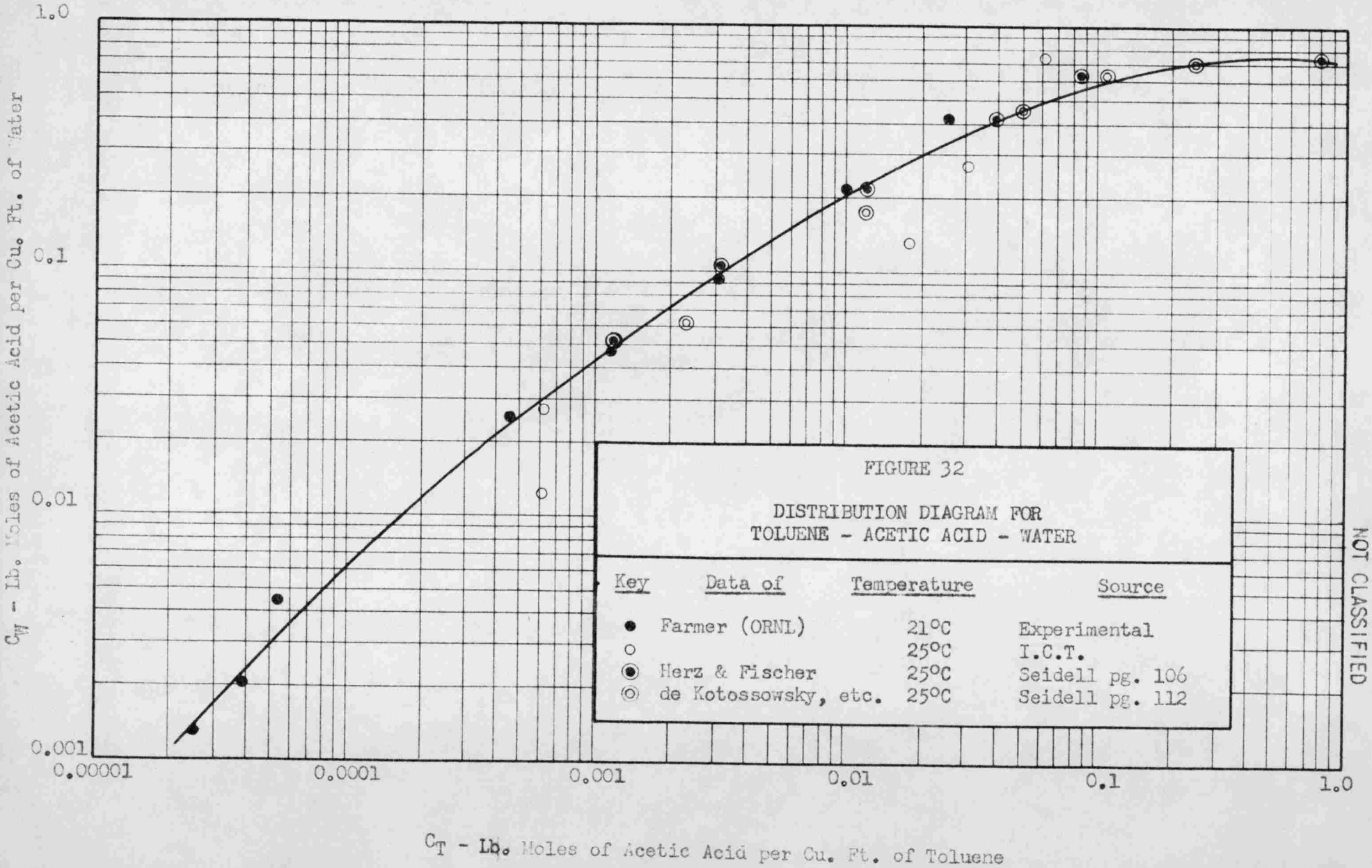


FIGURE 32

DISTRIBUTION DIAGRAM FOR
TOLUENE - ACETIC ACID - WATER

Key	Data of	Temperature	Source
●	Farmer (ORNL)	21°C	Experimental
○		25°C	I.C.T.
◉	Herz & Fischer	25°C	Seidell pg. 106
◎	de Kotossowsky, etc.	25°C	Seidell pg. 112

DWG. 8526
NOT CLASSIFIED

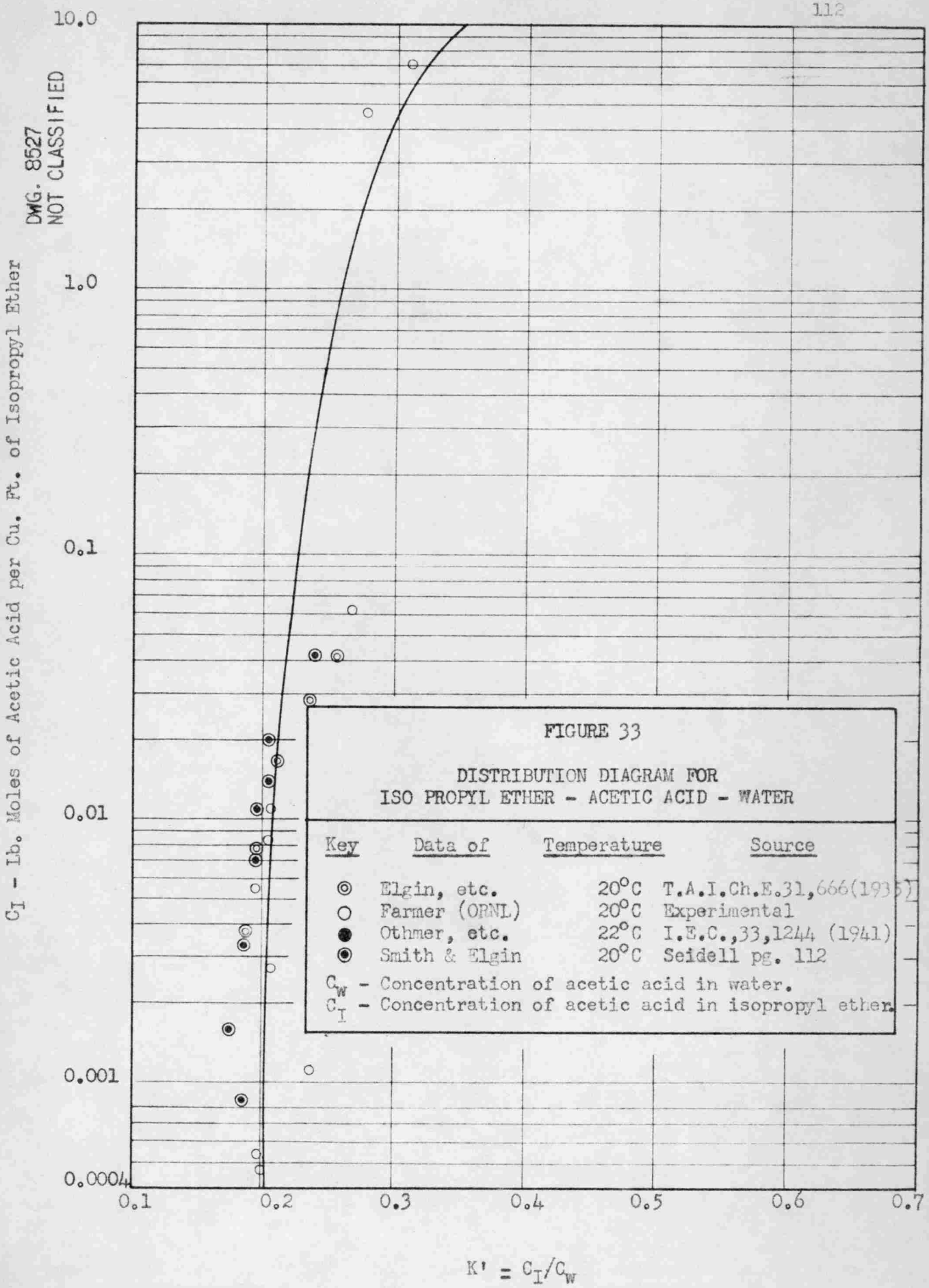


FIGURE 33

DISTRIBUTION DIAGRAM FOR
ISO PROPYL ETHER - ACETIC ACID - WATER

Key	Data of	Temperature	Source
◎	Elgin, etc.	20°C	T.A.I.Ch.E.31,666(1935)
○	Farmer (ORNL)	20°C	Experimental
●	Othmer, etc.	22°C	I.E.C.,33,1244 (1941)
⊙	Smith & Elgin	20°C	Seidell pg. 112

C_W - Concentration of acetic acid in water.
C_I - Concentration of acetic acid in isopropyl ether.

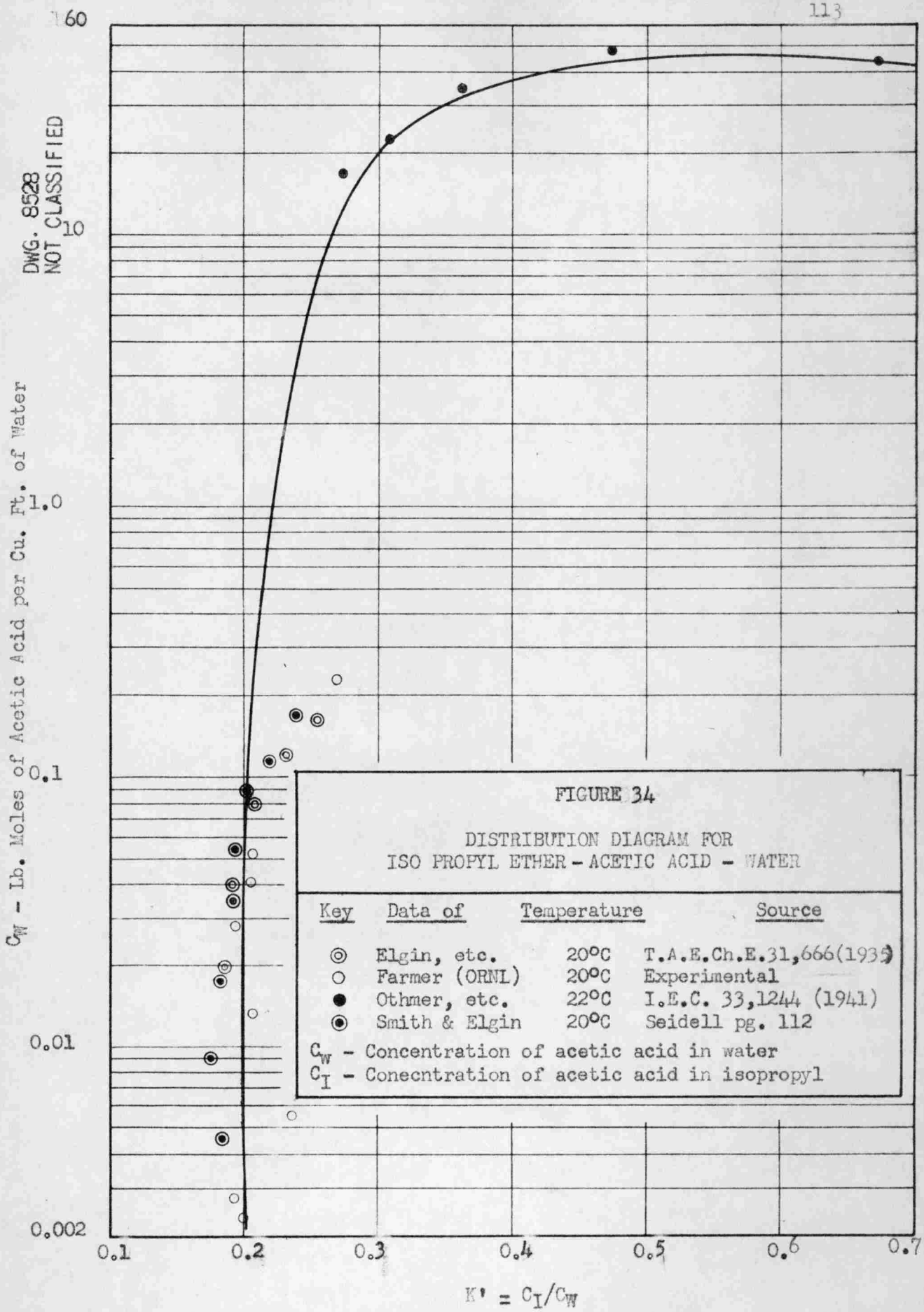


FIGURE 34

DISTRIBUTION DIAGRAM FOR
ISO PROPYL ETHER - ACETIC ACID - WATER

Key	Data of	Temperature	Source
◎	Elgin, etc.	20°C	T.A.E.Ch.E.31,666(1935)
○	Farmer (ORNL)	20°C	Experimental
●	Othmer, etc.	22°C	I.E.C. 33,1244 (1941)
◉	Smith & Elgin	20°C	Seidell pg. 112

C_W - Concentration of acetic acid in water
C_I - Conecntration of acetic acid in isopropyl

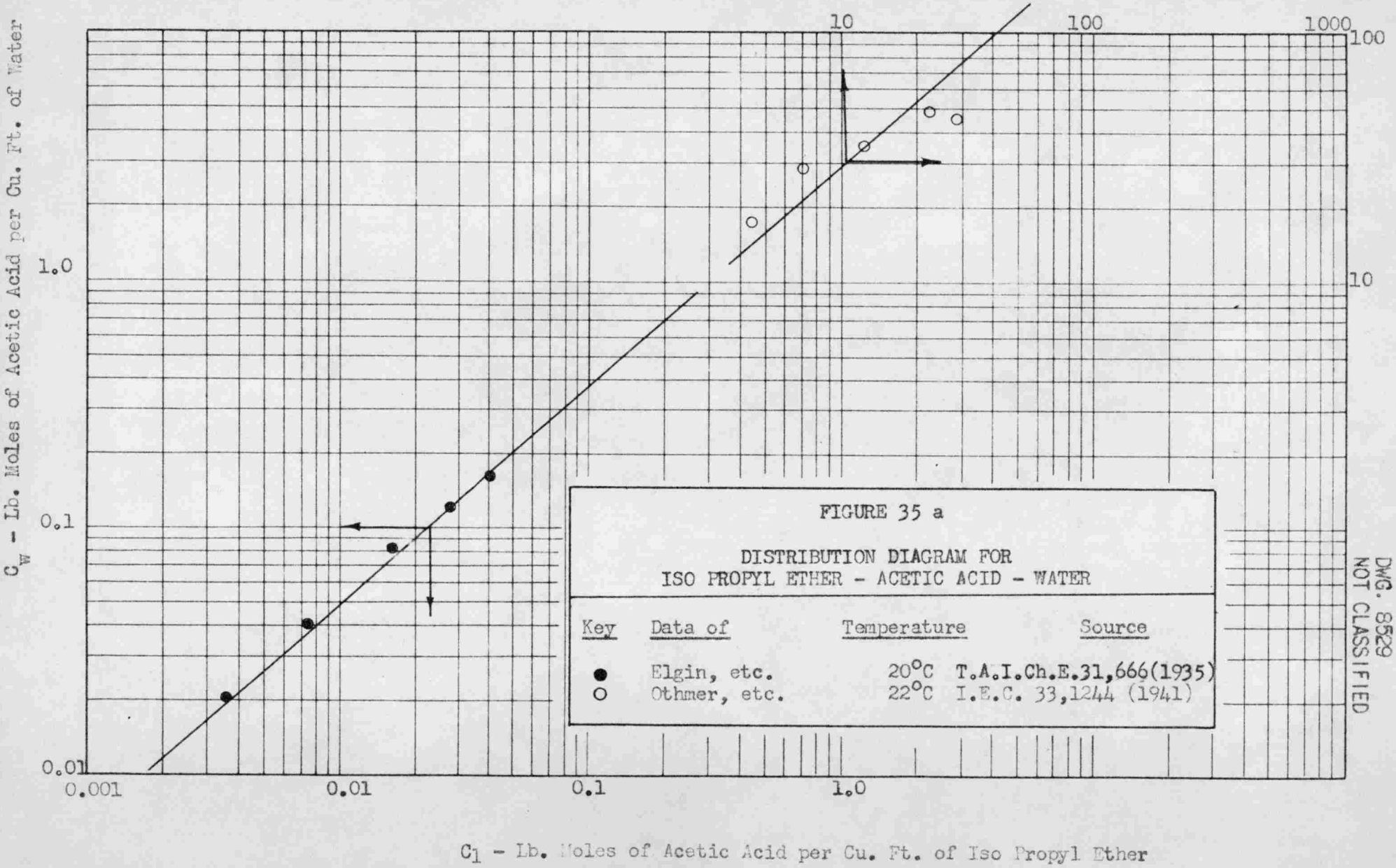


FIGURE 35 a

DISTRIBUTION DIAGRAM FOR
ISO PROPYL ETHER - ACETIC ACID - WATER

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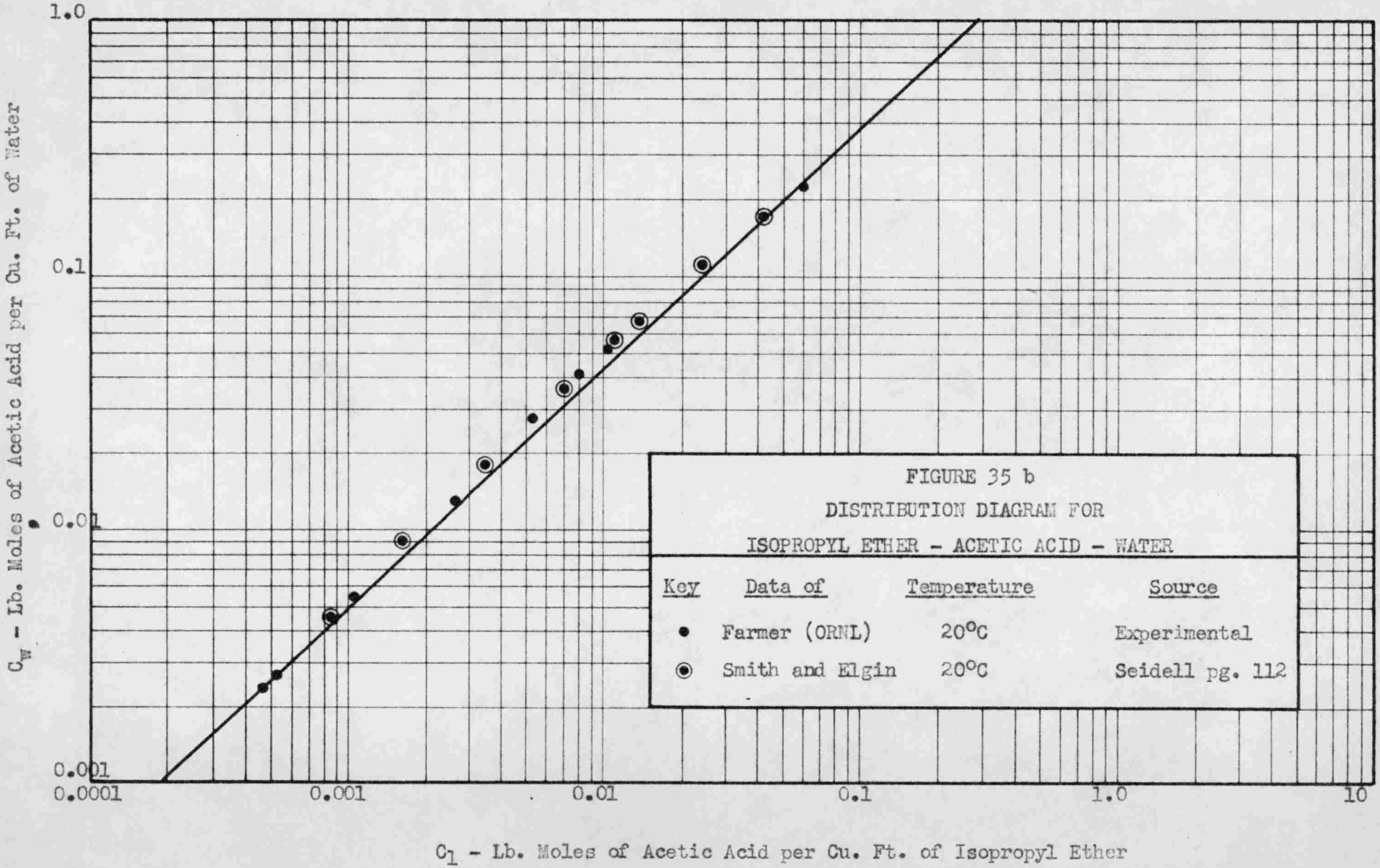

FIGURE 35 b
DISTRIBUTION DIAGRAM FOR
ISOPROPYL ETHER - ACETIC ACID - WATER

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FIGURE 36

DISTRIBUTION DIAGRAM FOR

CHLOROFORM - ACETIC ACID - WATER

Curve	Key	Data of	Temperature	Source
A	□	Bektourov	50°C	Seidell pg. 108
B	■	Bektourov	0°C	Seidell pg. 108
	◉		20°C	I.C.T.
	○		25°C	I.C.T.
	●	Farmer (ORNL)	20°C	Experimental
	△	Vartessian, etc.	18°C	I.E.C., 28, 1353 (1936)
	◎	Herz and Lewy	25°C	Seidell pg. 105
C		Mean of the Data		

C_w - concentration of acetic acid in water
C_c - concentration of acetic acid in chloroform

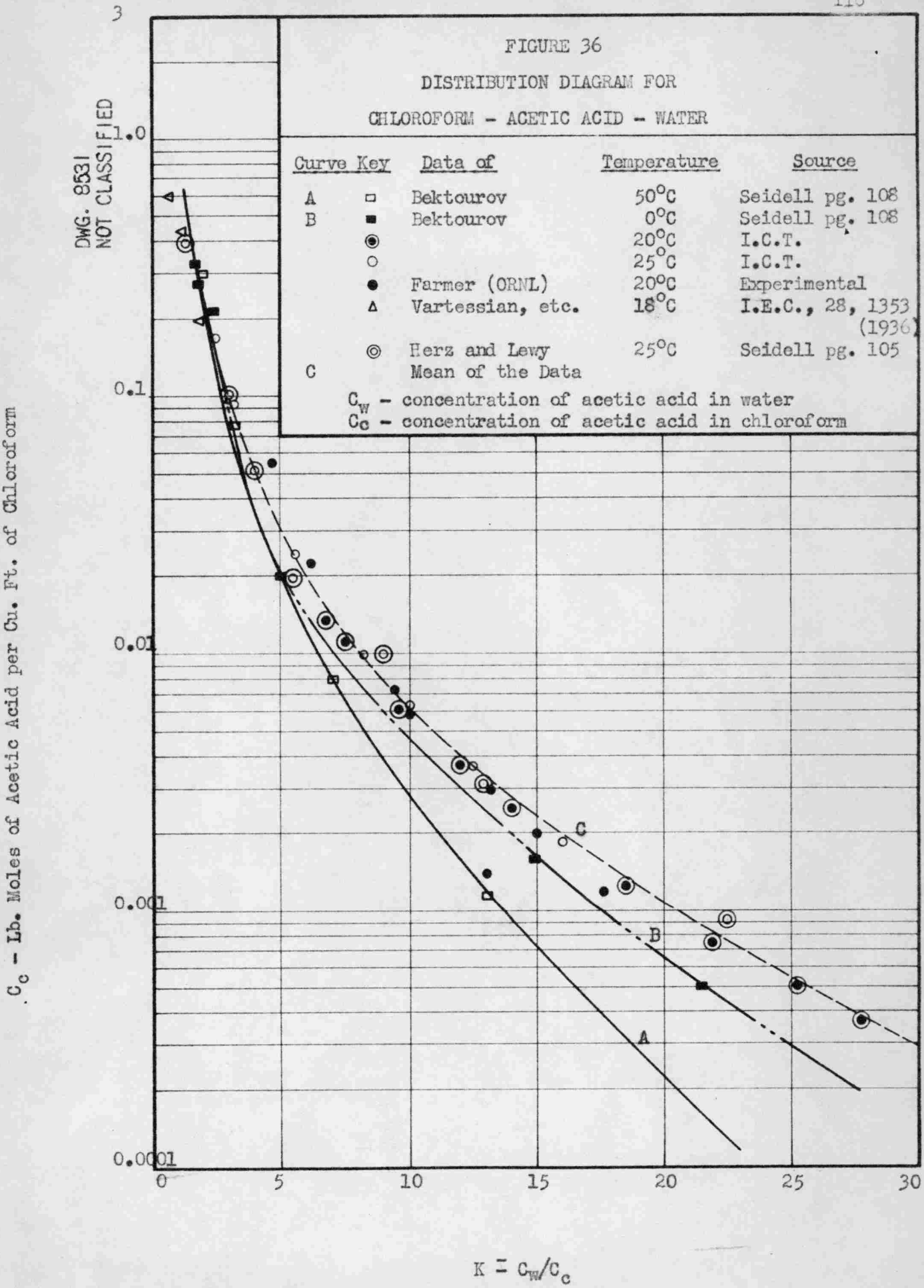

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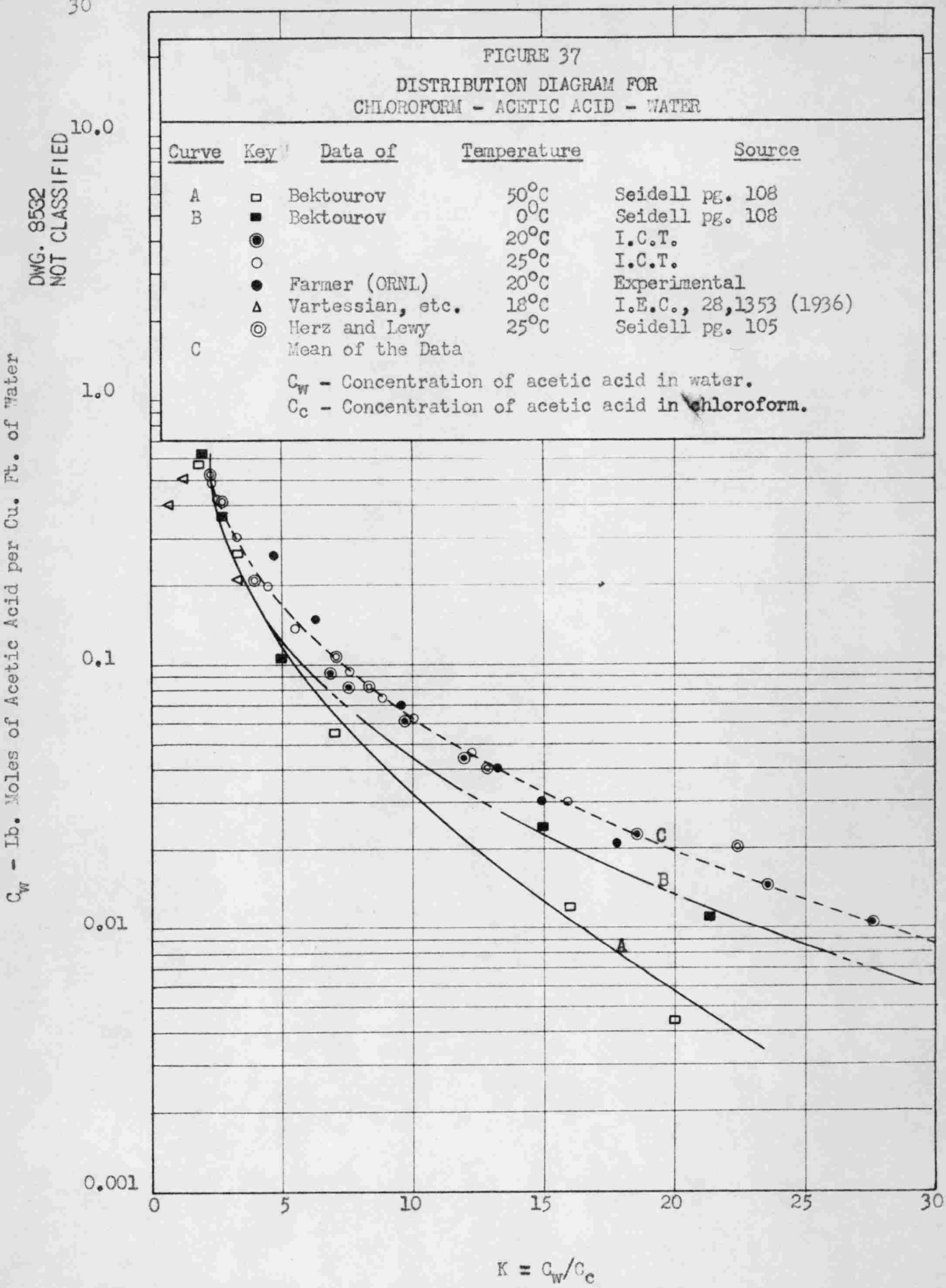
FIGURE 37

DISTRIBUTION DIAGRAM FOR
CHLOROFORM - ACETIC ACID - WATER

Curve	Key	Data of	Temperature	Source
A	□	Bektourov	50°C	Seidell pg. 108
B	■	Bektourov	0°C	Seidell pg. 108
	◉		20°C	I.C.T.
	○		25°C	I.C.T.
	●	Farmer (ORNL)	20°C	Experimental
	△	Vartessian, etc.	18°C	I.E.C., 28,1353 (1936)
	◎	Herz and Lewy	25°C	Seidell pg. 105
C		Mean of the Data		

C_w - Concentration of acetic acid in water.
C_c - Concentration of acetic acid in chloroform.



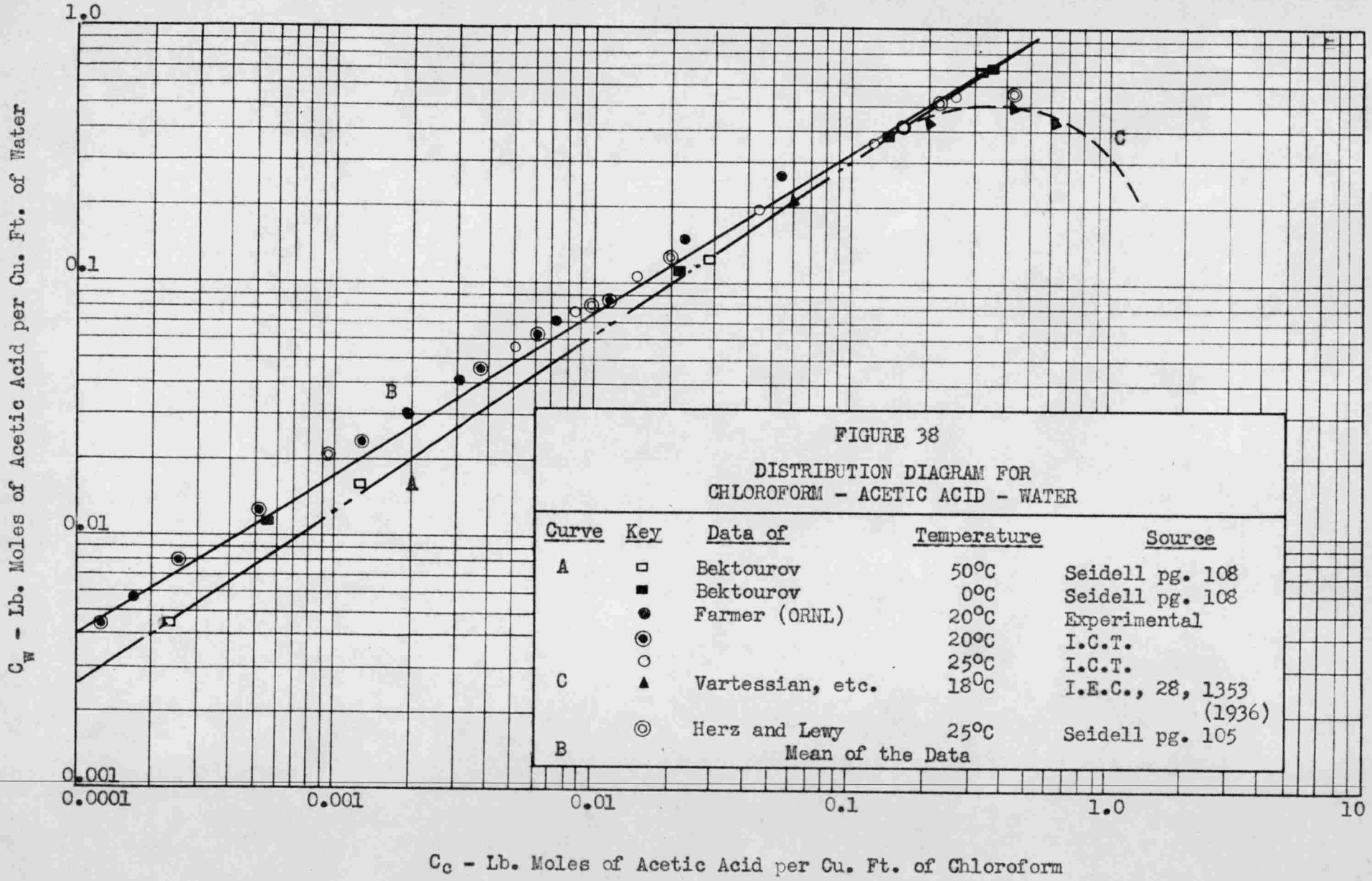


FIGURE 38

DISTRIBUTION DIAGRAM FOR
CHLOROFORM - ACETIC ACID - WATER

Curve	Key	Data of	Temperature	Source
A	□	Bektourov	50°C	Seidell pg. 108
	■	Bektourov	0°C	Seidell pg. 108
	●	Farmer (ORNL)	20°C	Experimental
	◉		20°C	I.C.T.
	○		25°C	I.C.T.
C	▲	Vartessian, etc.	18°C	I.E.C., 28, 1353 (1936)
	◎	Herz and Lewy	25°C	Seidell pg. 105
B		Mean of the Data		

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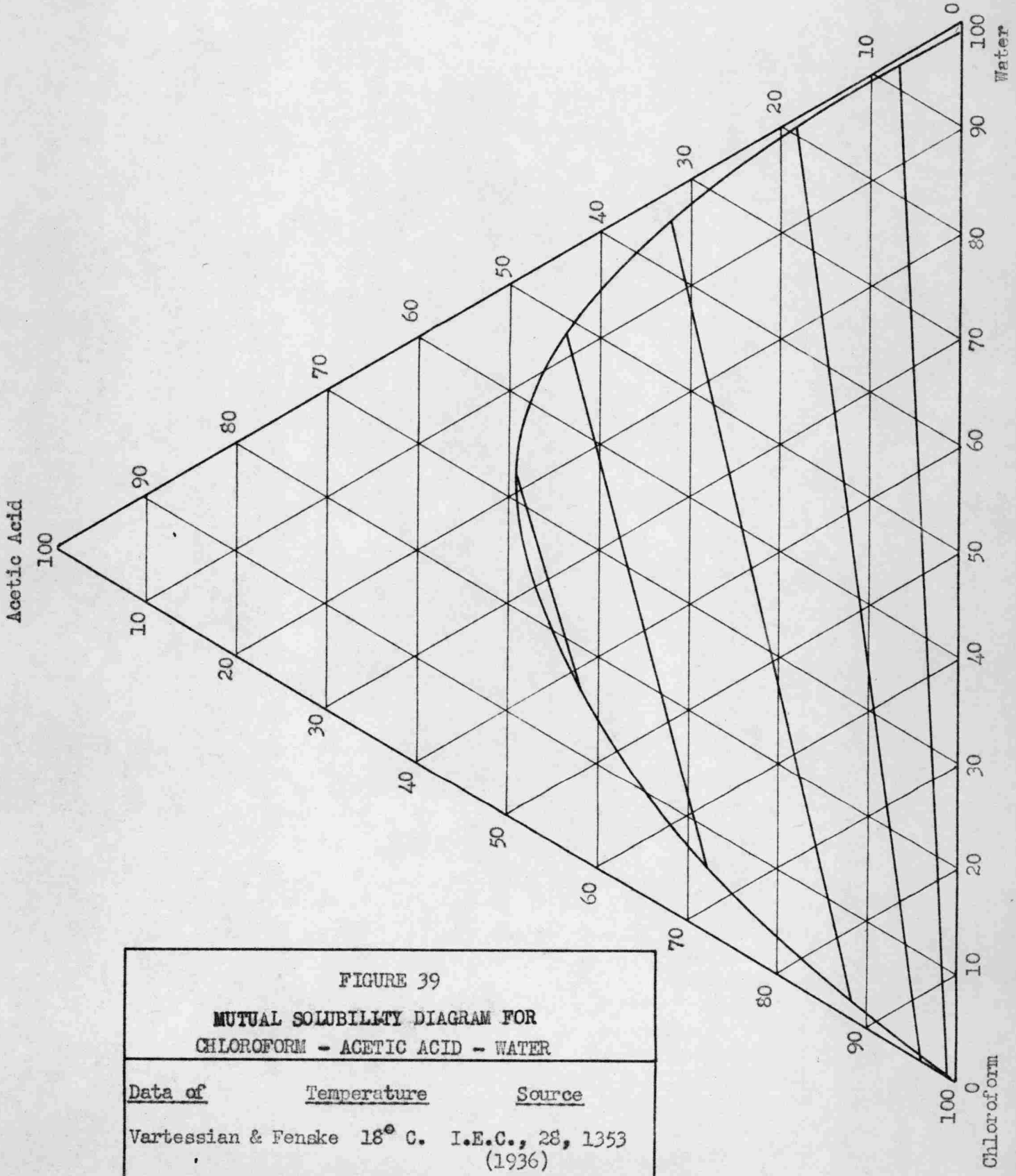

FIGURE 39

MUTUAL SOLUBILITY DIAGRAM FOR
CHLOROFORM - ACETIC ACID - WATER

Data of	Temperature	Source
Vartessian & Fenske	18° C.	I.E.C., 28, 1353 (1936)

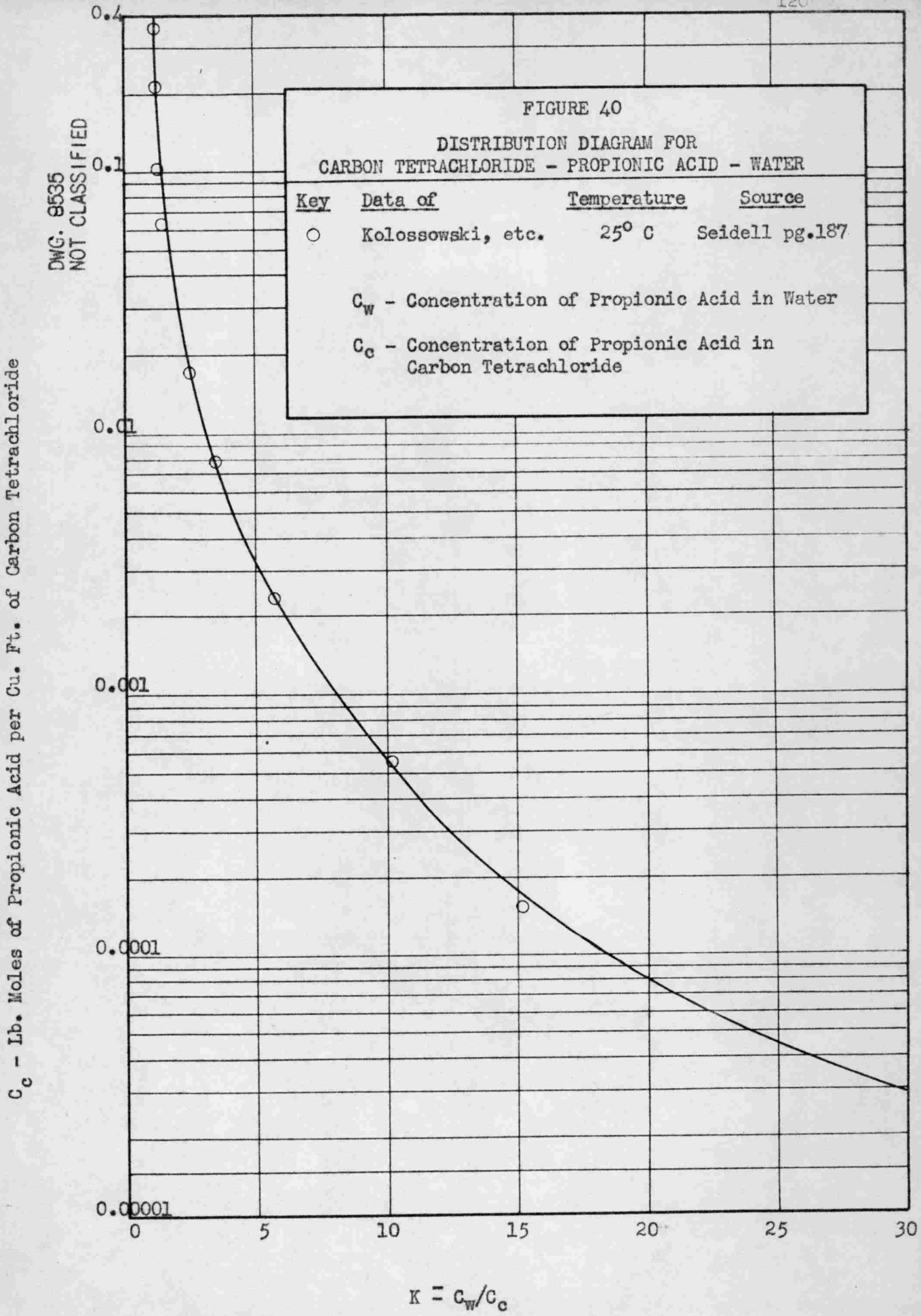

FIGURE 40

DISTRIBUTION DIAGRAM FOR
CARBON TETRACHLORIDE - PROPIONIC ACID - WATER

Key	Data of	Temperature	Source
○	Kolossowski, etc.	25° C	Seidell pg.187

C_w - Concentration of Propionic Acid in Water

C_c - Concentration of Propionic Acid in Carbon Tetrachloride

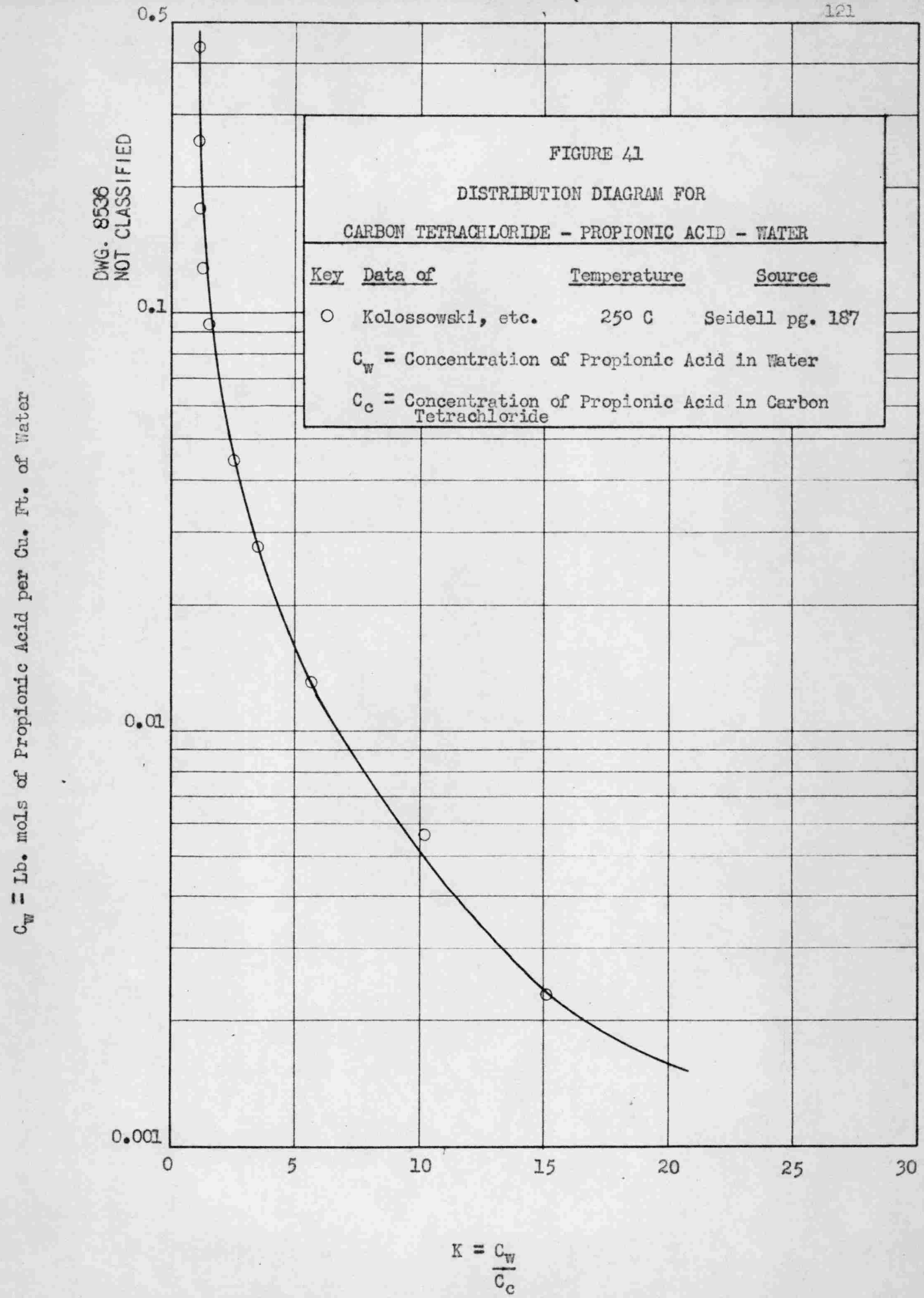
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FIGURE 41
DISTRIBUTION DIAGRAM FOR
CARBON TETRACHLORIDE - PROPIONIC ACID - WATER
Key Data of Temperature Source
○ Kolossowski, etc. 25° C Seidell pg. 187
C_w = Concentration of Propionic Acid in Water
C_c = Concentration of Propionic Acid in Carbon Tetrachloride
C_w = Lb. mols of Propionic Acid per Cu. Ft. of Water
0.5
0.1
0.01
0.001
0
5
10
15
20
25
30
$K = \frac{C_w}{C_c}$

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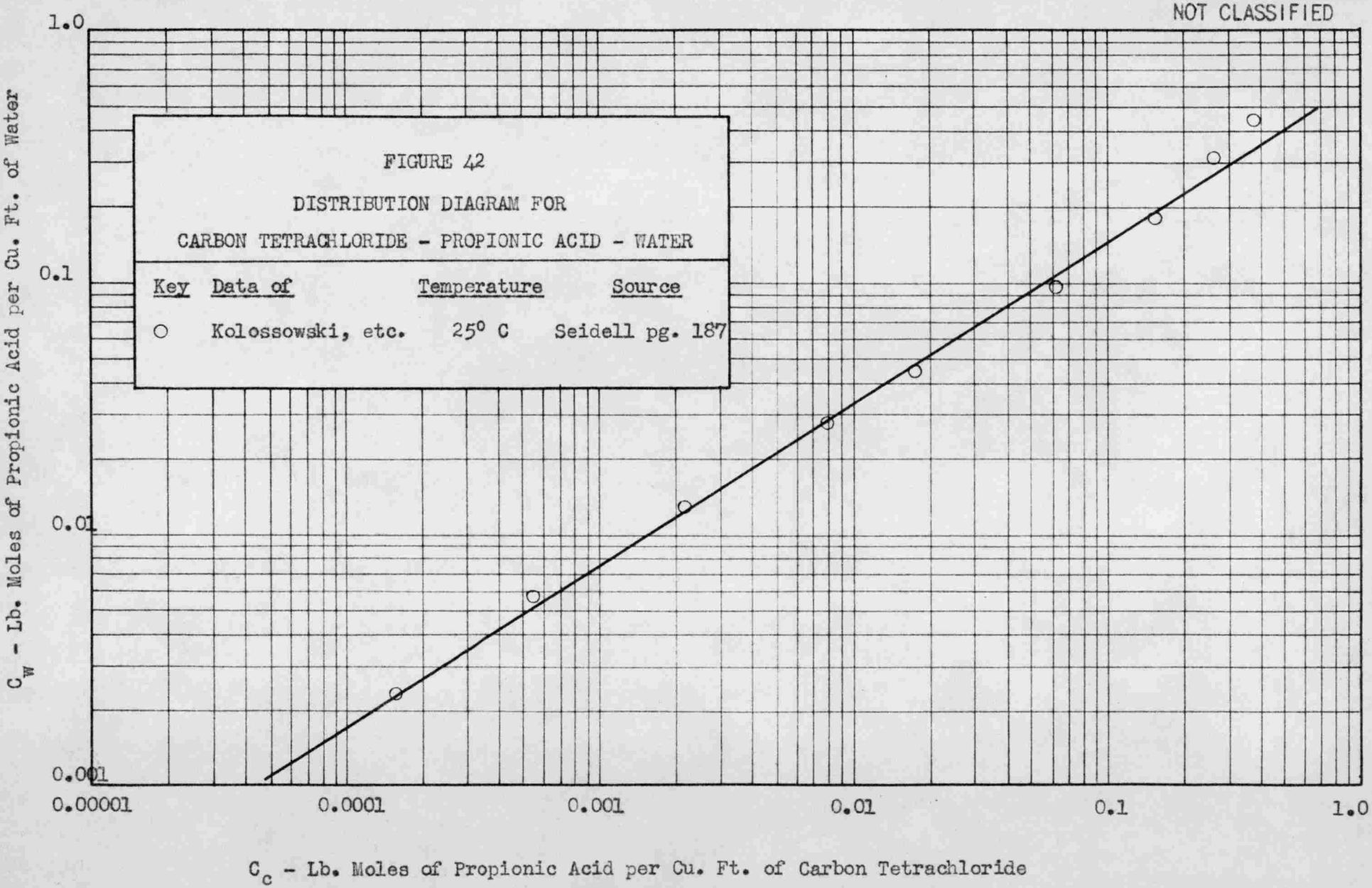

FIGURE 42

DISTRIBUTION DIAGRAM FOR

CARBON TETRACHLORIDE - PROPIONIC ACID - WATER

Key	Data of	Temperature	Source
○	Kolossowski, etc.	25° C	Seidell pg. 187

APPENDIX B

SOLVENT AND SOLUTE PHYSICAL PROPERTIES

The physical properties of the solvents used as the dispersed phase, are among the important variables upon which it is felt the mass transfer film coefficients depend. The three most important physical properties are the viscosity, density, and interfacial tension. In order to obtain accurate values of these properties it was necessary to measure experimentally them under the same conditions as employed in the solvent extraction runs.

The density was measured at 25° C. by means of a Westphal balance such as is described in Daniels, Mathews and Williams[10] on Page 321. This instrument uses a chainomatic type balance and depends upon Archimedes principle that the bouyancy is proportional to the weight of liquid displaced.

The relative viscosities of the solvents employed were measured by means of an Ostwald viscometer and density measurements described above. The absolute viscosities were then obtained by comparing the relative viscosities of the solvents to demineralized water as a reference standard. The apparatus and procedure are fulling described on Page 35 of Daniels, Mathews and Williams.[10]

In order to obtain rapid measurements of the interfacial tension a Cenco-Cu-Nouy Interfacial Tensiometer was employed. The interfacial

tension of the solvents against pure water were measured and are tabulated in Table XIX along with the corresponding values for varying concentrations of acetic acid in the solvent phase. In most cases the interfacial tension was found to be lowered by the presence of acetic acid. Bulletin 101 of the Central Scientific Company[6] gives complete details of operation and methods of determining and correcting interfacial tension values.

The accuracy of both the density and viscosity measurements is 99% or better. However, in the case of the interfacial tension considerable difficulty was experienced. The measurements of the interfacial tension of the pure solvents against water gave an accuracy of approximately 90%. When wetting agents or solutes are present in either phase the values obtained were in some cases inconsistent.

TABLE XIX

CHEMICAL PROPERTIES OF SOLUTIONS FOR SERIES J
RUNS IN WHICH INTERFACIAL TENSION IS VARIED

Sample	Run No.	Liquid	Acetic Acid Conc. lb.moles/cu.ft.	Additives % by Vol.	Temp. °C.	Density grs./ml.	I.T. versus Liquid	I.T. dynes/cm.
1		D.M. Water			25	0.9967	Carbon Tetrachloride (0.01 lb.moles/cu.ft. of Acetic Acid)	33.2
		D.M. Water			20		Carbon Tetrachloride	45.6
		D.M. Water			25		Carbon Tetrachloride	35.7
		D.M. Water			30		Carbon Tetrachloride	29.4
2		D.M. Water		Tergitol #4 0.01	25	0.9668	Carbon Tetrachloride	14.4
3	J-2	D.M. Water	0.00017	Tergitol #4 0.1%	25	0.9667	Carbon Tetrachloride	13.5
4		Carbon Tech.	0.01		25	1.5796	D.M. Water	33.2
5		D.M. Water		Tergitol #4 0.01%	25	0.9970	Carbon Tetrachloride	32.3
6	J-3	D.M. Water	0.0002	Tergitol #4 0.01%	25	0.9968	Carbon Tetrachloride	29.5
7		D.M. Water		Tergitol #4 0.33%	25	0.9963	Carbon Tetrachloride	4.3
8	J-4	D.M. Water	0.00016	Tergitol #4 0.33%	25	0.9970	Carbon Tetrachloride	5.7
9		D.M. Water		Tergitol #4 0.1%	25	0.9965	Carbon Tetrachloride	2.0
10		D.M. Water		Tergitol #7 0.33%	25	0.9963	Carbon Tetrachloride	8.5
11		D.M. Water		Tergitol #7 0.3%	25	0.9962	Carbon Tetrachloride	0.0

TABLE XIX (Con't.)

CHEMICAL PROPERTIES OF SOLUTIONS FOR SERIES J
RUNS IN WHICH INTERFACIAL TENSION IS VARIED

Sample	Run No.	Liquid	Additives % by Vol.	Temp. °C	Density grs./ml.	Viscosity cp	I.T. Versus Liquid	I.T. dynes/cm.
1A		D.M. Water	Tergitol #7 1%	25	0.9972	0.8973	Carbon Tetrachloride	0.0
2A		D.M. Water	Tergitol #7 0.1%	25	0.9970	0.8903	Carbon Tetrachloride	0.0
3A		D.M. Water	Tergitol #7 0.33%	25	0.9971	0.8853	Carbon Tetrachloride	0.0
4A		D.M. Water	Tergitol #7 0.01%	25	0.9970	0.8869	Carbon Tetrachloride	12.5
5A		D.M. Water	Tergitol #7 0.33%	25	0.9970	0.8903	Carbon Tetrachloride	12.1
6A		D.M. Water	Tergitol #4 1%	25	0.9975	0.9027	Carbon Tetrachloride	0.0
7A		D.M. Water	Tergitol #4 0.1%	25	0.9969	0.8834	Carbon Tetrachloride	12.8
8A		D.M. Water	Tergitol #4 0.33%	25	0.9970	0.8937	Carbon Tetrachloride	0.0
9A		D.M. Water	Tergitol #4 0.01%	25	0.9970	0.8937	Carbon Tetrachloride	26.8
10A		D.M. Water	Tergitol #4 0.033%	25	0.9969	0.8885	Carbon Tetrachloride	24.7

TABLE XX

CHEMICAL PROPERTIES OF THE SOLVENTS AS A FUNCTION OF VARYING ACETIC ACID CONCENTRATIONS

Sample	Solvent	Acetic Acid Conc. lb.moles/cu.ft.	Temp. °C.	Density grs./ml.	Viscosity cp.	I.T. versus D.M. Water dynes/cm.
1	Chloroform	0.01	25	1.4693	0.6244	24.2
19	Chloroform	0.005	25	1.4708	0.6226	27.5
13	Chloroform	0.000	25	1.4725	0.6059	33.9
2	Benzene	0.01	25	0.8734	0.6101	15.9
18	Benzene	0.005	25	0.8738	0.6134	18.2
11	Benzene	0.000	25	0.8732	0.6098	34.0
3	Toluene	0.01	25	0.8625	0.5654	17.0
17	Toluene	0.005	25	0.8611	0.5615	22.3
12	Toluene	0.000	25	0.8595	0.5606	26.6
4	Isopropyl Ether	0.01	25	0.7262	0.3624	19.4
20	Isopropyl Ether	0.005	25	0.7226	0.3556	17.1
10	Isopropyl Ether	0.000	25	0.7212	0.3485	17.3
5	Methyl Isobutyl Ketone	0.01	25	0.7790	0.5636	12.3
21	Methyl Isobutyl Ketone	0.005	25	0.7965	0.5633	11.9
14	Methyl Isobutyl Ketone	0.000	25	0.7959	0.5585	10.2
6	Carbon Tetrachloride	0.01	25	1.5813	0.9333	22.2
16	Carbon Tetrachloride	0.005	25	1.5822	0.9637	27.8
15	Carbon Tetrachloride	0.000	25	1.5860	0.9397	35.7

APPENDIX C

HYDRODYNAMIC RELATIONS

From an analysis of the experimental data it is obvious that the velocity of the dispersed drops and their hydrodynamic behavior is of great importance in determining the mass transfer behavior in the turbulent flow regime. In order to study the hydrodynamics of the liquid drops their shape was assumed to be that of a perfect sphere. Drop deformations were observed to take place in the turbulent regime. However, these deviations can be correlated by means of relations derived for the fluid mechanics of spheres.

A liquid drop when released in a fluid medium is subject to the forces of bouyancy, gravity, and, if in motion, also to drag. When these three forces are in equilibrium the motion of the drop is at a fixed velocity.

The bouyant force on the drop is equal to the weight of fluid displaced by the spherical drop. Thus for a true sphere:

$$F_{B_o} = \pi d^3 \rho g/6$$

where F_{B_o} - the force due to bouyancy

d - the drop diameter

ρ - the density of the fluid medium

Similarly, the force due to gravity is expressed by:

$$F_g = \pi d^3 \rho_s g/6$$

where F_g = the force of gravity

ρ_s - the density of the liquid within the drop

The analysis of the drag of a continuous fluid upon submerged spheres has been the object of many studies. The results of these experiments indicate that the overall drag on a particle such as a liquid drop is given by an expression of the form:

$$F_D = \frac{c}{2} \rho V^2 \ A$$

where F_D - the drag force

c - the drag coefficient, dimensionless

V - interfacial velocity

A - the projected area of the particle normal to the flow

When the liquid drop is completely immersed, dimensional analysis of the hydrodynamic behavior leads to a value of c which is a function of the Reynolds number. In Figure 43 is reproduced a plot of c versus Reynolds number for spheres from Page 1852 of Perry's.[20] This curve represents a mean of many experimental determinations. It is possible by analyzing this curve, to arrive at an expression as a function of Reynolds number for each of the three regimes of flow-viscous, transition and turbulent. In the viscous regime (Stokes law) which occurs at Reynolds number up to 2, the drag coefficient is expressed by $c \simeq 24/Re$. In the transitional or intermediate flow regime which extends to a Reynolds number of 300, the drag coefficient is equal to $18.5/Re^{0.6}$. Finally, in the turbulent flow regime (Newton's Law)

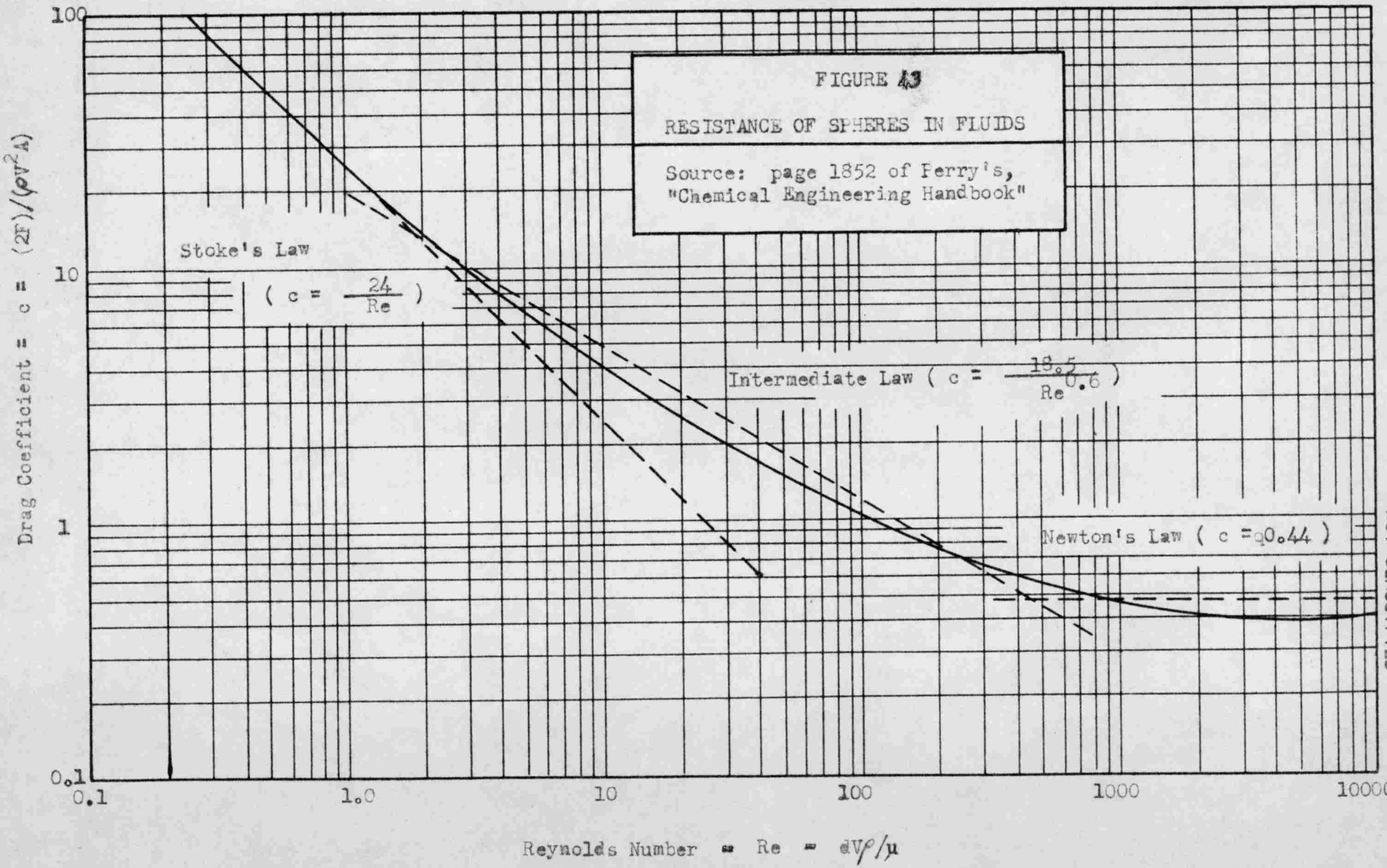

FIGURE 43

RESISTANCE OF SPHERES IN FLUIDS

Source: page 1852 of Perry's, "Chemical Engineering Handbook"

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which exists above a Reynolds number of 300, the drag coefficient is a constant and has a value of $c = 0.44$ for perfect spheres.

We can now balance our three forces in accordance with the direction of flow and densities of the dispersed and continuous phases. Thus, for the dispersion of a heavy liquid, as drops in a continuous phase of lower density, our balance of forces is given by the expression:

$$F_g = F_D + F_{B_o}$$

$$\pi d^3 \rho_s g/6 = \frac{c}{2} \rho V^2 A + \pi d^3 \rho g/6$$

The projected area of perfect sphere is a circle. Hence by substituting for A the expression $\pi d^2/4$ and rearranging the terms it is possible to derive a general expression for velocity as a function of drop diameter and c.

$$V^2 = \frac{4}{3} \frac{dg}{c} \frac{\rho_s - \rho}{\rho}$$

By replacing our drag coefficient by the experimentally determined expressions as a function of the Reynolds number, it is possible to express the velocity as an explicit function of the drop diameter over each of the three regimes of flow. In addition the velocity can be eliminated and the maximum or critical diameter determined for the upper limit of the Reynolds number over the regime to which each law applies.

Over the viscous regime of flow where Stokes law applies, velocity can be expressed as a function of sphere diameter by:

$$V = 54.5 \quad \frac{d^2}{\mu} \quad (\rho_s - \rho)$$

the maximum or critical diameter up to which this expression is valid is given by:

$$d_K = 0.333 \; \mu^{2/3} \left[\frac{1}{\rho(\rho_s - \rho)} \right]^{1/3}$$

This following formulas can similarly be derived for the transitional or intermediate flow regime:

$$V = \left[70.65 \quad \frac{d^{1.6}}{\mu^{0.6}} \quad \frac{\rho_s - \rho}{\rho^{0.4}} \right]^{0.715}$$

$$d_K = \quad 7.45 \; \mu^{2/3} \left[\frac{1}{\rho(\rho_s - \rho)} \right]^{1/3}$$

Most of the experimental data has been obtained in the turbulent regime of flow which extends above a Reynolds number of 300. Over this regime Newton's law applies, and the velocity can be expressed as a function of sphere diameter by:

$$V = 1.74 \quad \left[dg \quad \frac{\rho_s - \rho}{\rho} \right]^{1/2}$$

When dispersing drops of a low density liquid through a heavier continuous phase the drops tend to rise rather than fall as in the previous case. By rebalancing our forces, similar relations can be derived. Thus:

$$F_{B_o} = F_D + F_g$$

$$\pi d^3 \rho g/6 = \frac{c}{2} \rho V^2 \; A + \pi d^3 \rho_s g/6$$

It is possible to again replace the projected area by that of a sphere $\pi d^2/4$ and solve for an expression between the velocity, drop diameter and drag coefficient.

$$V^2 = \frac{4}{3}\,\frac{dg}{c}\,\frac{\rho - \rho_s}{\rho}$$

Our calculations can again be simplified by analyzing the data over the three regimes of flow and replacing c by its equivalent equation.

The solutions for the viscous regime are:

$$V = 54.5\,\frac{d^2}{\mu}\,(\rho - \rho_s)$$

$$d_K = 0.333\,\mu^{2/3}\left[\frac{1}{\rho(\rho - \rho_s)}\right]^{1/3}$$

The solutions for the transitional regime are:

$$V = \left[70.65\,\frac{d^{1.6}}{\mu^{0.6}}\,\frac{\rho - \rho_s}{\rho^{0.4}}\right]^{0.715}$$

$$d_K = 7.45\,\mu^{2/3}\left[\frac{1}{\rho(\rho_s - \rho)}\right]^{1/3}$$

Finally, for the turbulent regime of flow the velocity is given by the following expression:

$$V = 1.74\left[dg\,\frac{\rho - \rho_s}{\rho}\right]^{1/2}$$

Velocity as a function of drop diameter, assuming the drops to be perfect spheres was computed over the viscous, transitional and turbulent region of flow for the liquid systems used in the experiments. The results are plotted for the solvents - carbon tetrachloride, benzene,

toluene, chloroform, methyl isobutyl ketone and isopropyl ether dispersed as liquid drops through a continuous water phase. The velocities, obtained experimentally by measuring the total time of fall, are plotted along with the theoretical results in Figures 44 through 50.

In order to determine the accuracy of measuring the average experimental velocity by the time of fall it is necessary to determine the rate of acceleration. The theoretical expressions require an infinite time to reach a terminal constant velocity. However, the time required to reach some fraction f of the terminal velocity is a finite quantity and by choosing f sufficiently close to 1.0 will give an acceleration time which is adequate to evaluate the error in our measurement.

The forces acting on a liquid sphere of a low density fluid, dispersed in a heavier medium, can be balanced just as in determining the velocity. Thus:

$$F = F_g - F_D - F_{B_o}$$

The forces are then replaced by their equivalent expressions involving the dimensions and properties of the fluids. When common terms are cancelled the following expression results:

$$\frac{dV}{d\theta} = g \frac{(\rho_s - \rho)}{\rho^s} - \frac{3}{4} c \frac{\rho}{d \rho_s} V^2$$

The analysis is simplified by replacing the drag coefficient by its equivalent value over the three regimes of flow. Over the viscous regime

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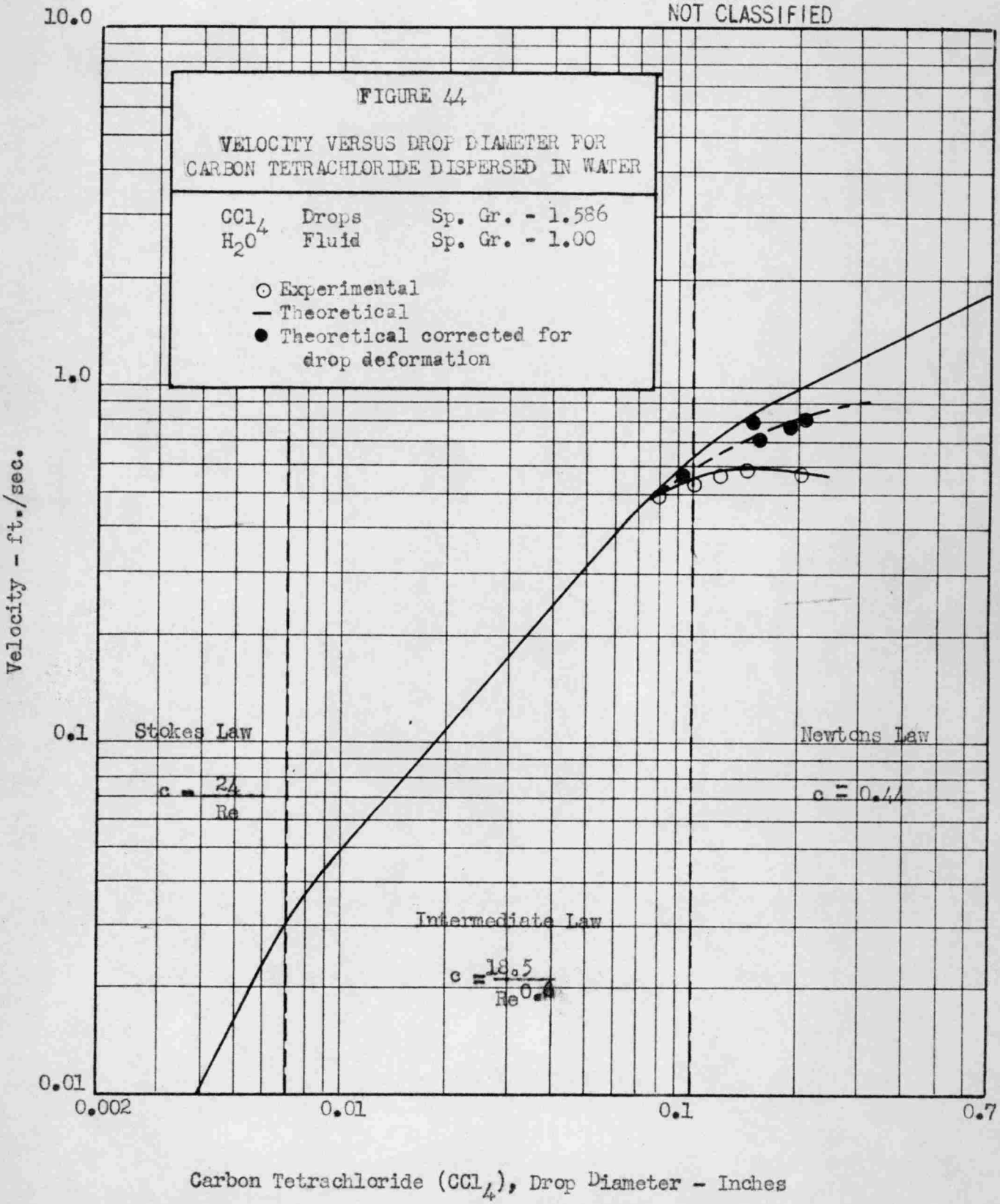

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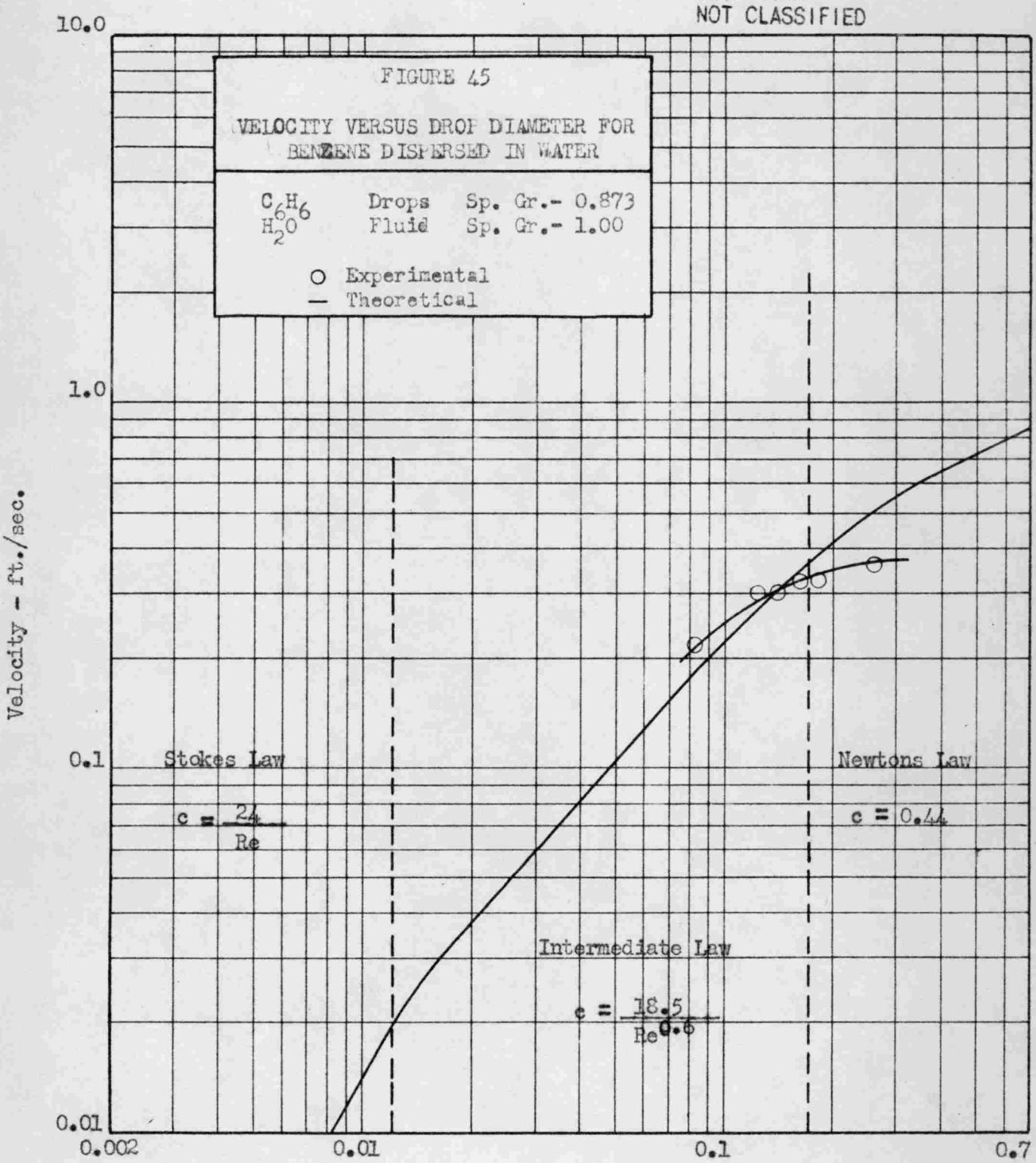

DWG. 8541
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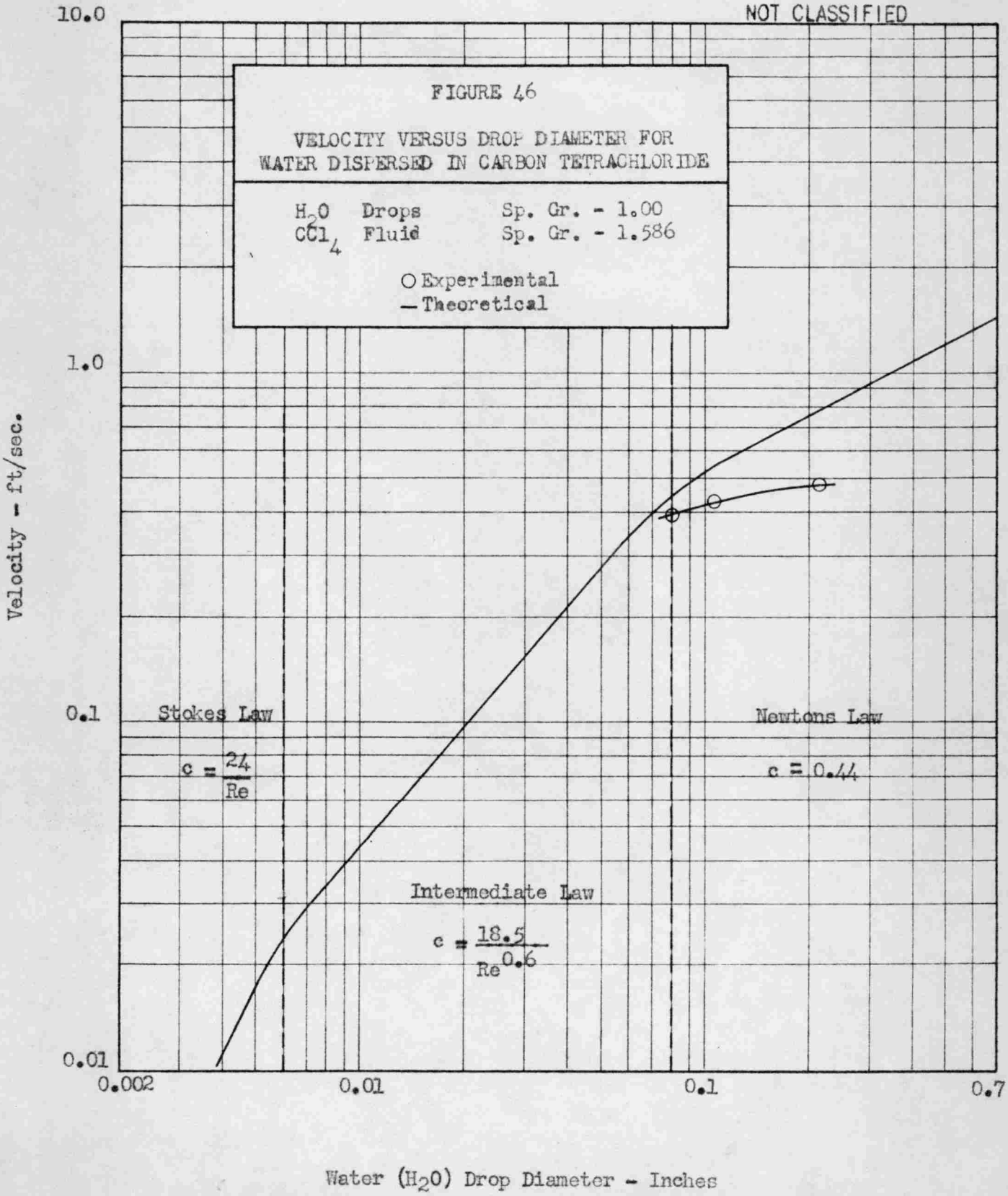

FIGURE 46

VELOCITY VERSUS DROP DIAMETER FOR WATER DISPERSED IN CARBON TETRACHLORIDE

H_2O	Drops	Sp. Gr. - 1.00
CCl_4	Fluid	Sp. Gr. - 1.586

○ Experimental
— Theoretical

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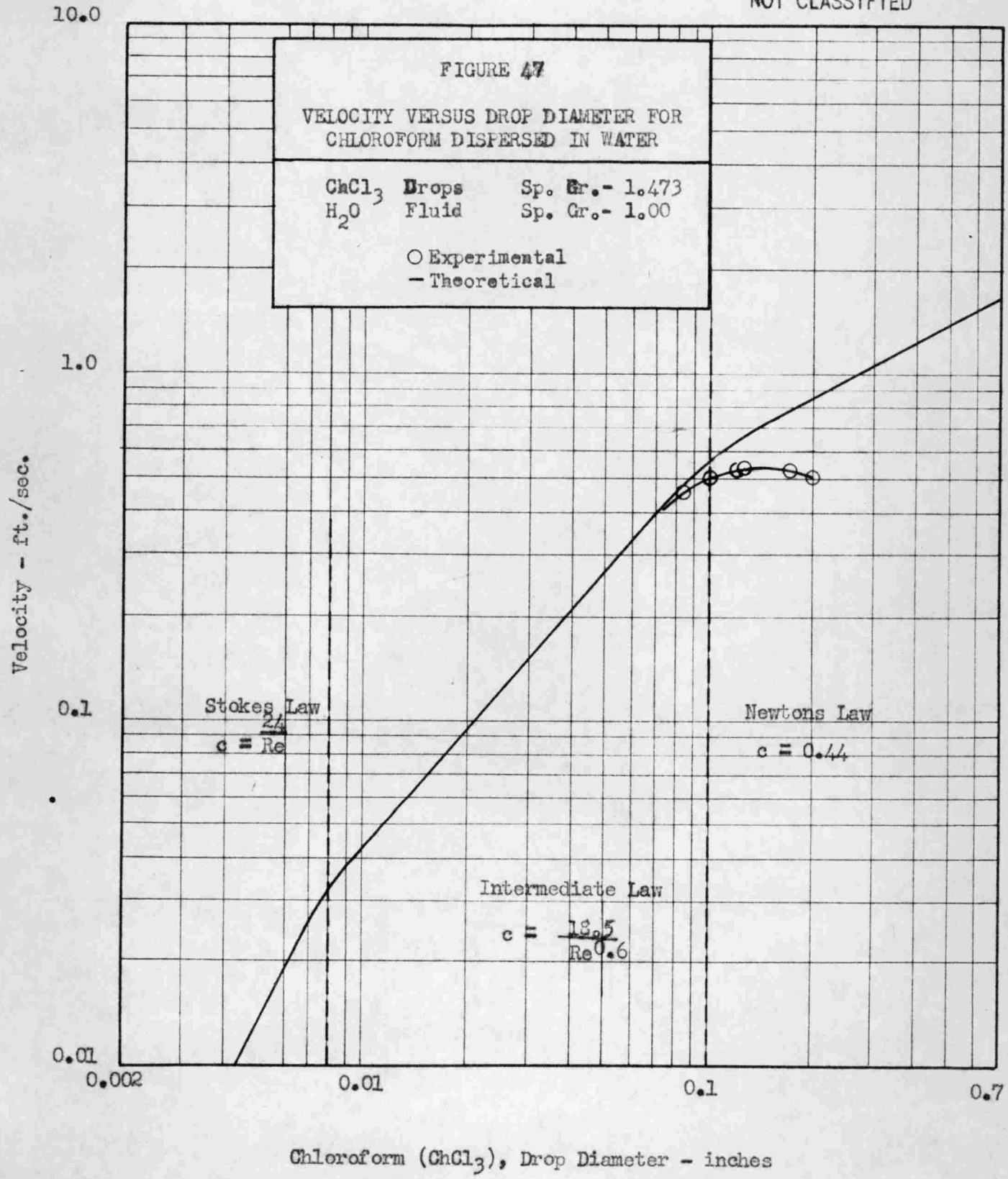

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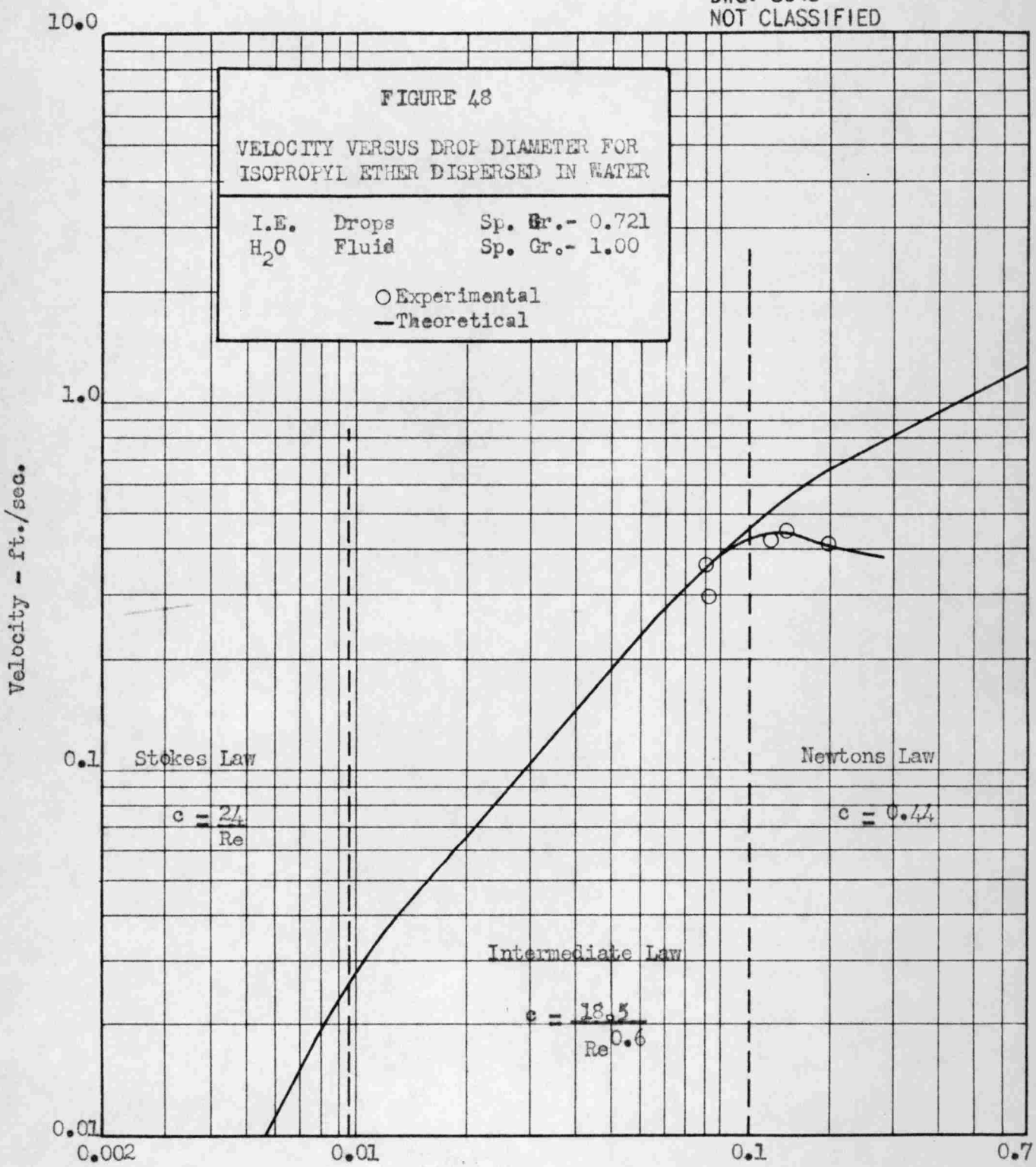

Isopropyl Ether (I.E.), Drop Diameter - Inches

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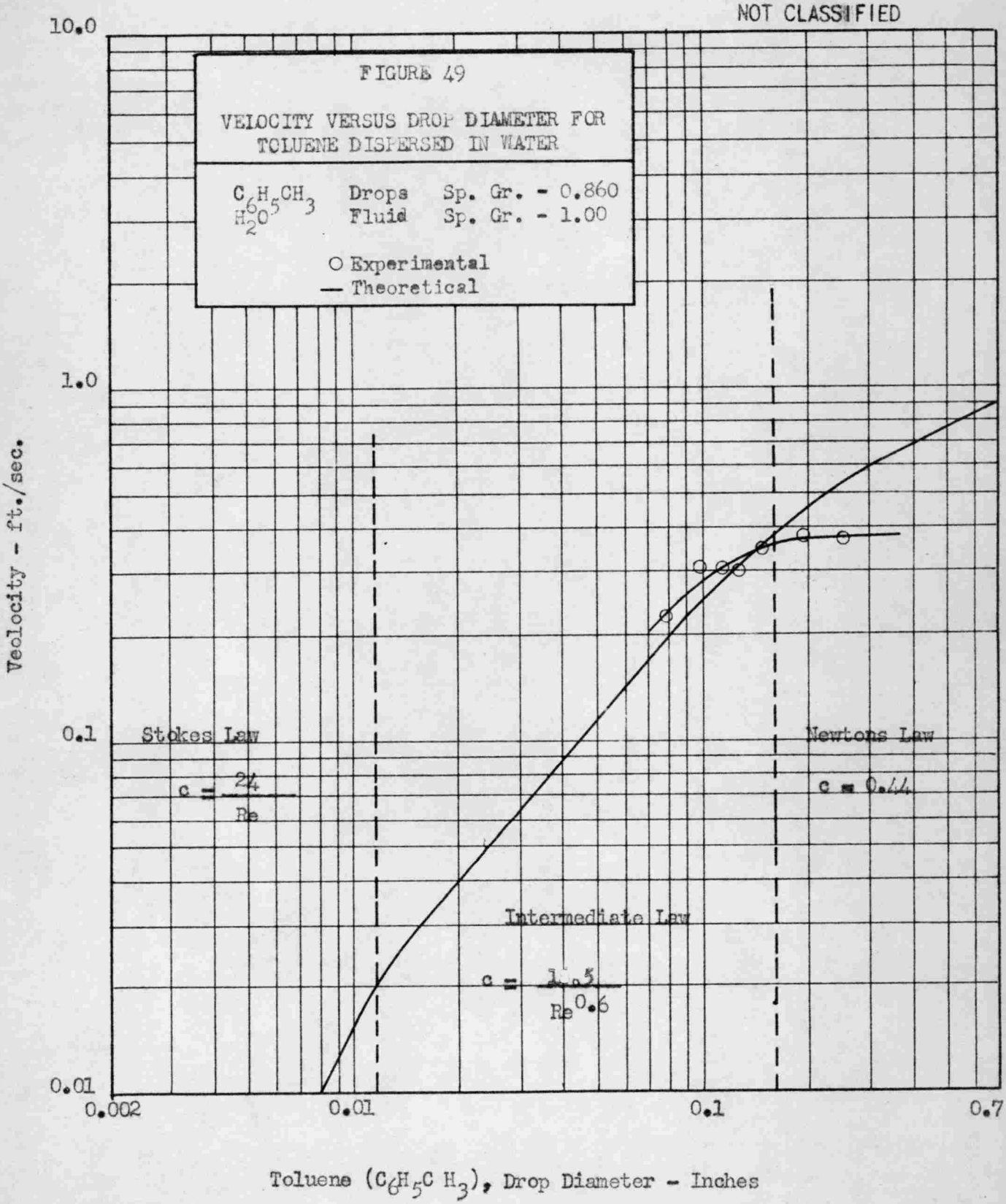

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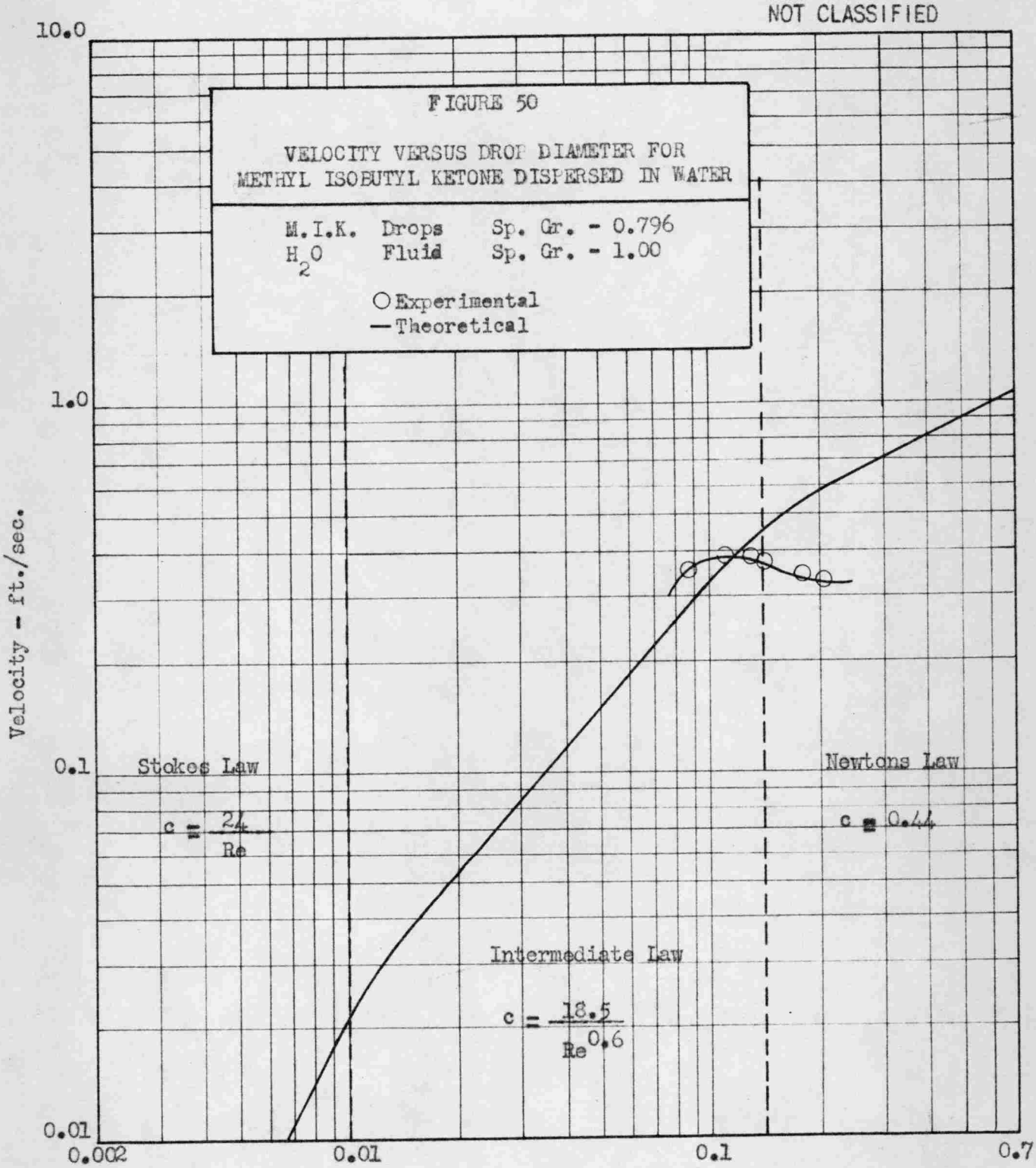

of flow for the above situation, the time required to accelerate to fraction f of terminal velocity is given by:

$$\theta_f = \frac{-\ln(1-f)}{18} \frac{d^2 \rho_s}{\mu}$$

In the transitional regime of flow the acceleration time is:

$$\theta_f = \frac{-\ln(1-f)}{19.4} \frac{d^{1.6} \rho_s}{\mu^{0.6} \rho^{0.4}}$$

Finally, for the turbulent regime of flow the time required to accelerate to fraction f of terminal velocity is given by:

$$\theta_f = \frac{1}{2\left[0.33\, g \frac{\rho(\rho_s - \rho 2)}{d \rho_s}\right]^{1/2}} \ln \left| \frac{\sqrt{\frac{g \rho_s \, 0 \rho}{\rho_s}} + \sqrt{\frac{0.33 \rho V}{d \rho_s}}}{\sqrt{\frac{g \rho_s - \rho}{\rho_s}} - \sqrt{\frac{0.33 \rho V}{d \rho_s}}} \right| \Bigg|_0^{fV}$$

Expressions for the acceleration time, similar to those above, can be derived for dispersing a low density solvent in a heavier continuous phase. The only difference between the resulting expressions for time θ_f and those above, is that $\rho_s - \rho$ is replaced by $\rho - \rho_s$.

The time required to accelerate to fraction f of terminal velocity is computed over a range of drop diameters for the three regimes of flow for chloroform and benzene dispersed in water. The results of this calculation are presented as a plot of acceleration time θ_f versus drop diameter for 95% of terminal velocity in Figure 51. In neither case is the time required for acceleration greater than 0.2 seconds. Since the time

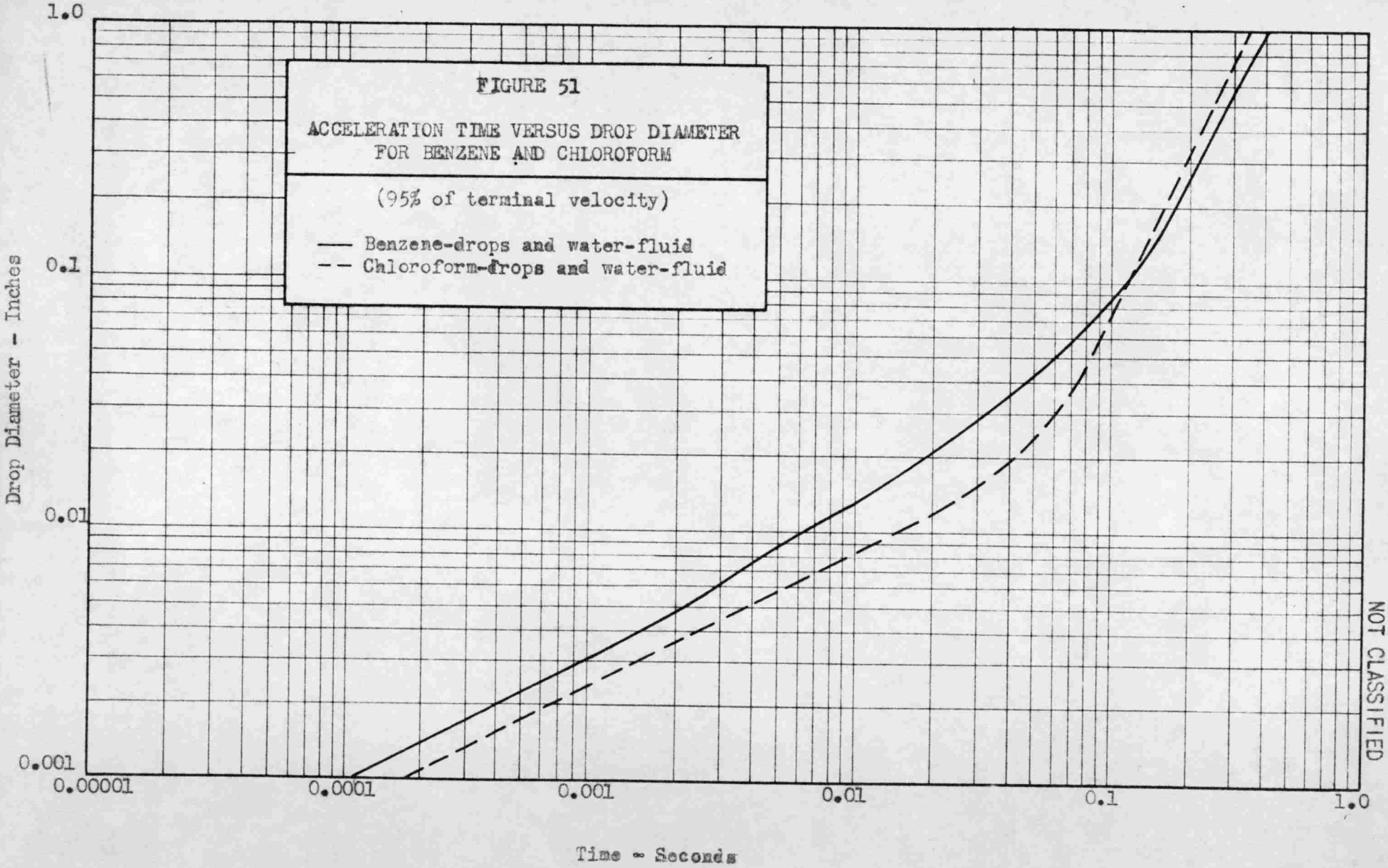

FIGURE 51

ACCELERATION TIME VERSUS DROP DIAMETER FOR BENZENE AND CHLOROFORM

DWG. 8546
NOT CLASSIFIED

of fall in a four foot column is of the order on the average of 10 seconds, the error involved in computing the velocity from the time of fall is less than 2%.

From the plots presented in Figures 44 through 50 it is obvious that the formulas for perfect spheres do not fit our experimental results above a Reynolds number of about 300 to 350. From experimental observations the liquid drops are seen to deform into ellipsoids of revolution around the major or horizontal axis at diameters which give rise to Reynolds numbers above 300 to 350. This observed deformation accounts in part for the deviation of the experimental results from that predicted for perfect spheres.

From the data presented in the drop deformation study it was possible to determine the true cause of the deviations in the velocity versus diameter plots. From measurements taken from the pictures it was possible to compute the true projected area in the direction of flow and also the diameter of the sphere whose volume is equivalent to that of the actual ellipsoid of revolution photographed. Using the projected area so computed, the corrected theoretical velocity was determined for a drag coefficient of 0.44 and plotted in Figure 44 along with the theoretical result based on perfect spheres and that measured experimentally. Although the corrected theoretical results using the true projected area come close to fitting the experimental results, the resulting velocities are still too large. This is accounted for by the fact that

the drag coefficient is not 0.44 but is numerically larger and in addition is a function of Reynolds number.

The true value of our drag coefficient can be computed by determining that value necessary to lower our corrected theoretical result to the observed value of the velocity. In the turbulent region of flow our drag coefficient by Newton's law is given by:

$$c = \frac{\pi d^3}{3 V^2} \frac{g}{A} \quad \frac{\rho_s - \rho}{\rho}$$

Drag coefficients were computed for several drop diameters in the turbulent regime of flow using the experimentally measured velocity, the true projected area, and the diameter of a sphere of equivalent volume taken from the data in the drop deformation study. The results are plotted as c versus Reynolds number in Figure 52 along with the corresponding value of c for perfect spheres. In addition the drag coefficient for ellipsoids of revolution whose major to minor axis ratio is 4 to 3 are shown. This latter data is taken from Binder[4] and gives a value of 0.60 for c for ellipsoids.

No information was to be found in the literature on drop deformation and the hydrodynamics of liquids dispersed as drops in a fluid medium. However, the rate of rise of gas bubbles in liquids, which is analogous to this, has been investigated by O'Brien and Gosline,[19] Miyagi[18] and many others. The tendency for the velocity of liquid drops to approach a maximum value and then decrease which was observed here was also found to take place in the case of gas bubbles rising in liquids.

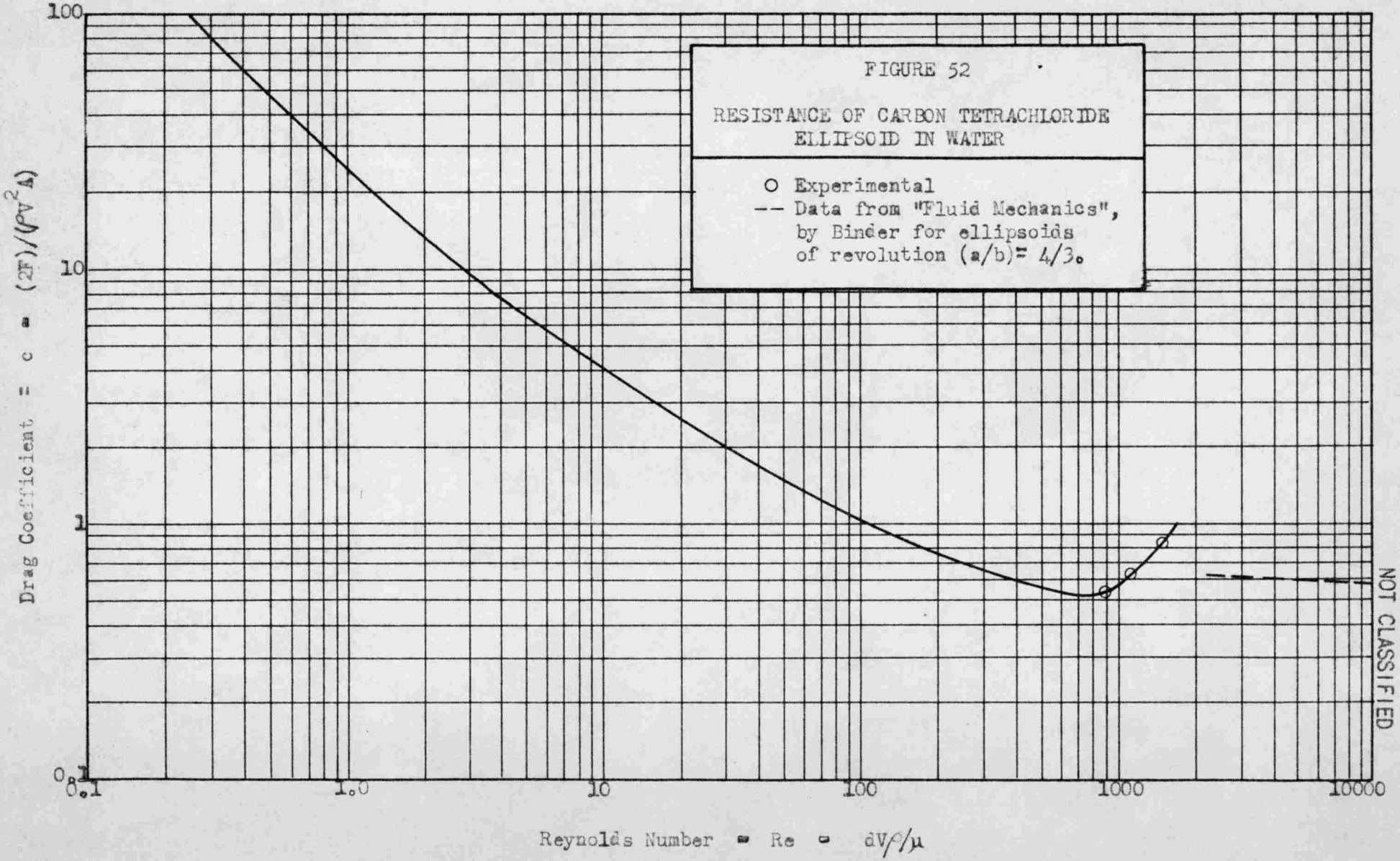

FIGURE 52

RESISTANCE OF CARBON TETRACHLORIDE ELLIPSOID IN WATER

○ Experimental
-- Data from "Fluid Mechanics", by Binder for ellipsoids of revolution (a/b) = 4/3.

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NOT CLASSIFIED

O'Brien and Gosline[19] observed this in studying air lifts. They attributed the deviation to the fact that small bubbles are controlled by viscosity and surface tension, whereas large bubbles are controlled by turbulence. Miyagi[18] found the deformation to be non-existant when the bubbles are small since the surface tension is sufficient to hold the bubbles in a spherical shape at small diameters, but as the diameter increases the force of surface tension decreases while the drag resistance tends to increase thus flattening the bubbles out into the shape of oblate spheroids.

O'Brien and Gosline[19] employed dimensional analysis to determine the relationship between the velocity and the fluid properties. They expressed the velocity as a function of:

$$V = f_1(g, \rho_o, \rho_1, I.T., r, d_c, \mu_o, \mu_1, P_a, P, c_p \text{ and } c_v)$$

This gave rise to groups relating the variables as follows:

$$f_2\left(\frac{g\,r}{V^2}, \frac{2\,V\,r\,\rho_1}{\mu_i}, \frac{\rho_1 r\ V^2}{I.T.}, \frac{2\,r}{d_c}, \frac{Pa}{P}, \frac{\nu_1}{\nu_o}, c_p, c_v\right)$$

If this group of dimensionless numbers is applied to the problem of liquid drops dispersed in a fluid medium, the last four of the above groups are insignificant. O'Brien and Gosline[19] included the ratio Pa/P besides the first four since pressure is important in dealing with gases. They plotted their results in terms of a drag coefficient ψ, defined by $\psi = 8\ g\ r/(3V^2)$, versus Reynolds number. The drag coefficient for gas bubbles (ψ) is found to decrease and pass through a

minimum value at about a Reynolds number of 100. Above this value it tends to increase in value. This behavior is the same as that observed for liquid drops.

Since the hydrodynamics of liquid drops is analogous to that of gas bubbles, the results of dimensional analysis above are applicable to liquid drop systems. The hydrodynamic behavior of liquid drops is therefore a function of the drag coefficient, Reynolds number, the Weber number and the ratio of the drop radius to column diameter, with the possible inclusion of the group ν_1/ν_o. For the particular situation where one is dispersing small drops in a large column as here, the drop radius to column diameter ratio is unimportant. However, in dealing with packed columns this group would have to be taken into account. The use of the Weber capillary group in describing the speed of bubbles rising in a liquid is also reported by Klinkenberg[17] in his paper on dimensional analysis.

The overall mass transfer coefficient is found to follow the same changes as the velocity. Therefore, the overall mass transfer coefficient would be expected to be a function of several of the group above, such as the Reynolds number and Weber capillary group.

APPENDIX D

DROP DEFORMATION STUDY

Definite shape changes were observed to occur in the dispersed drops as they moved through the column. These deformations were such that the drops were flattened into ellipsoids of revolution whose horizontal axis is greater than the vertical. The degree of deformation tended to increase with drop size and resulted in the horizontal axis approaching a dimension twice the vertical. The larger the drops became the more unstable their nature. As a result, they tended to tilt and wobble as they moved up or down the column in a spiral path. This same type of motion and deformation was observed by Myaugi[18] and O'Brien and Gosline[19] in the case of air bubbles rising through liquids.

It was felt that extraction is dependent on the hydrodynamics of the dispersed drops and hence affected by such behavior as drop deformation. Therefore, in order to study the drop deformation, a series of pictures were made of drops covering the range of diameters employed experimentally. Since the double-walled curved column itself was not acceptable for taking drop pictures, a separate flat-sided lucite tank was set up. The feed tank and group of nozzles normally used for dispersing liquids heavier than water was set up over the lucite tank.

In order to obtain clear pictures a system had to be found, the two phases of which would have contrasting colors. The system carbon tetrachloride-iodine-water with iodine dissolved in the carbon tetrachloride fitted the requirements perfectly, since iodine is practically insoluble in water.

The carbon tetrachloride-iodine was dispersed by using various sizes of nozzles and photographs made of the drops as they fell by means of stroboscopic camera techniques. The resulting photographs are shown in Figures 53 - 58. In order to gauge the dimensions of the drops, a 6 inch scale marked in millimeters was fastened to the front wall of the lucite tank so that the ruler would appear along with the drop in the final picture.

From measurements taken from these pictures it was possible to compute the theoretical volume based on an ellipsoid of revolution about the major axis. The diameter of a sphere of equivalent volume was then calculated. By comparing the relative surface area of the two shapes, the magnitude of error involved in using correlations based on perfect spheres can be determined.

The volume of an ellipsoid of revolution around the major axis is given by the expression:

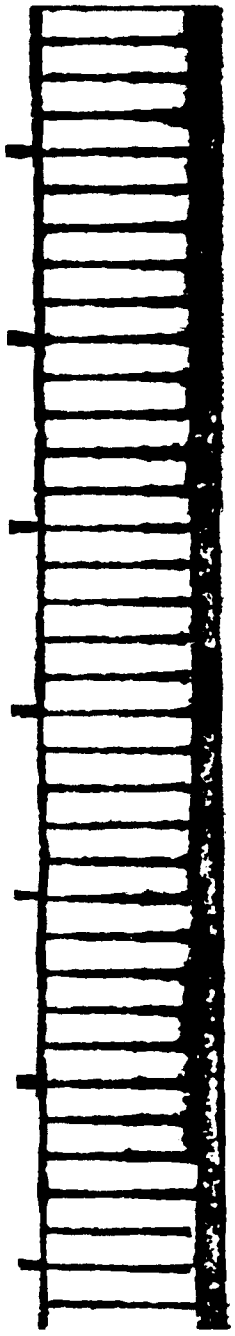
$$V' = 4\pi a^2 b/3$$

where a = the major axis

b = the minor axis

e = the eccentricity = c/a

$c = (a^2 - b^2)^{1/2}$



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NOT CLASSIFIED

FIG. 53

PHOTOGRAPH OF CARBON TETRACHLORIDE DROP FROM NOZZLE (Tube D.D. Med.)

Carbon Tetrachloride - Iodine (Dispersed) Water (Continuous)
Drop Diameter - 0.104 inches (Estimated on the basis of a true sphere.)
Scale - millimeters b/a - 0.908 (Deformation)

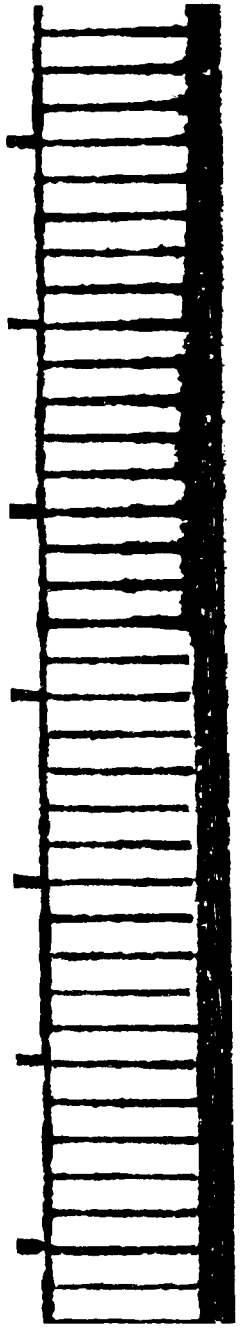

FIG. 54

PHOTOGRAPH OF CARBON TETRACHLORIDE DROP FROM NOZZLE (Cap. D.D. Small)

Carbon Tetrachloride - Iodine (Dispersed) Water (Continuous)
Drop Diameter - 0.105 inches (Estimated on the basis of a true sphere.)
Scale - millimeters b/a - 0.884 (Deformation)

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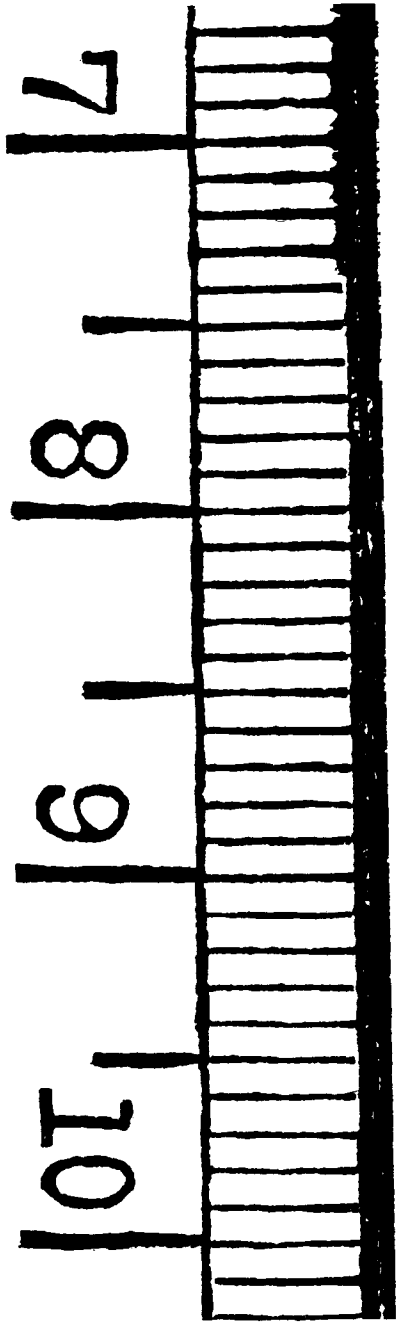

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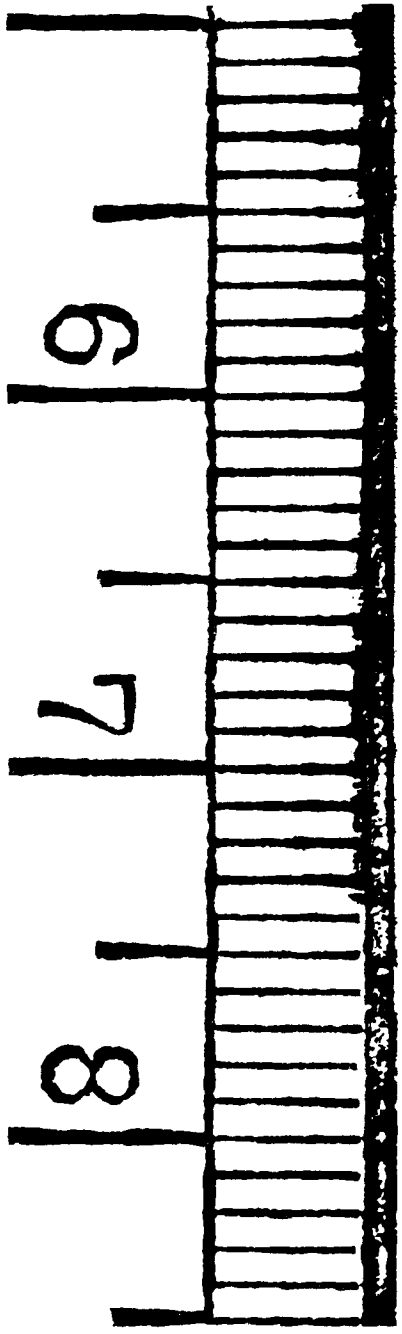
FIG. 55

PHOTOGRAPH OF CARBON TETRACHLORIDE DROP FROM NOZZLE (Tube D.D. Large)

Carbon Tetrachloride - Iodine (Dispersed) Water (Continuous)
Drop Diameter - 0.150 inches (Estimated on the basis of a true sphere.)
Scale - millimeters b/a - 0.88 (Deformation)



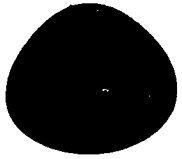


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FIG. 56

PHOTOGRAPH OF CARBON TETRACHLORIDE DROP FROM NOZZLE (7.5mm O.D., 2mm I.D.)

Carbon Tetrachloride - Iodine (Dispersed) Water (Continuous)
Drop Diameter - 0.154 inches (Estimated on the basis of a true sphere.)
Scale - millimeters b/a - 0.88 (Deformation)

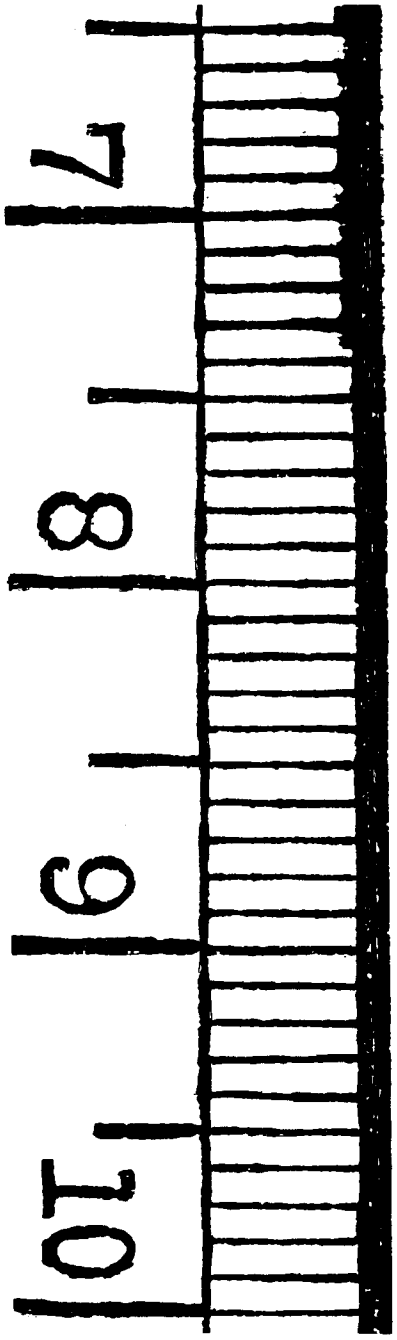

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FIG. 57

PHOTOGRAPH OF CARBON TETRACHLORIDE DROP FROM NOZZLE (7mm O.D., 5.5mm I.D.)

Carbon Tetrachloride - Iodine (Dispersed) Water (Continuous)
Drop Diameter - 0.190 inches (Estimated on the basis of a true sphere.)
Scale - millimeters b/a - 0.508 (Deformation)

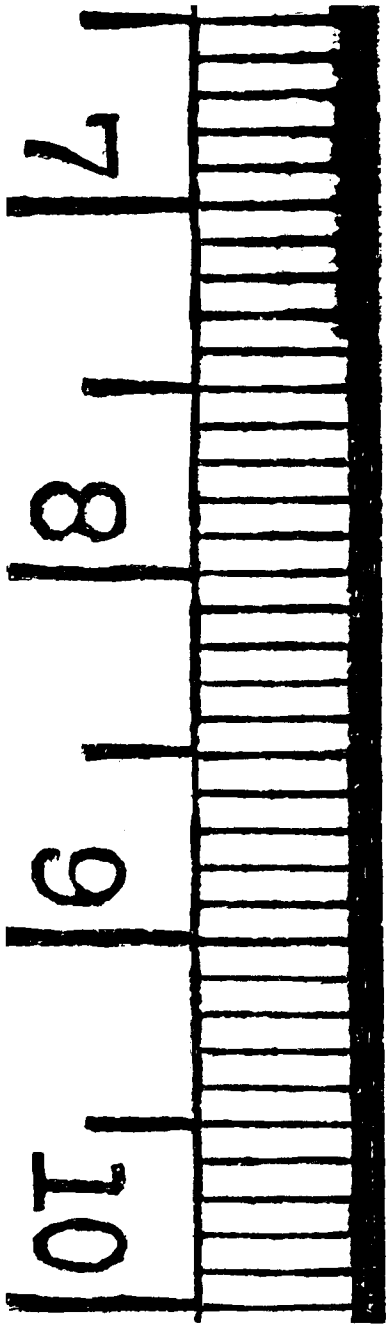

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FIG. 58

PHOTOGRAPH OF CARBON TETRACHLORIDE DROP FROM NOZZLE (8mm O.D., 6mm I.D.)

Carbon Tetrachloride - Iodine (Dispersed) Water (Continuous)
Drop Diameter - 0.205 inches (Estimated on the basis of a true sphere.)
Scale - millimeters b/a - 0.538 (Deformation)

The volume of a perfect sphere is given by the expression:

$$V' = \pi d^3/6 = 4 \pi r^3/3$$

The surface area of an ellipsoid of revolution is given by:

$$A = 2\pi a^2 + \pi (b^2/e) \ \ln \frac{1 + e}{1 - e}$$

while the area of the sphere is:

$$A = \pi d^2$$

Data from several of the photographs were taken and the volume and surface area of the ellipsoid of revolution and perfect sphere was computed. The error entailed in computing the overall mass transfer coefficient on the assumption that the drops are perfect spheres is very slight unless the diameter is at least 0.18 inches or greater and even then is less than 10% up to a diameter of 0.22 inches which is our maximum experimental range. A comparison for a range of drop diameters is presented in Tables XXI and XXII.

TABLE XXI

DROP DEFORMATION MEASUREMENTS FROM PHOTOGRAPHS

Figure No.	Nozzle	b Inches	a Inches	b/a	Drop Diameter (Est.) Inches
53	Tube D.D. Med.	0.100	0.110	0.908	0.104
54	Cap D.D. Small	0.102	0.115	0.883	0.105
55	Tube D.D. Large	0.139	0.158	0.88	0.150
56	7.5 mm. O.D. 2 mm. I.D.	0.159	0.1805	0.88	0.154
57	7 mm. O.D. 5.5 mm. I.D.	0.116	0.229	0.508	0.190
58	8 mm. O.D. 6 mm. I.D.	0.133	0.246	0.538	0.205

TABLE XXII

COMPARISON OF SURFACE AREA FOR ELLIPSOIDAL DROPS AND SPHERES OF EQUIVALENT VOLUME

Figure No.	Nozzle	Sphere $d_{calc.}$ Inches	d_{est} Inches	A_{sphere} (inches)^2	$A_{ellip.}$ (inches)^2	Error
54	Cap. D.D. Small	0.110	0.105	0.0385	0.0382	+ 0.5%
55	Tube D.D. Large	0.152	0.150	0.072	0.0723	- 0.4%
57	7 mm. O.D. 5.5 mm. I.D.	0.183	0.190	0.105	0.1146	- 8.4%

APPENDIX E

LAWS OF DIFFUSION

Diffusion Coefficients

In order to correlate the rate of mass transfer in a solvent extraction system it is necessary to evaluate the diffusion coefficients. The mechanism of diffusion in liquids is extremely complicated and as a result there are no exact correlations available. However, a number of empirical correlations have appeared in the literature which suffice to estimate the diffusion coefficients approximately.

A complete outline of the available theories for diffusion in liquids is presented earlier in this report. Although Arnold's[2] equation appears to be the most exact empirically, there is not sufficient data available to evaluate his equation for the systems involved here. Similarly, lack of information on the shape of the solute molecules prevents the use of Powell's[21] relation. The recent data presented by Wilke[30] although purely empirical appears to be the most workable and has a fair degree of accuracy, hence was employed here.

Wilke[30] has been fairly successful in correlating diffusion coefficients for solutes which are neither ionized or solvated by an expression given by:

$$D = \frac{T}{F\mu}$$

Where D - the diffusion coefficient
T - temperature
μ - viscosity
F - diffusion factor

The difference in behavior of different solute molecules in various solvents is taken into account in F. Thus F is expressed as a function of temperature, solute and solvent properties, and concentration. The diffusion factor is defined as a function of ϕ, "solvent factor", and is assumed to be constant for any particular solvent. In order to estimate diffusion coefficients a plot is constructed for F versus molal volume for line of constant ϕ where ϕ is set equal to 1 in the case of water as a basis. When sufficient data are not present on the particular solvent being used to evaluate ϕ it is recommended that a value of 0.9 be arbitrarily employed.

Using the above correlation, diffusion coefficients were computed for acetic acid diffusion in each of the six solvents employed and water. Where possible the exact value of ϕ was used. However, insufficient information made it necessary to use a ϕ of 0.9 in many cases. The results of the calculations are presented in Table XXIII.

The accuracy of the computed values was determined where possible by comparison with experimental values reported in the International Critical Tables. In the case of water the computed diffusion coefficient are almost exactly equal to that reported in the literature. The computed coefficient for benzene is 34.7×10^{-7} inches^2/second whereas the actual value appearing in the literature is 29.1×10^{-7} inches^2/second. This is slightly greater than the average deviation of 10% that Wilke obtained in comparing computed and experimental values for fourteen solvents.

TABLE XXIII

CALCULATED DIFFUSION COEFFICIENTS FOR ACETIC ACID IN VARIOUS SOLVENTS

Solvent	Acetic Acid Conc. lb.moles/cu.ft.	Temp. °C.	ϕ	F x 10^{-7}	μ cp.	D_i x 10^7 in.^{2}/sec.	n molecules
Water	0.01	25	1.0	2.56	1.0	18	1
Benzene	0.01	25	0.7	2.17	0.61	34.7	1.79
Carbon Tetrachloride	0.01	25	0.9	2.4	0.94	20.4	1.67
Chloroform	0.01	25	0.9	2.4	0.606	31.6	1.58
Isopropyl Ether	0.01	25	0.9	2.4	0.349	54.8	1
Methyl Isobutyl Ketone	0.01	25	0.9	2.4	0.559	34.2	1
Toluene	0.01	25	0.7	2.17	0.561	37.8	1.71

ratio of single to double molecules = $n/(2-n)$

The possibility of the computed diffusion coefficients being in error or of not fitting the experimental mechanism should not be overlooked. Solvation of acetic acid in polar organic liquids and association in non-polar organic liquids can add many complications in applying liquid diffusion coefficients arbitrarily. In explaining both molecular diffusion through a viscous fluid drop and molecular diffusion across a fictive boundary film the diffusion coefficient is extremely important.

In explaining the results and correlations for the overall mass transfer coefficient in the turbulent regime of flow, it has been assumed that mass transfer occurs by means of diffusion across a thin outer film at the boundary of the drop. In examining mass transfer across this film the rate of travel of both single molecules and double associated molecules are studied separately. Hence, the diffusion coefficient for single and double molecules should be used. However, there is insufficient information to determine the individual diffusion coefficients and hence an average diffusion coefficient based on the normal mixture of molecules existing at a specific concentration is used. This average diffusion coefficient probably comes closer to the diffusion coefficient for single rather than double molecules since they should tend to diffuse at a faster rate.

If diffusion is examined by means of the mechanism presented by Glasstone, Laidler and Eyering,[14] a clearer picture is obtained of the mechanism of mass transfer. They visualize the process of diffusion as one of liquid molecules sliding past each other and thus jumping from one hole to another. Hence, it is quite reasonable that single

molecules should be able to diffuse faster than double.

If one combines the above concept of diffusion with the theories of association and dissociation it is possible to explain many of the experimental results. Thus, when single and double molecules diffuse to the solvent water interface the single molecules are readily accepted by the water and slide across the interface into holes in the water. However, when double molecules reach the interface the water does not accept them, since incompatible forces exist.

When dealing with water as the dispersed phase and transferring a solute to the continuous solvent phase there is an additional mechanism present which may also cause considerable uncertainty regarding the use of our computed diffusion coefficients. This mechanism is the ion-dipole attraction which exists between water and solutes such as acetic acid, which readily ionizes at low concentrations. If one attempts to explain the mechanism of mass transfer on the basis of a fictive film with the concentration essentially zero at the interface, then there is likely to be a strong tendency for ionization in polar liquids at this same interface. However, the ionic state is not compatible with the continuous non-polar solvent phase, hence a low rate of transfer may result.

The importance of the mechanism of diffusion is shown from this analysis. The value of our final correlations for mass transfer will depend largely upon the evaluation of the diffusion coefficient and proper allowances for interference mechanisms such as the ion-dipole attraction

as well as a correct evaluation of the size and type of diffusion molecules present.

Unsteady State Diffusion Calculations

The basic differential equations for diffusion have been extensively developed in the literature for the more common geometrical shapes. These are all based on a solution of Fick's law of diffusion or the equation of diffusion which is given by:

$$\frac{\partial C}{\partial \theta} = k \nabla^2 C$$

where C - concentration
k - diffusivity
θ - time
∇^2 - Laplacian operator

We can obtain a solution of this diffusion equation applicable to our problem of dispersed fluid spheres by replacing the Laplacian operator by spherical coordinates.

$$\frac{\partial C}{\partial \theta} = D_1 \left[\frac{\partial^2 C}{\partial r^2} + \frac{2}{r} \frac{\partial C}{\partial r} \right]$$

In order to complete the boundary value problem it is necessary to specify both initial and boundary conditions. For the conditions employed in the experiments the boundary condition can be defined as one of zero concentration. Thus:

$$C = 0 \text{ for } r = r_o \text{ and } \theta > 0$$

As a result of the end-effects the interior of the drop is thoroughly mixed at zero time, therefore the initial condition is given by:

$$C = C_f \text{ for } 0 \leq r \leq r_o \text{ and } \theta = 0$$

The selection of the above initial and boundary conditions appears to fit the actual experimental observations. Hence, if the rate of mass transfer for diffusion of acetic acid out of a dispersed drop is assumed to be by means of molecular diffusion only, the solution of the above boundary value problem should give this rate.

In order to reduce the differential equation to a more solvable form, substitute U = Cr above. This yields the expression:

$$\frac{\partial U}{\partial \theta} = D_i \frac{\partial^2 U}{\partial r^2}$$

By assuming a solution of the form $U = \theta \Gamma$ the variables can be separated and a general solution is obtained.

$$U = A\, e^{-\lambda^2 D_i \theta} \begin{cases} \sin(\lambda r) \\ \cos \end{cases}$$

where both A and λ are constants.

By substituting in the boundary and initial conditions and replacing U by Cr the particular solution given below is obtained.

$$U = Cr = -\frac{2\, C_f\, r_o}{\pi} \sum_{n=1}^{\infty} \frac{\cos n\pi}{n} e^{-\left(\frac{n\pi}{r_o}\right)^2 D_i \theta} \sin\left(n\pi \frac{r}{r_o}\right)$$

The mass of solute transferred to the continuous phase across the outer boundary of the spherical drop is given by:

$$Q = \int_0^{\theta} -4\pi r_o^2 D_i \left(\frac{\partial C}{\partial r}\right)_{r_o} d\theta$$

The concentration expression derived above, is differentiated with respect to r and substituted into the mass transfer equation above. The

mass transferred across boundary r_o is then:

$$Q = \frac{8\, C_f\, r_o^3}{\pi} \sum_{n=1}^{\infty} \frac{1}{n^2} \left[1 - e^{-\left(\frac{n\pi}{r_o}\right)^2 D_i \theta} \right]$$

It is possible to make a material balance for the mass of solute transferred from a spherical drop during contact time. This equation is given by:

$$Q = -\frac{8}{6} \pi r_o^3 \, (C_r - C_f)$$

Equating the two equations for the mass of solute stripped from a spherical drop and rearranging the terms, an expression is obtained for the fraction of solute unextracted as a function of the drop radius, diffusivity and time of contact.

$$\frac{C_r}{C_f} = \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \, e^{-\left(\frac{n\pi}{r_o}\right)^2 D_i \theta}$$

In the group of experiments in which the dispersed drops are stripping solute from the continuous phase the boundary conditions are somewhat different. However, the answer which results on solving the differential equation is identical to that shown above. Thus in the experiments in which carbon tetrachloride is dispersed and acetic acid is stripped from a continuous water phase the boundary condition is given by:

$$C = C_c \quad \text{for } r = r_o \quad \text{and } \theta > 0$$

This is a legitimate assumption since the volume of the column is 2500 mls. as contrasted to a thru-put of 50 mls. of dispersed phase.

Hence this, combined with the preferential solubility of acetic acid in water, results in the concentration of the continuous phase remaining practically constant throughout the run. Finally the initial condition is one of zero concentration or of some fixed value. Hence, it can be given by:

$$C = 0 \quad \text{for} \quad \theta = 0 \text{ and } 0 \leq r \leq r_o$$

The solution of the differential equation given previously for these boundary and initial conditions is the same as the former result. However, C_r is the difference between the equilibrium value of C_c and the final dispersed phase product concentration, whereas C_f equals the equilibrium value of C_c.

In order to reduce the labor involved in computing C_r/C_f for the conditions of each run, values of C_r/C_f were computed for a range of values of the parameter $D_i\theta/r_o^2$. The resulting values of C_r/C_f are plotted in Figures 59 and 60 versus $D_i\theta/r_o^2$. The smaller the value of $D_i\theta/r_o^2$ becomes the more terms in the series solution become significant. For values of $D_i\theta/r_o^2$ greater than 0.10 the first term of the series becomes controlling and considerable simplification in the result is possible.

In manipulations with the overall mass transfer coefficient it is possible to reduce the expression for K_o to one involving $\ln(C_f/C_r)$ when the continuous phase concentration is approximately zero. If the unsteady state solution of the diffusion equation is substituted into this expression, the result can be expressed in terms of the overall mass transfer coefficient as follows:

$$K_o = \frac{L}{A} \ln \frac{\pi^2}{6} \frac{1}{\sum_{n=1}^{\infty} \frac{1}{n^2} e^{-\left(\frac{n\pi}{r_o}\right)^2 D_i\theta}}$$

When $D_i\theta/r_o^2$ is greater than 0.10 it is possible to represent the series expansion above by the first term. Thus, if this simplification is introduced the equation becomes:

$$K_o = \frac{L}{A} \left[0.5008 + \frac{9.89\, D_i\theta}{r_o^2} \right]$$

In order to simplify computations using these latter two expressions, a number of values of ln (C_f/C_r) were computed for various values of $D_i\theta/r_o^2$ and plotted in Figure 61.

In the turbulent regime of flow where extraction results tend to be greater than those predicted by the diffusion equations, it is the practice to use the overall mass transfer coefficient based on a fictive film. However, an alternate method of correlation is to use the diffusion equations above and employ a diffusion coefficient which fits the data. This diffusion coefficient is defined as the eddy diffusion coefficient. By means of Figures 59 and 60 and the experimentally measured values of C_f/C_r the eddy diffusion coefficients, D_e, were computed for the series of runs in which a fixed drop diameter of 0.15 inches was employed. The results of this calculation, for the six solvents employed, are presented in Table IV. It is however more difficult to correlate results in the turbulent regime by means of D_e than by means of K_o, based on dimensionless groups.

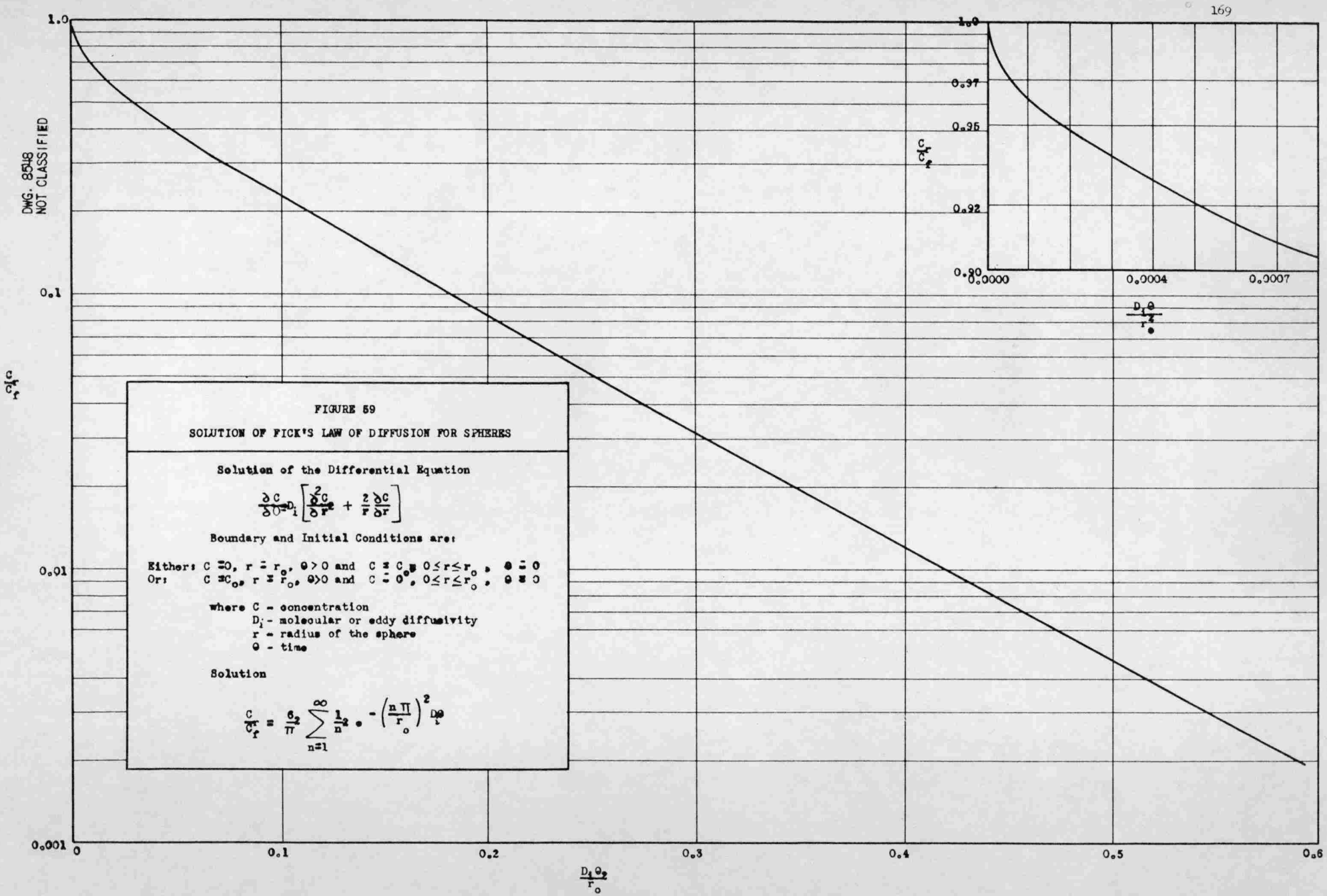

FIGURE 59

SOLUTION OF FICK'S LAW OF DIFFUSION FOR SPHERES

Solution of the Differential Equation

$$\frac{\partial C}{\partial \theta} = D_i \left[\frac{\partial^2 C}{\partial r^2} + \frac{2}{r} \frac{\partial C}{\partial r} \right]$$

Boundary and Initial Conditions are:

Either: $C = 0$, $r = r_o$, $\theta > 0$ and $C = C_o$, $0 \leq r \leq r_o$, $\theta = 0$
Or: $C = C_o$, $r = r_o$, $\theta > 0$ and $C = 0$, $0 \leq r \leq r_o$, $\theta = 0$

where C - concentration
D_i - molecular or eddy diffusivity
r - radius of the sphere
θ - time

Solution

$$\frac{C}{C_f} = \frac{6_2}{\pi} \sum_{n=1}^{\infty} \frac{1}{n^2} \cdot - \left(\frac{n \pi}{r_o} \right)^2 D_i \theta$$

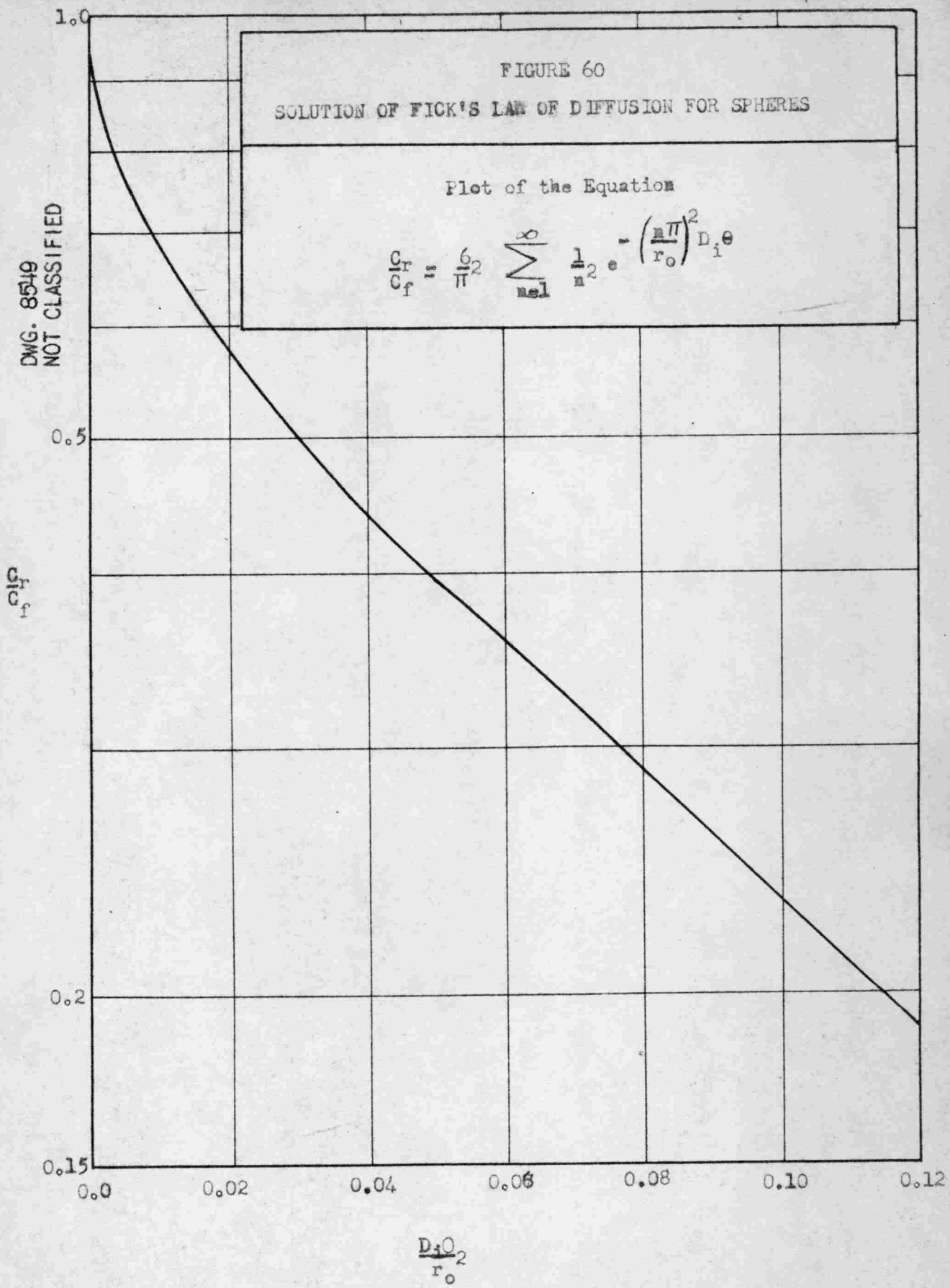

FIGURE 60

SOLUTION OF FICK'S LAW OF DIFFUSION FOR SPHERES

Plot of the Equation

$$\frac{C_r}{C_f} = \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} e^{-\left(\frac{n\pi}{r_o}\right)^2 D_i \theta}$$

DWG. 8550
NOT CLASSIFIED

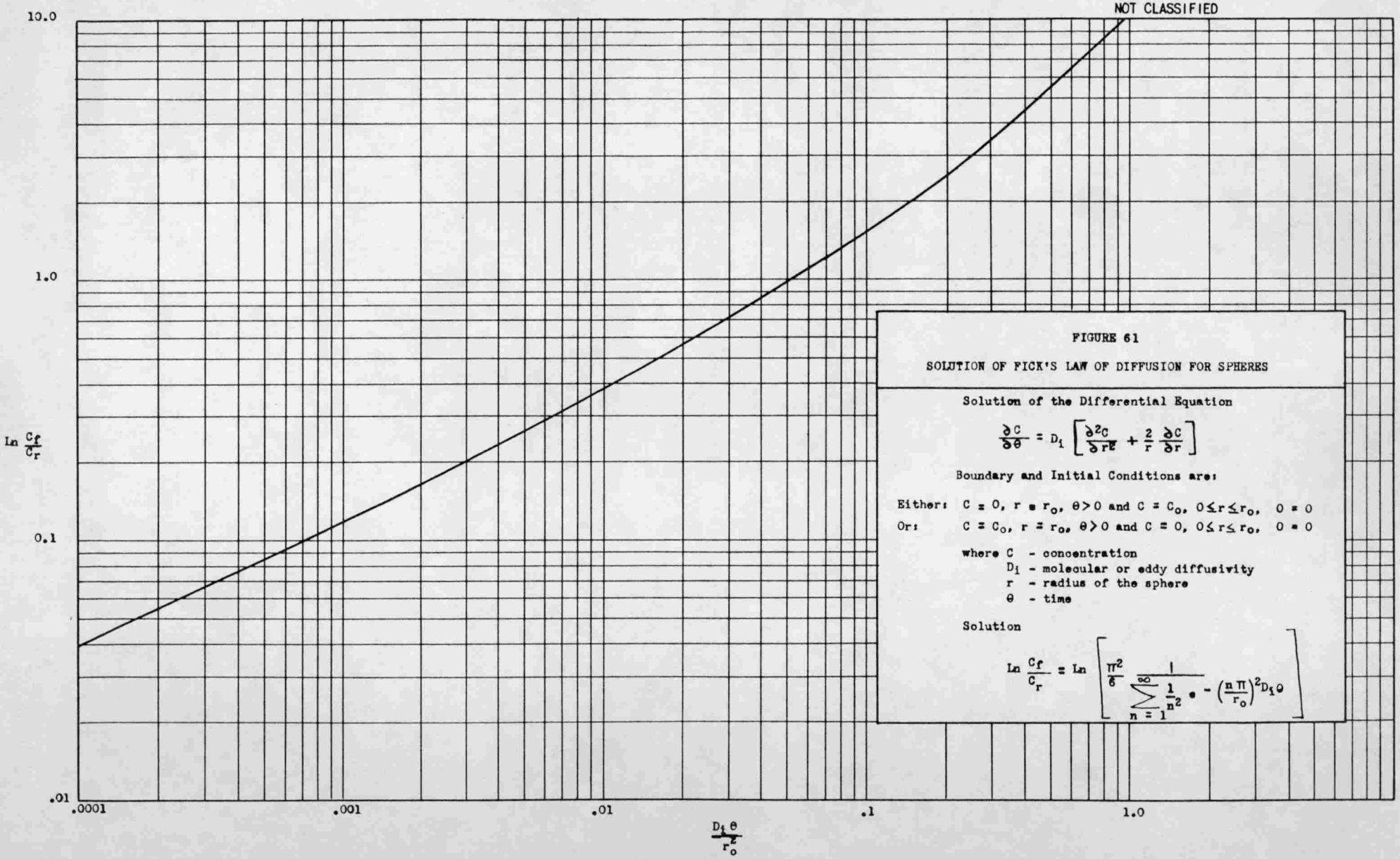

FIGURE 61

SOLUTION OF FICK'S LAW OF DIFFUSION FOR SPHERES

Solution of the Differential Equation

$$\frac{\partial C}{\partial \theta} = D_i \left[\frac{\partial^2 C}{\partial r^2} + \frac{2}{r} \frac{\partial C}{\partial r} \right]$$

Boundary and Initial Conditions are:

Either: $C = 0,\ r = r_o,\ \theta > 0$ and $C = C_o,\ 0 \leq r \leq r_o,\ \theta = 0$

Or: $C = C_o,\ r = r_o,\ \theta > 0$ and $C = 0,\ 0 \leq r \leq r_o,\ \theta = 0$

where C - concentration
D_i - molecular or eddy diffusivity
r - radius of the sphere
θ - time

Solution

$$\ln \frac{C_f}{C_r} = \ln \left[\frac{\pi^2}{6} \frac{1}{\sum_{n=1}^{\infty} \frac{1}{n^2} e^{-\left(\frac{n\pi}{r_o}\right)^2 D_i \theta}} \right]$$

The results of the drop deformation study indicate that the true shape of the drops in the turbulent region is that of an ellipse rather than a sphere. The effect of this shape factor on the diffusion rate is however less than 10%. Hence, the more obtuse relations for an oblate spheroid are not employed.

In an effort to explain the mechanism of mixing and turbulence within the drop, the possibility of vortex formation arose. Thus the drag of the continuous fluid on the sides of the drop could conceivably cause a laminar vortex within the drop, whose shape would resemble that of a doughnut without a center hole. The fluid within the drop would hence flow opposite to the direction of fall on the outside surface and countercurrently down the center of the drop. The mechanism of transfer would then be by diffusion across the laminar flowing layers. It is possible to solve the diffusion equation for this situation if we assume the diffusion pattern to be that of a cylinder of infinite length.

The differential equation for Fick's law of diffusion in cylindrical coordinates is given by:

$$\frac{\partial C}{\partial \theta} = D_i \left[\frac{\partial^2 C}{\partial r^2} + \frac{1}{r} \frac{\partial C}{\partial r} \right]$$

The boundary and initial conditions applicable to this situation are identical to those used for the previous solution in spherical coordinates.

$$C = 0 \text{ for } r = r_o \text{ and } \theta > 0$$
$$C = C_f \text{ for } 0 \leq r \leq r_o \text{ and } \theta = 0$$

In order to solve this differential equation assume a solution in the form of $R \cdot \Theta$ and separate the variables. This gives the following equation:

$$C = \sum_{n=1}^{\infty} A_n \; J_o \; (Rn \; \frac{r}{r_o}) \; e^{-\left(\frac{Rn}{r_o}\right)^2 D_1 \theta}$$

On substituting in the boundary and initial conditions given above the result is simplified to the particular solution:

$$C = 2 \, C_f \sum_{n=1}^{\infty} \frac{1}{Rn \; J_1 \; (Rn)} \; J_o \; (Rn \; \frac{r}{r_o}) \; e^{-\left(\frac{Rn}{r_o}\right)^2 D_1 \theta}$$

The amount of solute transferred from the cylinder to the water using the boundary equation and the solution above is equated to the equation for the amount of solute transferred based on overall measurements. It is then possible to obtain the desired equation:

$$\frac{C_r}{C_f} = 4 \sum_{n=1}^{\infty} \frac{1}{Rn^2} \; e^{-\left(\frac{Rn}{r_o}\right)^2 D_1 \theta}$$

By means of the "Tables of Functions", of Jahnke and Emde, it was possible to determine solutions for the positive roots of Rn, the zero order bessel equation. Values of C_r/C_f were then computed for a range of values of $D_1\theta/r_o^2$ and plotted in Figures 62 and 63. In solving the series above, the first term only gives sufficient accuracy when $D_1\theta/r_o^2$ is greater than 0.12.

The solution of the differential equation given above was used to compute values of $\ln(C_f/C_r)$ as a function of $D_i\theta/r_o^2$. The results are plotted in Figure 64. It is possible then rapidly to compute values of K_o based on the mechanism of diffusion.

The results of the experiments when compared using the above expression indicate that vortex formation in the form of laminar flow layers does not explain the rate of mass transfer observed. This does not however mean that vortex formation could not take place within the drop.

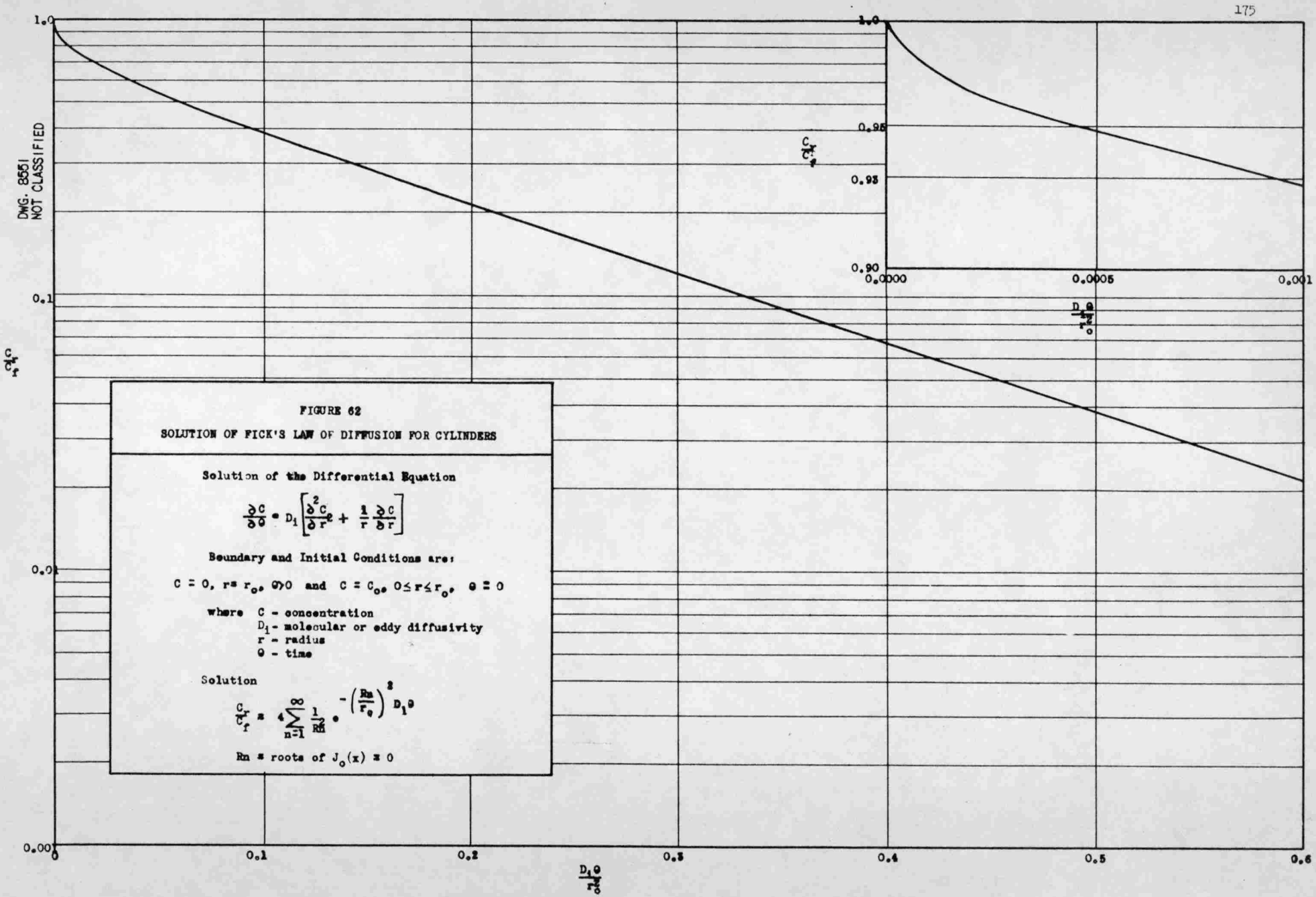

FIGURE 62

SOLUTION OF FICK'S LAW OF DIFFUSION FOR CYLINDERS

Solution of the Differential Equation

$$\frac{\partial C}{\partial \theta} = D_1 \left[\frac{\partial^2 C}{\partial r^2} + \frac{1}{r} \frac{\partial C}{\partial r} \right]$$

Boundary and Initial Conditions are:

$C = 0,\ r = r_o,\ \theta > 0$ and $C = C_o,\ 0 \leq r \leq r_o,\ \theta = 0$

where C - concentration
D_1 - molecular or eddy diffusivity
r - radius
θ - time

Solution

$$\frac{C}{C_f} = 4 \sum_{n=1}^{\infty} \frac{1}{Rn^2} e^{-\left(\frac{Rn}{r_o}\right)^2 D_1 \theta}$$

Rn = roots of $J_o(x) = 0$

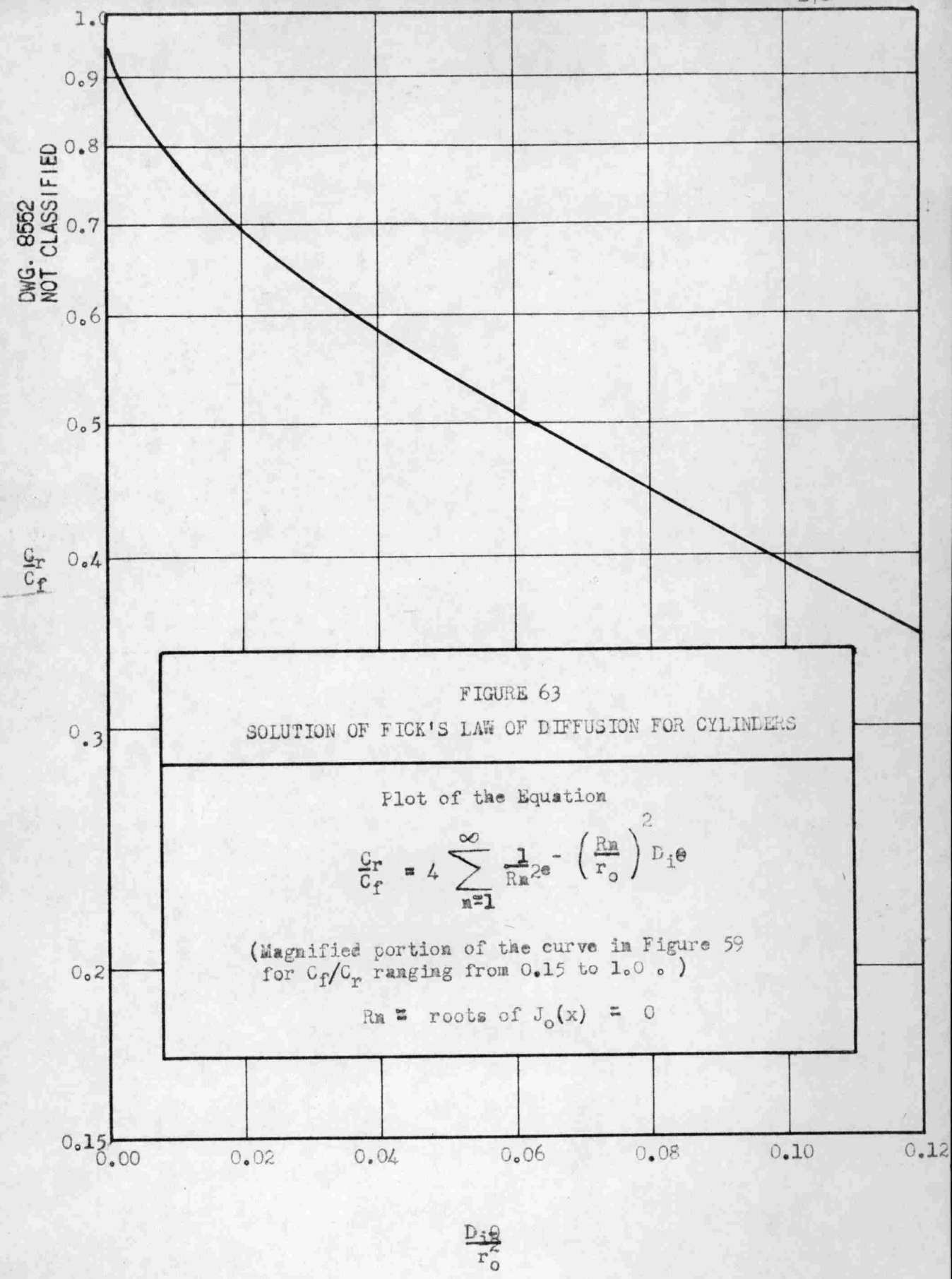

FIGURE 63

SOLUTION OF FICK'S LAW OF DIFFUSION FOR CYLINDERS

Plot of the Equation

$$\frac{C_r}{C_f} = 4 \sum_{n=1}^{\infty} \frac{1}{Rm^2} e^{-\left(\frac{Rm}{r_o}\right)^2 D_i\theta}$$

(Magnified portion of the curve in Figure 59 for C_f/C_r ranging from 0.15 to 1.0 .)

Rm = roots of $J_o(x) = 0$

DWG. 8553
NOT CLASSIFIED

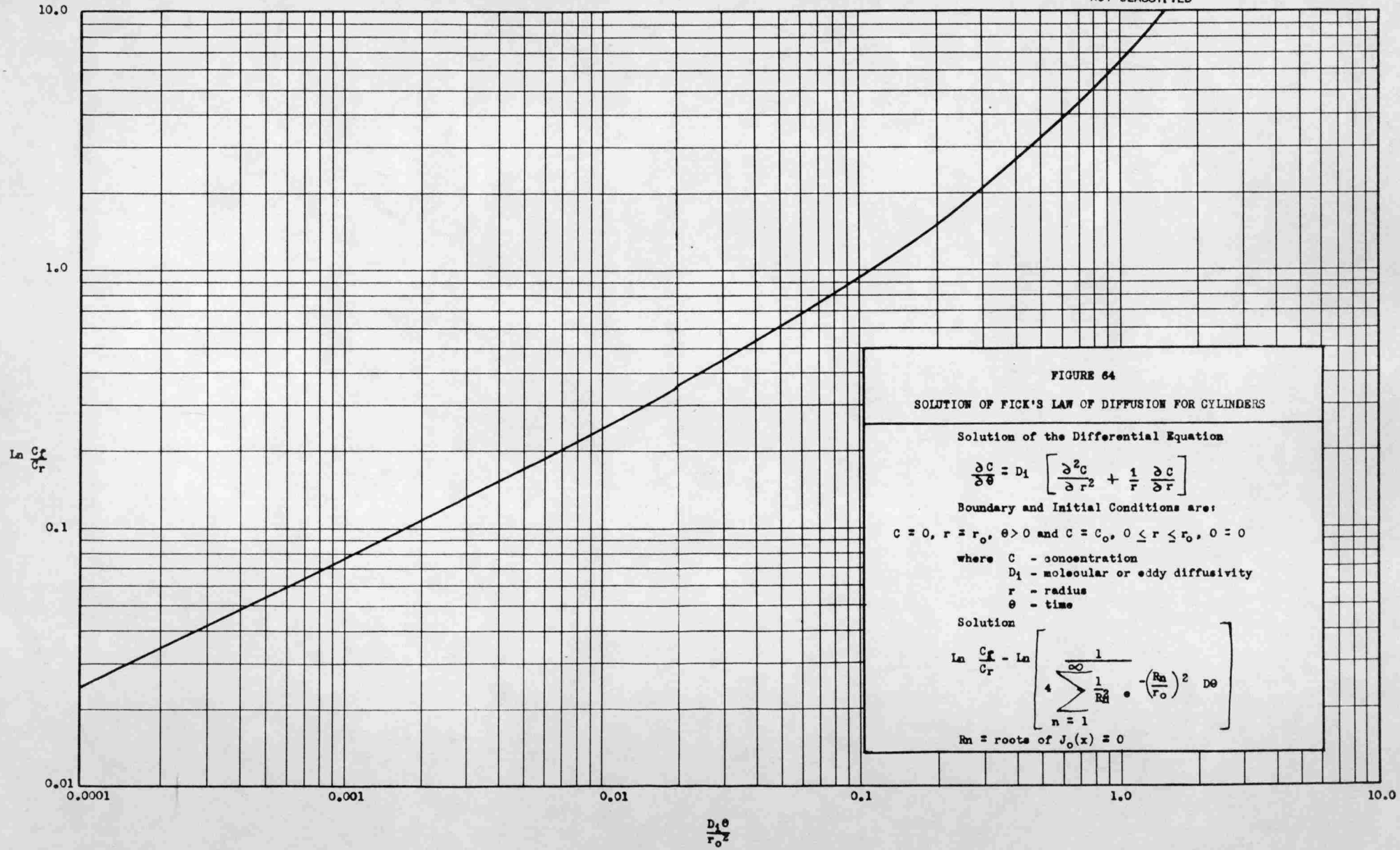

FIGURE 64

SOLUTION OF FICK'S LAW OF DIFFUSION FOR CYLINDERS

Solution of the Differential Equation

$$\frac{\partial C}{\partial \theta} = D_i \left[\frac{\partial^2 C}{\partial r^2} + \frac{1}{r} \frac{\partial C}{\partial r} \right]$$

Boundary and Initial Conditions are:

$C = 0,\ r = r_o,\ \theta > 0$ and $C = C_o,\ 0 \leq r \leq r_o,\ \theta = 0$

where C - concentration
D_i - molecular or eddy diffusivity
r - radius
θ - time

Solution

$$\text{Ln} \frac{C_f}{C_r} - \text{Ln} \left[\frac{1}{4 \sum_{n=1}^{\infty} \frac{1}{R_n^2} \cdot e^{-\left(\frac{R_n}{r_o}\right)^2 D\theta}} \right]$$

R_n = roots of $J_o(x) = 0$

APPENDIX F

CORRELATION OF DATA BASED ON FILM CONCEPTS

Fictive film theories for correlating mass heat and momentum transfer in the turbulent flow regime has been developed. This type of correlation has been applied with success to situations in which the flow of fluid is in the form of films or fills annuli and pipes. In the case of the liquid drop system however, we are dealing with a fluid with a closed boundary in contrast to these other cases. When the boundary is stationary, as in the viscous flow regime, the liquid drop is spherical and the fluid inside is stationary or at least in viscous motion. In the turbulent regime of flow, however, there are strong forces of fluid resistance acting on the drop and as a result a turbulent condition exists in the core of the drop.

There are two means by which a turbulent core with an outer film can be visualized for a fluid drop. The first could arise as a result of surface shear creating a vortex at the outer surface of the drop. The flow of the vortex would be laminar and hence mass transfer would have to be by diffusion across the laminar layer. The core of the drop would then be turbulent from dissipation of the vortex. The second means by which turbulence can be visualized within the drop, is by virtue of the oscillation and unstable motion of the drop as a result of deformation in the turbulent regime of flow.

The general equation for the mass transfer coefficient is simplified due to the nature of the experiments employed. Thus the overall mass transfer coefficient can be treated as if equal to the dispersed phase film coefficient. Since the continuous phase is at approximately zero concentration common terms of the driving force and mass transfer expressions cancel. The simplified mass transfer coefficient becomes:

$$K_s = k_s = \frac{V}{A\ H} \ln \frac{C_f}{C_r}$$

The concept of one film is valid certainly in the turbulent regime of flow and is also probably valid in the transitional regime. In the viscous regime however, there is a strong possibility of the existence of two films. Most of the experiments were in the turbulent regime, therefore the expressions above are suitable.

To make the mass transfer expression more workable and be able to interpret the results in terms of the variables involved, several terms above can be replaced by equivalent expressions. When this is done, the following equation is obtained:

$$K = \frac{V\ d}{6\ H} \ln \frac{C_f}{C_r}$$

In analyzing the experimental data it was found that more extraction occurred during drop formation or at the jack-leg interface than is expected from diffusion calculations. In order to correlate the mass transfer coefficient as a function of the properties of the system we must deal with ordered and measurable conditions. Since these end effects involve an area in a dynamic state as contrasted to a static

area for free-fall of the drops, it was necessary to separate the mass transfer due to end effects from that of free-fall. To do this it was necessary to measure the rate of extraction over varying column heights under fixed conditions.

By rearranging the previous equation for the mass transfer coefficient, the column height can be expressed as a function of product and feed concentrations.

$$H = \frac{V\,d}{6\,K} \ln \frac{C_f}{C_r} = (\text{constant}) \ln \frac{C_f}{C_r}$$

By using the same nozzle in each column height run for a particular system the drop diameter, d, can certainly be maintained constant and hence V will be a constant. If both V and d are held constant then K will be a constant provided it is independent of concentration. Both the distribution ratio K and the degree of association for the solvents used in the experiments, varied less than 5% from zero column height up to the maximum height of 5 feet. In the studies with carbon tetrachloride using varying concentrations of acetic acid in the feed, the overall extraction coefficient remained practically constant over the range of concentration which is met in those systems employing organic liquids in which acetic acid association takes place. Therefore, it is possible to treat all the terms but those involving H and $\ln(C_f/C_r)$ as constants.

If C_f/C_r is plotted on semi-log paper versus column height, the amount of extraction as a result of end effects can be evaluated by

extrapolating to zero column height. A plot of this kind is shown in Figure 14 in comparing the results of the various systems for a drop diameter of approximately 0.15 inches. The mathematical relations governing this extrapolation thus are:

$$\Delta H = H = \left[\ln(C_f/C_r)\right]_{H=H} - \left[\ln(C_f/C_r)\right]_{H=0}$$

Hence:

$$(C_f/C_r)_{\text{free-fall}} = (C_f/C_r)_{\text{overall}} / (C_f/C_r)_{\text{end-effect}}$$

It is possible, using the amount of extraction due to the end effects to compute overall transfer coefficients. It is extremely difficult to determine a rate of extraction correlation for the end effects as a function of the properties of the system, since they are affected by the diameter of the nozzle, the drop formation time and in addition the area is in a dynamic state. Therefore equations were not developed for these results.

Having separated the amount of extraction for free-fall from that due to end-effects, it is possible to compare these results with the properties of our systems. Since the area is in a static state, it can be replaced in the extraction equation by the area of a sphere. Free-fall extraction data were obtained for each of the solvent-water systems for the same drop diameter and column height. Drop diameter was used for convenience as a key variable rather than Reynolds number, although the latter approach does simplify analysis. The dispersed phase

film coefficients or overall mass transfer coefficients computed for the free-fall condition are tabulated in Table II.

The film coefficient obtained is expected to be a function of the variables of the system. Thus:

$$K_o = f_1 (\mu, V, \rho, d, D_i \text{ and I.T.})$$

If the methods of dimensional analysis are applied to this expression, the relation between the variables comes out to be as follows:

$$\frac{Kd}{D_i} = f_2 \left(\frac{dV\rho}{\mu}\right)^a \left(\frac{u}{\rho D_i}\right)^b \left(\frac{d\rho V^2}{I.T.}\right)^c$$

The first term is equivalent in mass transfer to the Nusselt group in heat transfer. The first term on the right is the familiar Reynolds number. The second is the Schmidt number which is a measure of the properties of our fictive film. The last group is the Weber capillary group. This latter group is a measure of the deformation of the drop.

The introduction of interfacial tension is a new step in correlating mass transfer coefficients. Since most mass transfer correlations have been for wetted-wall columns and similar apparatus where the interfacial tension does not influence the shape of the fluid, it is not surprising that this variable has not appeared before. The interfacial tension affects the results obtained here only by its influence on the size and shape of the drops. In series J in which interfacial tension was varied the only changes observed in the extraction coefficient were believed to be a result of changes in the shape of the drop and not a result of surface forces acting on the individual diffusing solute molecules.

If interfacial tension is assumed not to affect our results, then the correlation for the overall mass transfer coefficient is given by:

$$\frac{Kd}{D_i} = f_3 \left(\frac{dV}{u}\rho\right)^a \left(\frac{u}{\rho D_i}\right)^b$$

This same relation is obtained from analogy to heat and mass transfer for a condition of high relative velocity at the interface of the drop and continuous phase. Thus, Wilhelm[29] obtained the relation:

$$k_L \propto D_i (Re)^n (\text{Schmidt group})^m \propto D_i \left(\frac{dG}{\mu}\right)^n \left(\frac{\mu}{D_i \rho}\right)^m$$

Gilliland and Sherwood[13] and Fallah, Hunter and Nash[12] correlated data on wetted-wall column experiments by means of this relation. The value of n lies very close to 0.8 and m to 0.4 in these latter experiments. The above relation is also very similar to that commonly employed for heat transfer in turbulent motion.

$$\frac{hd}{k} = 0.0225\ (Re)^{0.8}\ (Pr)^{0.4}$$

In analyzing the experimental data a fair correlation was obtained using this expression derived from the analogous turbulent flow heat transfer expression, provided one dealt with a fixed diameter. However, in order to fit Figure 15 where the overall mass transfer coefficient varies with diameter, the terms d and V must appear as a net term of $d^{0.6}\ V^{2.4}$. Hence an additional term involving $(dV^2)^{0.8}$ must be present in our correlation. The Weber capillary group thus not only completes the

expression obtained by dimensional analysis but also introduces the variables in the proper order to fit our experimental data.

By analyzing the experimental results by means of the expression obtained from dimensional analysis, it is possible to evaluate the constant of proportionality and the power of the Schmidt group. The resulting expression is given by:

$$\frac{Kd}{D_i} = 0.03\left(\frac{dV\rho}{\mu}\right)_d^{0.8}\left(\frac{\mu}{\rho D_i}\right)_d^{0.44}\left(\frac{d\,V^2\rho_c}{I.T.}\right)^{0.8}$$

A comparison between the predicted results from this equation and those obtained experimentally are shown in Table V.

In order to explain the experimental results, it is necessary to take the degree of association into account. This is fully covered in the body of this paper and therefore will not be discussed further here.

In order to fit the data more closely, an additional term ν_c/ν_d can be introduced. This aids the correlation in a few instances but results in a poorer fit of the data in others, and hence is excluded from our final result. The omission of the term $(\rho - \rho_c)$ also appears to be valid since this is a measure of bouyancy and is accounted for by the introduction of velocity into our correlation. Similarly, the omission of the term $2r/d_c$ is valid since our drops are small relative to the column diameter and hence wall effects are negligible.

The methods of mathematical determinants were applied to the results obtained, exclusive of corrections for association. The set of equations which result were unable to be solved without including all the variables found in our previous dimensional analysis solution. Using these variables the following expression was obtained:

$$\frac{K_o d}{D_i} = 4.6 \times 10^{10} \quad \frac{D_i^{0.35} \; V^{2.4} \; \rho^{2} \; d^{1.87}}{\mu^{2} \; I.T.^{1.87}}$$

where K_o = overall mass transfer coefficient, ft./hr.

D_i = molecular diffusivity, in^2/sec.

V = velocity, ft./sec.

ρ = density, grs./cc.

μ = viscosity, cp.

I.T. = Interfacial tension, I.T.

If an association correction factor $A = n/(2-n)$ is included in the numerator of the right hand term, then a fairly reasonable correlation of the experimental data is obtained. This expression however, does not represent the data as well as our previous evaluation based on dimensional analysis, since the above equation is arrived at by applying determinants to the results prior to correcting for association. Hence, the true relation of the variables is obscured.

APPENDIX G

EXPERIMENTAL RESULTS AND CALCULATIONS

The same measurements and analysis of data were made for almost all the experiments. The method employed can therefore be generalized. Each series of experiments was denoted by a letter of the alphabet running from A thru Z. Following this notation came the number of the experiment in each series.

The physical dimensions of the apparatus were measured and noted. The nozzle employed was designated by a code showing the general type employed. (e.g. C.T. stands for capillary tube). In addition the inside and outside diameter of the nozzle was measured. After the nozzle was fastened in place, the height (H) from the nozzle to the interface was measured. The volume of continuous phase contained in a five foot high section of the column is equal to approximately 2500 mls. Although the continuous phase liquid level extended in many experiments the length of the column, the volume used in concentration calculations was proportional to the height (H) and 500 mls. per foot of height (H). This assumption is valid since diffusion of the solute into that portion of the continuous phase through which drops do not pass was relatively small. This assumption has been verified by concentration traverses the length of the column.

The temperature of both the continuous and dispersed phases were recorded. The continuous phase temperature was taken as that of the jacket water temperature (T_c). A Weston metal thermometer was

located near the feed tank and the dispersed phase temperature (T_d) was read on this instrument. In addition the room temperature T was recorded. Fluctuations in feed temperature were controlled by regulating the thermostat governing the room temperature.

A 50 ml. graduate was used as a product receiver. The time required to fill the graduate or put 50 mls. of dispersed liquid through the column was recorded by an electric timer and denoted by θ. In order to compute the drop velocity, the time required for a drop to fall from the nozzle to the interface was clocked with a stopwatch. This was recorded on the data sheets as θ_t. Finally the rate at which the drops form was determined by a stopwatch and recorded as θ_f. The usual procedure was to time 25 drops and obtain the time per drop θ_f, by averaging.

In the majority of the experiments the solvent was the dispersed phase and water was the continuous phase. Water being expendable the column was drained and refilled after each run with water containing no acetic acid or impurities. At the end of each run 50 ml. samples were removed from the bottom and top tits by means of 50 ml. volumetric flasks. These were transferred to 250 ml. erlenmeyer flasks and the acetic acid content was determined by titration with 0.1 N. sodium hydroxide using thymol blue as an indicator. The concentratins of acetic acid in the top and bottom are denoted by C_T and C_B.

The amount of extraction taking place was determined by analyzing the feed and product. A 50 ml. sample was removed from the feed tank using a 50 ml. volumetric flask and transferred to a 250 ml. erlenmeyer flask. The entire product in the 50 ml. graduate was also transferred to another erlenmeyer flask. When a solvent was being used for the feed stream sufficient water had to be added to the erlenmeyers to extract the acetic acid. The acetic acid concentration was then determined by titration with sodium hydroxide as before.

The results were converted to terms which can be employed in correlations. The principle correlation is that based on the one film theory using the overall mass transfer coefficient based on the dispersed phase. The overall mass transfer coefficient is given by:

$$K_oA \ \ln \Delta C = L\,(C_f - C_r)$$

where K_o = overall mass transfer coefficient, lb.moles/hr.sq.ft.lb.mole/cu.ft.

L = flow rate, cu.ft./hr.
C_f = feed concentration, lb.moles/cu.ft.
C_r = product concentration, lb.moles/cu.ft.
A = interfacial area, sq. ft.

The logarithmic mean driving force (ΔC) can be expressed in terms of concentrations by the following expression:

$$\ln \Delta C = \frac{(C_f - C_T^*) - (C_r - C_B^*)}{\ln \dfrac{C_f - C_T^*}{C_r^* - C_B^*}}$$

where $C_T^* = K'C_T$ = equilibrium concentration of continuous phase lb.moles/cu.ft.

$C_B^* = K'C_B$ = equilibrium concentration of continuous phase lb.moles/cu.ft.

Since both the distribution ratio (K') and the top and bottom sample concentrations are small, the above equation can be simplified by eliminating the equilibrium concentration terms. This introduces a maximum error of at most 3% in any of the experiments. Thus, our expression for the overall mass transfer coefficient can usually be simplified to:

$$K_o = \frac{L}{A} \ln \frac{C_f}{C_r}$$

The dispersed drops varied from spheres at small diameters to ellipsoids at larger diameters. As was shown in the drop formation study (Appendix D), a negligible error is introduced if we treat the drops as perfect spheres in determining both volume and interfacial area. The number of drops formed per hour or drop rate (D) was maintained constant throughout any experimental run by flow regulation. Therefore, for a thru-put of 50 mls. the diameter of the drops was computed by $\frac{\pi d^3}{6} D \theta = 50$

where d = drop diameter, ft.
θ = experiment time, hrs.
D = drop rate, drops/hr.

Having determined the diameter (d) from this expression for the volume throughput of spherical drops, it was possible to compute both the interfacial area and flow rate. These are given by:

$$A = D \, \theta_t \, \pi d^2$$

$$L = \frac{\pi d^3 \, D}{6 \, \theta}$$

where θ_t = contact time, hrs.

The results of computations such as these are shown for all experimental runs in Table XXIV through Table XLIX. In order to eliminate excess tabulation, data sheet values are not shown.

Various techniques can be employed in treating the experimental data in order to obtain more uniform answers. Both A and L can be replaced in the expression for the overall mass transfer coefficient by terms involving drop rate, drop diameter and time, The time of contact can also be replaced by H/V where H is column height and V the velocity. The final expression for K_o is then given by:

$$K_o = \frac{V}{6} \frac{d}{H} \ln \frac{C_f}{C_r}$$

A normalizing technique can be used to compute K_o from this expression. This consists of plotting velocity versus drop diameter and fraction extracted versus drop diameter. Values can then be read from the smooth curves through the data, and K_o values computed from these numbers. This technique was used in determining the overall mass transfer coefficient for series A runs, but due to the time involved was not employed in correlating subsequent results.

In addition to the data already mentioned, in several special experimental series of runs, other information was noted. Thus, in series G the flow rate of the continuous phase was determined by means of a rotameter which was later calibrated to give flow rates for rotameter readings. The Reynolds number was computed for both the drop diameter $(Re)_d$ and the column diameter $(Re)_c$. In series H runs, the

concentration of sodium hydroxide in the continuous phase was determined by titration with standardized sulfuric acid using phenolthalein as an indicator. The acetic acid concentration of the continuous phase is then obtained by difference.

Sample Calculation

The following data were recorded from the data sheet.

Experimental run - A 1

Nozzle - 5 mm. Capillary tube

Jacket temperature (T) - 21° C.

Total experiment time (θ) - 2224 seconds

Dispersed phase thru-put - 50 mls.

Drop rate (D) - 25 drops per 11.3 seconds

Time of Contact (θ_t) - 8.75 seconds

Column Height (H) - 4 ft. 11 inches

Feed sample concentration (C_f) - 84 mls. - 0.1028 N. sodium hydroxide.

Product sample, 50 mls. (C_r) - 42 mls. - 0.1028 N. sodium hydroxide.

Initial column concentration - 0 mls. - 0.1028 N. sodium hydroxide.

Top column sample (C_T) - 1.4 mls. 0.1028 N. sodium hydroxide.

Bottom column sample (C_B) - 1.2 mls.-0.1028 N. sodium hydroxide.

The data were then converted to terms appearing in the expression for the overall mass transfer coefficient.

Drop size:

$$\left(\frac{\pi\, d^3}{6}\right)\left(\frac{25}{11.3}\right)\left(2224\right) = 50$$

$$d = 0.269 \text{ cm. or } 0.106 \text{ inches.}$$

Concentrations

Feed $\quad C_f = \dfrac{(84)\ (0.1028)}{802.5} = 0.01075$ lb.moles/cu.ft.

Product $\quad C_r = \dfrac{(42)\ (0.1028)}{802.5} = 0.00538$ lb.moles/cu.ft.

Bottom column sample $\quad C_B = \dfrac{(1.2)\ (0.1028)}{802.5} = 0.000154$ lb.moles/cu.ft.

Top column sample $\quad C_T = \dfrac{(1.4)\ (0.1028)}{802.5} = 0.00018$ lb.moles/cu.ft.

Logarithmic mean driving force

Since K' is equal to 0.05 we can ignore equilibrium column concentrations.

$$\ln \Delta C = \frac{0.01075 - 0.00538}{\ln \dfrac{0.01075}{0.00538}} = 0.00777$$

Amount of acetic acid extracted

$$C_f - C_r = 0.01075 - 0.00538 = 0.00537 \text{ lb.moles/cu.ft.}$$

Flow rate

$$L = \frac{(50)\ (3600)}{(28{,}320)\ (2224)} = 0.00286 \text{ cu.ft./hr.}$$

Interfacial area

$$A = \left(\frac{25}{11.3}\right)\ (8.75)\ (\pi)\ \frac{(0.106)^2}{144} = 0.00475 \text{ ft.}^2$$

Overall mass transfer coefficient

$$K_o = \frac{(0.00268)}{(0.00777)} \frac{(0.00537)}{(0.00475)} = 0.417 \ \frac{\text{lb.moles/hr. ft.}^2}{\text{lb. moles/cu.ft.}}$$

Column Height

$$H = 4.917 \text{ feet}$$

Velocity

$$V = 4.917/8.75 = 0.562 \text{ ft./sec.}$$

Percent Extraction

$$(0.00537/0.01075)\ 100 = 50\%$$

The results of computations similar to these for all the experiments are tabulated in the following tables.

TABLE VI

DISTRIBUTION DATA FOR CARBON TETRACHLORIDE-ACETIC ACID-WATER

Data from the International Critical Tables, Volume V.

Temperature 25°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_C	C_W	$K' = C_C/C_W$
0.0156	0.260	0.060
0.0623	0.388	0.106
0.1247	0.725	0.172
0.187	0.770	0.243
0.249	0.795	0.313

Data determined experimentally at Oak Ridge National Laboratory by W. S. Farmer.

Temperature 21°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_C	C_W	$K' = C_C/C_W$
0.00873	0.21	0.0416
0.00798	0.232	0.0343
0.00192	0.0973	0.0198
0.000198	0.023	0.00855
0.000299	0.0327	0.00913
0.000150	0.0124	0.0121
0.0000374	0.00345	0.0108
0.0000311	0.00185	0.0168

TABLE VI (CONT'D)

Data from Solubilities of Organic Chemicals, by Seidell.
Results of Bektourov on page 108

Temperature 25°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_C	C_W	$K' = C_C/C_W$
0.0000588	0.00456	0.0129
0.00162	0.064	0.0253
0.00441	0.116	0.0382
0.00884	0.168	0.0526
0.0133	0.223	0.0595
0.0228	0.308	0.074
0.0463	0.483	0.0962
0.0832	0.637	0.1305
0.106	0.694	0.153

Data from Solubilities of Organic Chemicals, by Seidell.
Results of Herz and Lewy on page 105

Temperature 25°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_C	C_W	$K' = C_C/C_W$
0.0187	0.312	0.06
0.0312	0.416	0.075
0.0499	0.52	0.096
0.0603	0.623	0.0967
0.125	0.728	0.172
0.262	0.792	0.33

Definition:

C_C - concentration of acetic acid in carbon tetrachloride

C_W - concentration of acetic acid in demineralized water

TABLE VII

DISTRIBUTION DATA FOR CARBON TETRACHLORIDE-ACETIC ACID-WATER (CONTAINING 0.1% TERGITOL NO. 4)

Data determined experimentally at Oak Ridge National Laboratory by W. S. Farmer.

Temperature 20°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_C	C_W	$K' = C_C/C_W$
0.0183	0.441	0.0416
0.00735	0.227	0.0324
0.00196	0.0971	0.0201
0.000623	0.0498	0.0125
0.000997	0.00747	0.0133
0.000112	0.00392	0.0286
0.000137	0.00226	0.0605
0.000112	0.00112	0.10

TABLE VIII

DISTRIBUTION DATA FOR METHYL ISOBUTYL KETONE-ACETIC ACID-WATER

Data from Industrial and Engineering Chemistry, October, 1941, pages 1240-1248.
Results of D. F. Othmer, R. E. White and E. Trueger.

Temperature 22°C.

Concentration of Acetic Acid Lb. Moles/ Cu. Ft.		Distribution Coefficient
C_M	C_W	$K = C_W/C_M$
5.7	9.43	1.65
10.5	15.9	1.51
19.1	26.7	1.4
22.9	31.3	1.37
25.6	33.6	1.31
27	35.1	1.3

TABLE VIII (CONT'D)

Data determined experimentally at Oak Ridge National Laboratory by W. S. Farmer.

Temperature 21°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_M	C_W	$K = C_W/C_M$
0.0069	0.014	2.11
0.0052	0.011	2.11
0.00352	0.0073	2.07
0.00178	0.00377	2.11
0.000623	0.00155	2.48
0.000436	0.000935	2.14
0.000262	0.000511	1.95
0.0377	0.0694	1.84

Data from Industrial and Engineering Chemistry, September, 1939, pages 1144-
Results of T. K. Sherwood, J. E. Evans and J. V. A. Longcor

Temperature 25°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_M	C_W	$K = C_W/C_M$
1.56	2.96	1.9
7.4	12.2	1.65
14.4	21.5	1.5
20.5	27.2	1.33
25.6	34.1	1.33
27.9	36	1.29

Definition:

C_M - concentration of acetic acid in methyl isobutylketone

C_W - concentration of acetic acid in water

TABLE IX

MUTUAL SOLUBILITY FOR METHYL ISOBUTYL KETONE-ACETIC ACID-WATER

Data from Industrial and Engineering Chemistry, September, 1939, pages 1144-
Results of T. K. Sherwood, etc.

Temperature 25°C.

Mutual Solubility Data			Tie Line Data	
Ketone %	Water %	Acetic Acid %	Ketone Layer Acetic Acid %	Water Layer Acetic Acid %
1.55	98.45	0	1.87	2.85
3.7	76.8	19.5	8.9	11.7
10.5	57.5	32.0	17.3	20.5
17.4	48.4	34.2	24.6	26.2
26.0	39.6	34.4	30.8	32.8
37.6	29.1	33.3	33.6	34.6
51.6	19.2	29.2		
66.4	12.0	21.6		
81.6	6.5	11.9		
97.9	2.12	0		

Additional data can be found in
Industrial and Engineering Chemistry, October, 1941, pages 1240-1248
Results of D. F. Othmer, R. E. White and E. Trueger.

TABLE X

DISTRIBUTION DATA FOR BENZENE-ACETIC ACID-WATER

Data from the International Critical Tables, Volume V.

Temperature 15 - 16°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_B	C_W	$K = C_W/C_B$
0.0115	0.164	14.3
0.00312	0.0741	23.8
0.000255	0.0141	55.1
0.000112	0.0068	60.7
00.0000262	0.00209	80.1

TABLE X (CONT'D)

Data from the International Critical Tables, Volume V

Temperature 25°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_B	C_W	$K = C_W/C_B$
0.0679	0.47	6.93
0.046	0.38	8.26
0.0382	0.339	8.87
0.0302	0.295	9.76
0.0160	0.205	12.9
0.0140	0.185	13.3
0.00985	0.162	16.4
0.00633	0.116	18.3
0.00445	0.0942	21.2
0.00276	0.0715	25.9
0.00103	0.0395	38.4
0.000779	0.0331	42.5
0.1938	0.693	3.58
0.168	0.675	4.02
0.091	0.541	5.95

Data determined experimentally at Oak Ridge National Laboratory by W. S. Farmer.

Temperature 21°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_B	C_W	$K = C_W/C_B$
0.0288	0.392	13.6
0.0156	0.203	13.0
0.00349	0.0878	25.1
0.00134	0.0467	35
0.000499	0.0245	49.1
0.0000748	0.00461	61.6
0.0000374	0.002	53.3
0.0000187	0.00124	66

TABLE X (CONT'D)

Data from Solubilities of Organic Chemicals, by Seidell
Results of Herz and Fischer on page 106

Temperature 25°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_B	C_W	$K = C_W/C_B$
0.00135	0.052	38.5
0.00434	0.104	24
0.0161	0.208	12.9
0.0315	0.312	9.9
0.0515	0.416	8.08

Data from Solubilities of Organic Chemicals, by Seidell
Results of Bektourov on page 107

Temperature 0°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_B	C_W	$K = C_W/C_B$
0.000241	0.0195	82.6
0.000705	0.0383	54.4
0.00259	0.0868	33.4
0.00669	0.153	23.0
0.0159	0.249	15.6
0.0283	0.352	12.5
0.0472	0.453	9.6
0.0795	0.608	7.63
0.153	0.733	4.77
00.254	0.796	3.13
0.401	0.736	1.83

Temperature 25°C.

Concentration of Acetic Acid Lb. Moles/Cu.Ft.		Distribution Coefficient
C_B	C_W	$K = C_W/C_B$
0.00124	0.0483	39.0
0.00177	0.0625	35.3
0.0026	0.079	30.4

TABLE X (CONT'D)

Temperature 25°C. (Cont'd)

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_B	C_W	$K = C_W/C_B$
0.00825	0.165	20.0
0.0174	0.252	14.4
0.0295	0.353	12.0
0.0513	0.482	9.4
0.0725	0.559	7.71
0.0932	0.631	6.77
0.303	0.761	2.51
0.381	0.695	1.82

Temperature 60°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_B	C_W	$K = C_W/C_B$
0.00215	0.0578	26.6
0.00412	0.0885	21.5
0.00766	0.116	15.1
0.0188	0.207	11.0
0.0236	0.245	10.4
0.033	0.234	8.96
0.033	0.296	8.96
0.0513	0.4	7.70
0.0837	0.543	6.49
0.152	0.67	4.40
0.256	0.728	2.84
0.354	0.725	2.04

Definition:

C_B - concentration of acetic acid in benzene

C_W - concentration of acetic acid in water

TABLE XI

MUTUAL SOLUBILITY DATA FOR BENZENE-ACETIC ACID-WATER

Data for Solubility of Organic Chemicals, by Seidell
Results of Waddell on page 107

Temperature 25°C.

Benzene Layer			Water Layer		
Acetic Acid %	Benzene %	Water %	Acetic Acid %	Benzene %	Water %
0.46	99.5	0.02	9.4	0.18	90.42
3.10	96.75	0.15	28.2	0.53	71.3
5.20	94.6	0.25	37.7	0.84	61.5
8.7	90.9	0.42	48.3	1.82	49.9
16.3	82.9	0.79	61.4	6.1	32.5
30.5	67.4	2.13	66.0	13.8	20.2
52.5	39.6	7.60	52.8	39.6	7.6

TABLE XII

DISTRIBUTION DATA FOR CHLOROFORM-ACETIC ACID-WATER

Data determined experimentally at Oak Ridge National Laboratory by W. S. Farmer.

Temperature 20°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_C	C_W	$K = C_W/C_C$
0.0546	0.259	4.74
0.0233	0.147	6.3
0.00735	0.0698	9.5
0.00302	0.0399	13.2
0.00118	0.021	17.8
0.000157	0.00548	35
0.00191	0.03	15.1

TABLE XII (CONT'D)

Data from Industrial and Engineering Chemistry, November, 1936, page 1357
Results of Vartessian and Fenske

Temperature 18°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_C	C_W	$K = C_W/C_C$
0.208	0.416	2.0
0.0615	0.208	3.38
0.43	0.52	1.21
0.63	0.416	0.66

Data from Solubilities of Organic Compounds, by Seidell
Results of Herz and Lewy on page 105

Temperature 25°C .

C_c	C_w	$K = \frac{C_w}{C_c}$
0.000925	0.0208	22.5
0.00325	0.0415	12.85
0.0062	0.0624	10.05
0.0101	0.0831	8.22
0.01485	0.104	7
0.0206	0.1248	6.05
0.053	0.208	3.92
0.106	0.312	2.94
0.159	0.416	2.62
0.228	0.52	2.28
0.41	0.543	1.325

Data from the International Critical Tables, Volume V

Temperature 20°C.

0.0001247	0.00451	36.2
0.000249	0.00777	31.2
0.0003735	0.01035	27.7
0.000498	0.01258	25.25
0.000623	0.01465	23.5
0.000748	0.0165	22.1
0.001247	0.02315	18.55
0.00249	0.035	14.05
0.003735	0.0446	11.95
0.00498	0.0527	10.60
0.00623	0.0603	9.68

TABLE XII (CONT'D)

Data from the International Critical Tables, Volume V (Cont'd)
Temperature 20°C.

C_c	C_w	$K = \frac{C_w}{C_c}$
0.00748	0.0669	8.94
0.00872	0.073	8.36
0.00997	0.0781	7.84
0.0112	0.831	7.43
0.01247	0.0885	7.10
0.0137	0.0935	6.82
Temperature = 25°C.		
0.00187	0.0298	15.92
0.00249	0.0358	14.36
0.00374	0.0461	12.33
0.00498	0.0546	10.98
0.00623	0.0624	10.02
0.00748	0.0697	9.32
0.00872	0.0765	8.77
0.00997	0.0826	8.29
0.012	0.0942	7.86
0.01247	0.0935	7.50
0.0137	0.0985	7.19
0.01495	0.104	6.94
0.0162	0.109	6.73
0.0249	0.1383	5.55
0.0436	0.194	4.45
0.0623	0.2365	3.8
0.0935	0.299	3.2
0.1247	0.349	2.8

Data from Solubilities of Organic Compounds, by Seidell
Results of Bektourov on page 108

Temperature = 0°C.

C_c	C_w	K
0.000515	0.01105	21.44
0.00162	0.0243	15.0
0.0072	0.0559	7.76
0.0206	0.103	5.00
0.0649	0.215	3.32
0.135	0.356	2.64
0.222	0.484	2.18
0.27	0.543	2.02
0.331	0.59	1.78

TABLE XII (CONT'D)

Data from Solubilities of Organic Compounds, by Seidell (Cont'd)

Temperature = 50°C.

C_c	C_w	K
0.000221	0.00441	20.0
0.000735	0.0118	16.0
0.001178	0.01545	13.1
0.0081	0.0567	7.0
0.0287	0.137	4.77
0.081	0.268	3.31
0.147	0.405	2.75
0.2105	0.502	2.38
0.304	0.581	1.92

Definition:

C_c - concentration of acetic acid in chloroform

C_w - concentration of acetic acid in water

TABLE XIII

MUTUAL SOLUBILITY DATA FOR
CHLOROFORM - ACETIC ACID- WATER

Data from Industrial and Engineering Chemistry, November, 1936 page 1355.

Results of Vartession and Fenske

Temperature = 18°C

Points were taken off of Figure 2 in the above report and a corrosponding triangular diagram prepared.

TABLE XIV

DISTRIBUTION DATA FOR TOLUENE-ACETIC ACID-WATER

Data determined experimentally at Oak Ridge National Laboratory by W. S. Farmer.

Temperature = 21°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_T	C_W	$K = C_W/C_T$
0.0256	0.407	15.9
0.0102	0.215	21
0.00312	0.0905	29
0.00118	0.0480	40.6
0.000474	0.0247	52.1
0.0000748	0.00505	67.5
0.0000374	0.00211	56.3
0.0000249	0.00131	52.6

Data from the International Critical Tables, Volume V

Temperature 25°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_T	C_W	$K = C_W/C_T$
0.000623	0.00125	50
0.00623	0.0274	22.7
0.0187	0.131	14.3
0.0311	0.27	11.5
0.0436	0.44	9.9
0.0623	0.744	8.37
0.081	1.1	7.35

TABLE XIV (CONT'D)

Data from Solubilities of Organic Chemicals, by Seidell
Results of Herz and Fischer on page 106

Temperature 25°C

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_T	C_W	$K = C_W/C_T$
0.00124	0.052	42
0.00341	0.104	30.5
0.0118	0.208	17.7
0.0236	0.312	13.2
0.0388	0.416	10.7
0.0607	0.52	8.57
0.0868	0.624	7.2

Definition:

C_T - concentration of acetic acid in toluene

C_W - concentration of acetic acid in water

Data from Solubilities of Organic Chemicals, by Seidell
Results of de Kolossowsky and Mengenineon on page 112

Temperature 25°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_T	C_W	$K = C_W/C_T$
0.00234	0.06	25.6
0.00645	0.118	18.3
0.012	0.175	14.6
0.0185	0.231	12.5
0.0259	0.285	11.0
0.0353	0.339	9.7
0.0521	0.447	8.55
0.0758	0.546	7.21
0.109	0.64	5.88
0.157	0.715	4.51
0.237	0.76	3.20
0.779	0.779	1.0

TABLE XV

DISTRIBUTION DATA FOR ISOPROPYL ETHER-ACETIC ACID-WATER

Data from Industrial and Engineering Chemistry, October, 1941, pages 1240-1248
Results of D. F. Othmer, R. E. White, and E. Trueger

Temperature 25°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_I	C_W	$K' = C_I/C_W$
7.08	22.8	0.311
4.59	16.7	0.274
12.6	34.8	0.362
22.7	48.1	0.472
29.4	43.7	0.673

Data determined experimentally at Oak Ridge National Laboratory by W. S. Farmer.

Temperature 20°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_I	C_W	$K' = C_I/C_W$
0.0107	0.0516	0.207
0.00838	0.0410	0.205
0.00549	0.0282	0.195
0.00271	0.013	0.208
0.00107	0.00554	0.239
0.000536	0.00274	0.196
0.000474	0.00237	0.2
0.0613	0.229	0.268

TABLE XV (CONT'D)

Data from Transactions of the American Institute of Chemical Engineers, 31, 667 (1935)

Results of Elgin and Browning

Temperature 20°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_I	C_W	$K' = C_I/C_W$
0.00372	0.02	0.186
0.0078	0.04	0.195
0.0168	0.08	0.21
0.028	0.12	0.233
0.0411	0.16	0.257

Data from Solubilities of Organic Chemicals, by Seidell
Results of Smith and Elgin on page 112

Temperature 20°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_I	C_W	$K' = C_I/C_W$
0.000847	0.00456	0.185
0.000873	0.00489	0.178
0.00158	0.00904	0.177
0.00349	0.0186	0.188
0.0036	0.0189	0.190
0.0074	0.0375	0.196
0.00891	0.0431	0.206
0.0108	0.0554	0.196
0.0143	0.069	0.207
0.0175	0.0847	0.206
0.0203	0.0891	0.228
0.0249	0.112	0.222
0.042	0.173	0.242

TABLE XVI

MUTUAL SOLUBILITY DATA FOR ISOPROPYL ETHER-ACETIC ACID-WATER

Data from Industrial and Engineering Chemistry, October, 1941, pages 1240-1248

Results of D. F. Othmer, R. E. White and E. Trueger

Temperature 23-24°C.

Mutual Solubility Data			Tie Line Data	
Isopropyl Ether %	Acetic Acid %	Water %	Isopropyl Ether Layer Acetic Acid %	Water Layer Acetic Acid %
50.3	36.8	13.0	9.4	21.9
31.05	45.1	23.8	6.1	16.1
16.7	48.4	34.9	16.75	33.5
13.25	48.1	38.6	30.2	46.3
41.85	47.3	10.85	39.0	42.1
3.5	37.6	58.8		

TABLE XVII

DISTRIBUTION DATA FOR CARBON TETRACHLORIDE-PROPIONIC ACID-WATER

Data from Solubilities of Organic Chemicals, by Seidell
Results of Kolossowski, Bekturof and Kulikow on page 187

Temperature 25°C.

Concentration of Acetic Acid Lb. Moles/Cu. Ft.		Distribution Coefficient
C_C	C_W	$K = C_W/C_C$
0.0000249	0.000804	31.5
0.000151	0.00229	15.1
0.000547	0.00559	10.2
0.0023	0.0131	5.68
0.00797	0.0277	3.48
0.0176	0.0447	2.53
0.0644	0.0953	1.48
0.102	0.13	1.28
0.153	0.18	1.18
0.219	0.26	1.19
0.26	0.323	1.24
0.37	0.447	1.20

TABLE XVIII

MUTUAL SOLUBILITIES OF SOLVENTS IN WATER AT 25°C

Solvent	Solubility grs. / 100 mls. of water
Chloroform[2b]	0.62
Methyl Isobutyl Ketone[1]	2.0
Benzene[2c]	0.18
Toluene[2d]	0.05
Carbon Tetrachloride[2a]	0.08
Isopropyl Ether[1]	0.90

Solvent	Solubility grs. / 100 mls. of solvent
Chloroform[2b]	0.15
Methyl Isobutyl Ketone[1]	2.2
Benzene[2c]	0.06
Toluene[2d]	0.05
Carbon Tetrachloride[2a]	0.016
Isopropyl Ether[1]	0.57

Data from:

1- Synthetic Organic Chemicals, Carbide and Carbon Chemicals Corporation.

2- Solubilities of Organic Chemical, by Seidell.

(a) Results of Gross on page 2.
(b) Results of Kerz on page 12.
(c) Results on page 368.
(d) Results on page 542.

TABLE XXIV

SERIES A

EXTRACTION VERSUS DROP DIAMETER
CARBON TETRACHLORIDE-ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter Inches	K ft./hr.	C_f lb.moles/cu.ft.	C_R lb.moles/cu.ft.	% Extraction	H ft.	V Ft./sec	T_c °C
1-A	5mm. Cap.	0.106	0.41	0.01075	0.00538	50	4.917	0.562	21
2-	5mm. Cap.	0.105	0.35	0.01075	0.00601	44.3	4.917	0.562	21.5
3-	5mm. Cap.	0.107	0.34	0.01075	0.00612	43	4.917	0.562	22
4-	6mm. Cap.	0.126	0.33	0.01075	0.00692	35.5	4.917	0.585	22.5
5-	6mm. Cap.	0.127	0.39	0.01075	0.00641	40.4	4.917	0.585	23.5
6-	6mm. Cap.	0.126	0.33	0.01075	0.00685	36.3	4.917	0.585	23
7-	7mm. Cap.	0.14	0.34	0.01075	0.00715	33.4	4.875	0.585	22.5
8-	7mm. Cap.	0.138	0.30	0.01075	0.0075	30.3	4.875	0.585	22
9-	7mm. Cap.	0.14	0.31	0.01075	0.0075	30.2	4.875	0.585	22
10-	6mm. Tube	0.188	0.37	0.01075	0.0077	28.3	4.875	0.56	23
11-	6mm. Tube	0.189	0.42	0.01075	0.00734	31.7	4.875	0.56	22
12-	7mm. Tube	0.204	0.34	0.01075	0.0081	22.8	4.1875	0.537	21.5
13-	7mm. Tube	0.203	0.30	0.0105	0.00824	21.5	4.1875	0.537	22
14-	8mm. Tube	0.223	0.375	0.0105	0.0081	23	4.416	0.565	21.5
15-	8mm. Tube	0.22	0.36	0.0105	0.00813	22.6	4.416	0.565	21.5
16-	7mm. Cap.	0.144	0.325	0.0108	0.00747	30.8	4.875	0.587	20
17-	7mm. Cap.	0.146	0.31	0.0108	0.00761	29.6	4.875	0.587	21.5
18-	6mm. T.D.D.	0.166	0.30	0.0108	0.00795	26.4	4.958	0.583	22
19-	6mm. T.D.D.	0.164	0.30	0.0108	0.0079	26.8	4.958	0.583	22
20-	6mm. T.C.	0.083	0.43	0.0108	0.00505	53.2	4.915	0.53	22.5
21-	6mm. T.C.	0.084	0.316	0.01064	0.00525	50.8	4.916	0.518	20
22-	7mm. Tube	0.213	0.35	0.01064	0.00814	23.5	4.75	0.566	21
23-	8mm. Tube	0.22	0.36	0.01064	0.0081	23.8	4.79	0.58	21

TABLE XXIV (CONT'D)

SERIES A

EXTRACTION VERSUS DROP DIAMETER
CARBON TETRACHLORIDE-ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	L cu.ft./hr.	A sq.ft.	C_W lb.moles/cu.ft.	T_d °C
1-A	0.00286	0.0047	0.000154	
2-	0.00273	0.00457	0.00018	
3-	0.00294	0.00488	0.000141	
4-	0.00484	0.0065	0.000141	
5-	0.00493	0.00652	0.000154	
6-	0.00469	0.00635	0.000154	
7-	0.00637	0.00769	0.000154	
8-	0.00596	0.00726	0.000192	
9-	0.00623	0.00742	0.000155	
10-	0.0146	0.0134	0.000155	
11-	0.01505	0.0148	0.000179	
12-	0.0197	0.01516	0.0000834	
13-	0.0179	0.01392	0.000143	
14-	0.0207	0.01468	0.000179	
15-	0.0231	0.01652	0.000179	
16-	0.00743	0.00856	0.0001665	
17-	0.00688	0.01176	0.000165	
18-	0.0106	0.01092	0.000165	31
19-	0.01	0.0106	0.0001525	31
20-	0.00178	0.003888	0.000165	26
21-	0.00161	0.0036	0.0001505	22.5
22-	0.0233	0.0178	0.000225	26
23-	0.0231	0.0174	0.000225	29

TABLE XXIV (CONT'D)

SERIES A

EXTRACTION VERSUS DROP DIAMETER
CARBON TETRACHLORIDE-ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp. No.	θ_F secs.	θ_T secs.
1-A	0.452	8.75
2-	0.46	8.75
3-	0.448	8.75
4-	0.448	8.4
5-	0.452	8.4
6-	0.456	8.4
7-	0.464	8.35
8-	0.48	8.35
9-	0.48	8.35
10-	0.50	8.7
11-	0.492	8.7
12-	0.468	7.8
13-	0.504	7.8
14-	0.58	7.8
15-	0.50	7.8
16-	0.44	8.3
17-	0.496	8.3
18-	0.47	8.5
19-	0.472	8.5
20-	0.36	9.3
21-	0.408	9.5
22-	0.452	8.4
23-	0.50	8.25

TABLE XXV

SERIES B

EXTRACTION VERSUS DROP RATE (θ_t)
CARBON TETRACHLORIDE-ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter Inches	K ft./hr.	C_f lb.moles/cu.ft.	C_R lb.moles/cu.ft.	% Extraction	H ft.
1-B	7mm. Cap.	0.147	0.275	0.01065	0.00774	27.3	4.897
2-	7mm. Cap.	0.151	0.272	0.01065	0.00791	25.7	4.897
3-	7mm. Cap.	0.151	0.286	0.01065	0.00787	26.2	4.897
4-	7mm. Cap.	0.145	0.322	0.01065	0.00733	31.2	4.916
5-	7mm. Cap.	0.147	0.326	0.01065	0.00735	31.0	4.916
6-	7mm. Cap.	0.151	0.329	0.01065	0.00743	30.2	4.916
7-	7mm. Cap.	0.148	0.335	0.01065	0.00736	31.3	4.916

V ft./sec.	L cu.ft./hr.	A sq.ft.	C_w lb.moles/cu.ft.	T_c °C	T_d °C	θ_f sec.
0.593	0.00804	0.00917	0.000125	21.5	24.5	0.424
0.593	0.0157	0.0171	0.00015	21	26.5	0.24
0.593	0.0106	0.0114	0.00015	22	27	0.36
0.593	0.0048	0.0056	0.000125	21.5	31	0.68
0.593	0.00591	0.00675	0.000113	22	32	0.58
0.593	0.00627	0.00688	0.000163	22	26	0.60
0.593	0.00307	0.00344	0.000147	21	22	1.16

TABLE XXVI

SERIES C

EXTRACTION VERSUS DISPERSED PHASE TEMPERATURE
CARBON TETRACHLORIDE-ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter Inches	K ft./hr.	C_f lb.moles/cu.ft.	C_r lb.moles/cu.ft.	% Extraction	H Ft.
1-C	7mm. Cap.	0.148	0.265	0.0107	0.00776	27.5	4.916
2-	7mm. Cap.	0.15	0.232	0.0107	0.00767	28.3	4.916
3-	7mm. Cap.	0.149	0.337	0.0107	0.00737	31.1	4.916
4-	7mm. Cap.	0.151	0.347	0.0107	0.00724	32.3	4.916

V ft./sec.	L cu.ft./hr.	A sq.ft.	C_w lb.moles/cu.ft.	T_c °C	T_d °C	θ_f secs.
0.596	0.00733	0.0082	0.00015	21	22	0.48
0.596	0.0087	0.00965	0.0001472	21	25	0.42
0.596	0.00875	0.00965	0.0001472	22	29	0.414
0.596	0.00891	0.00975	0.0001595	22	30	0.42

TABLE XXVII

SERIES D

EXTRACTION VERSUS CONTINUOUS PHASE TEMPERATURE
CARBON TETRACHLORIDE-ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter Inches	K ft./hr.	C_f lb.moles/cu.ft.	C_r lb.moles/cu.ft.	% Extraction	H Ft.
1-D	7mm. Cap.	0.147	0.361	0.01085	0.00717	33.9	4.959
2-	7mm. Cap.	0.149	0.384	0.01085	0.00704	35.1	4.959
3-	7mm. Cap.	0.149	0.44	0.0108	0.00653	39.5	4.959
4-	7mm. Cap.	0.15	0.443	0.0106	0.00661	37.7	4.959
5-	7mm. Cap.	0.149	0.32	0.01035	0.0072	30.5	4.959
6-	7mm. Cap.	0.15	0.421	0.00977	0.00616	36.9	4.959

V ft./sec.	L cu.ft./hr.	A sq. ft.	C_w lb.moles/cu.ft.	T_c °C	T_d °C	θ_f secs.
0.597	0.00726	0.00837	0.0001477	26.5	25.5	0.468
0.597	0.00775	0.00872	0.0001477	33.5	28.5	0.46
0.597	0.00721	0.00818	0.000246	46	29	0.49
0.597	0.00784	0.0087	0.00015	35	30	0.468
0.597	0.00735	0.00837	0.00015	31	25	0.48
0.597	0.00806	0.00885	0.00015	36.5	29	0.46

TABLE XXVIII

SERIES E

EXTRACTION VERSUS COLUMN HEIGHT
CARBON TETRACHLORIDE-ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter Inches	K ft./hr.	C_t Lb.moles/cu.ft.	C_r Lb.moles/cu.ft.	% Extraction	H ft.
1-E	7mm. Cap.	0.152	0.652	0.0105	0.00714	32	3.75
2-	7mm. Cap.	0.1495	0.61	0.01035	0.00766	26	3.04
3-	7mm. Cap.	0.164	0.94	0.0102	0.00785	23	1.917
4-	7mm. Cap.	0.164	1.03	0.00997	0.00837	16	0.833

V ft./sec.	L cu.ft./hr.	A sq. ft.	C_w lb.moles/cu.ft.	T_c °C	T_d °C	θ_f secs.
0.595	0.00722	0.0058	0.0001745	22	24	0.548
0.575	0.00695	0.00498	0.000312	21	23.5	0.52
0.59	0.0119	0.00477	0.000312	21	23.5	0.40
0.417	0.0101	0.00244	0.000349	20	23	0.48

TABLE XXIX

SERIES F

EXTRACTION VERSUS ACETIC ACID CONCENTRATION
CARBON TETRACHLORIDE - ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter inches	K ft./hr.	C_t lb.moles/cu.ft.	C_r lb.moles/cu.ft.	Ln. ΔC.	C_B lb.moles/cu. ft.	C_T lb.moles/cu. ft.
F-1	7 mm Cap.	0.15	0.349	0.0105	0.00724	0.00878	0.000137	0.000187
F-2	7 mm Cap.	0.151	0.385	0.00772	0.005	0.00625	0.0000996	0.000214
F-3	7 mm Cap.	0.152	0.507	0.00434	0.00259	0.00339	0.0000685	0.000112
F-4	7 mm Cap.	0.155	0.519	0.00187	0.00112	0.00146	0.0000435	0.0000622
F-5	7 mm Cap.	0.157	0.596	0.000686	0.000386	0.000523	0.0000249	0.0000324
F-6	*7 mm Cap.	0.153	2.95	0.0565	0.00193	0.0161	0.000585	0.00448

% Extraction	H ft.	V ft./sec.	L cu.ft./hr.	A sq.ft.	T_c °C	T °C	T_d	θ_t seconds	θ_f seconds
31	4.875	0.61	0.00749	0.00797	17	19	24	8.0	0.492
35.2	4.875	0.61	0.0075	0.00795	17.5	22.5	25	8.0	0.5
40.2	4.875	0.633	0.0076	0.00775	17.5	21.5	25	7.7	0.5
40.2	4.875	0.633	0.00827	0.0082	17.5	26	28	7.7	0.492
43.7	4.875	0.65	0.00884	0.00852	17.5	26	28	7.5	0.472
96.7	4.875	0.56	0.00878	0.0101	17.5	27	28	8.7	0.44

NOTE: * Exceeded acetic acid solubility in CCL_4.

TABLE XXX

SERIES G

EXTRACTION VERSUS CONTINUOUS PHASE FLOW RATE
CARBON TETRACHLORIDE - ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter inches	K ft./hr.	C_f lb.moles/cu.ft.	C_R	$\ln. \Delta C.$	C_T	% Extraction	H ft.	V ft/sec.
G-1	7 mm Cap.	0.146	0.347	0.01051	0.00748	0.00892	0.0000748	28.9	4.33	0.61
G-2	7 mm Cap.	0.147	0.350	0.01051	0.00743	0.00888	0.0000249	29.3	4.33	0.63
G-3	7 mm Cap.	0.150	0.362	0.01051	0.00723	0.00875	0.0000187	31.2	4.33	0.624
G-4	7 mm Cap.	0.149	0.382	0.01051	0.00713	0.0087	0.0000187	32.2	4.33	0.625
G-5	7 mm Cap.	0.149	0.394	0.01051	0.00716	0.00873	0.0000245	31.8	4.33	0.618
G-6	7 mm Cap.	0.1495	0.37	0.01051	0.00735	0.00883	0.0000245	30	4.33	0.616
G-7	7 mm Cap.	0.15	0.368	0.01051	0.00748	0.00892	0.000147	28.9	4.33	0.619

L cu.ft./hr.	A sq. ft.	L_c ft/hr	Red	T_C °C	T °C	T_d °C	θ_t secs.	θ_f secs.	Re_C to H_2O	V_C H_2O Velocity ft./sec.
0.00706	0.0069	30	661	18.5	26.5	27.5	7.1	0.48	9.05	0.0084
0.00712	0.00704	100	657	18.5	26	27.6	7.2	0.48	30.2	0.0276
0.00722	0.00746	193	633	18.5	26	28	7.6	0.50	59.4	0.0535
0.00669	0.00681	199	630	18.5	27	28.5	7.6	0.54	61	0.0552
0.0067	0.00653	147	627	18.5	25.5	27.5	7.5	0.532	44.2	0.0407
0.00697	0.00676	52.7	668	18.5	26	27.5	7.2	0.52	16.25	0.0146
0.00688	0.00635	0.0	690	18.5	25	27	7.0	0.54	0.0	0.0

TABLE XXXI

SERIES H

EXTRACTION VERSUS SODIUM HYDROXIDE CONCENTRATION IN CONTINUOUS PHASE
CARBON TETRACHLORIDE-ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter Inches	K ft./hr.	C_t lb.moles/cu.ft.	C_r lb.moles/cu.ft.	Ln. ΔC.
H-1	7 mm Cap.	0.15	*	0.0	0.0	
H-2	7 mm Cap.	0.15	0.387	0.0105	0.0105	0.0086
H-3	7 mm Cap.	0.15	0.485	0.0104	0.0064	0.00825
H-4	7 mm Cap.	0.15	*	0.0	0.0	
H-5	7 mm Cap.	0.15	0.46	0.0105	0.0065	0.00843
H-6	7 mm Cap.	0.149	0.43	0.0105	0.00666	0.00845

Column at Start	Free NaOH Bottom at end lb. moles/cu. ft.	Top at end	C_B lb. moles/cu.ft.	C_T lb. moles/cu.ft.	% Extraction
0.000104	0.000104	0.000104	0.0	0.0	0.0
0.000083	0.00005	0.0	0.000033	0.00012	35.3
0.0	0.0	0.0	0.000125	0.00026	38
0.00063	0.00063	0.00063	0.0	0.0	0.0
0.00063	0.000518	0.000518	0.000112	0.000112	37.7
0.0	0.0	0.0	0.000125	0.000249	36.6

H ft.	V ft./sec.	L cu.ft./hr.	A sq. ft.	T_c °C	T °C	T_d °C	θ_t seconds	θ_f seconds
4.875	0.61	0.00792	0.00908	17.5	22.5	25	8.0	0.46
4.875	0.59	0.00822	0.00904	17.5	23.0	26.0	8.25	0.448
4.875	0.61	0.00778	0.00831	17.5	23.0	26.0	8.0	0.472
4.875	0.633	0.00697	0.00713	17.5	16.0	21	7.7	0.53
4.875	0.633	0.0075	0.00766	17.5	17.0	22.5	7.7	0.493
4.875	0.633	0.0068	0.00727	17.5	20.0	21	7.8	0.52

Notes: * Blank run to determine NaOH transfer from H_2O to CCL_4. Negligible transfer was found to take place.

TABLE XXXII

SERIES J

EXTRACTION VERSUS INTERFACIAL TENSION
CARBON TETRACHLORIDE-ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter inches	K ft./hr.	C_t lb.moles/cu.ft.	C_r lb.moles/cu.ft.	Ln. Δ C.	Wetting Agent
J-1	7 mm Cap.	0.15	0.553	0.01053	0.00593	0.00798	
J-2	7 mm Cap.	0.129	0.325	0.01053	0.00693	0.0086	Tergitol No. 4
J-3	7 mm Cap.	0.145	0.313	0.0106	0.00756	0.009	Tergitol No. 4
J-4	7 mm Cap.	0.116	0.645	0.0106	0.00526	0.00763	Tergitol No. 4
J-5	7 mm Cap.	0.0965	0.342	0.01075	0.00514	0.00761	Tergitol No. 7
J-6	7 mm Cap.	0.125	0.285	0.01075	0.00723	0.00882	Tergitol No. 7
J-7	7 mm Cap.	0.152	0.361	0.01075	0.00724	0.0089	
J-8*	7 mm Cap.	0.0595	0.357	0.01075	0.00216	0.00536	Tergitol No. 7

	Conc. in H_2O	I. T.	C_B lb. moles/cu.ft.	C_T lb. moles/cu.ft.	% Extraction	H ft.	V ft./sec.	L cu.ft./hr.
J-1			0.0002	0.00019	43.7	4.875	0.625	0.00896
J-2	0.1% by Vol.	14.4	0.000162	0.000188	34.2	4.875	0.591	0.00588
J-3	0.01% by Vol.	32.3	0.0002	0.00015	28.7	4.875	0.625	0.0079
J-4	0.333% by Vol.	4.3	0.000162	0.000188	50.5	4.875	0.519	0.0075
J-5	0.1% by Vol.	2.0	0.000187	0.000162	52.3	4.875	0.473	0.00286
J-6	0.033% by Vol.	8.5	0.000125	0.000162	32.8	4.875	0.554	0.005
J-7		33.2	0.000162	0.000473	32.8	4.875	0.61	0.0087
J-8*	0.3% by Vol.	0.0	0.000237	0.00025	79.9	4.875	0.357	0.00153

A sq. ft.	T_c °C	T °C	T_d °C	θ_t seconds	θ_f seconds
0.00934	17.5	17.0	21.5	7.8	0.41
0.00757	17.5	18.0	22.0	8.25	0.392
0.0085	17.5	21.0	23.0	7.8	0.42
0.00815	17.5	20.5	23.0	9.4	0.34
0.00615	16.0	20.5	22.5	10.3	0.34
0.007	16.0	23.5	26.0	8.8	0.428
0.00915	16.0	23.5	27.0	8.0	0.44
0.00687	16.0	23.5	27.0	13.3	0.15

Note: * Foam at the interface.

TABLE XXXIII

SERIES K

EXTRACTION VERSUS DROP DIAMETER METHYL ISOBUTYL KETONE - ACETIC ACID (DISPERSED) WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter inches	K ft./hr.	C_t lb.moles/cu.ft.	C_r lb.moles/cu.ft.	Ln.ΔC.	C_B lb.moles/cu.ft.	C_T lb.moles/cu.ft.
k-2	6 mm Tube	0.182	1.5	0.01073	0.0015	0.00469	0.000287	0.000311
k-3	5 mm Cap.	0.11	1.22	0.0108	0.00102	0.00414	0.000311	0.000274
k-4	7 mm Cap.	0.141	1.48	0.01095	0.00111	0.0043	0.000237	0.000262
k-5	6 mm Cap.*	0.128	1.47	0.01078	0.000914	0.004	0.000249	0.000249
k-6	D.D. Tube	0.0859	0.698	0.01078	0.000178	0.00589	0.0002055	0.000081
k-7	7 mm Tube	0.1975	1.47	0.01078	0.00172	0.00494	0.000423	0.000386
k-8	7.3 mm Cap.**	0.137	1.56	0.01078	0.000934	0.00403	0.000237	0.000237

% Extraction	H ft.	V ft./sec.	L cu.ft./hr.	A sq.ft.	T_c °C	T °C	Td °C	θ_t seconds	θ_f seconds
86	4.083	0.34	0.01	0.0132	15	21	26.0	12.0	0.66
90.5	4.083	0.389	0.00306	0.00592	15	21	25	10.5	0.468
89.8	4.0	0.367	0.00623	0.00961	15	20.5	24	10.9	0.492
91.5	4.083	0.384	0.00474	0.00795	14	20	23	10.65	0.48
98.4	3.958	0.36	0.00157	0.00404	14.5	20	24	11	0.44
84	4.083	0.333	0.014	0.01745	14.5	20	24	12.3	0.6
91.4	4.0	0.377	0.0063	0.00988	14.5	20	24	10.6	0.44

NOTE:

* 6.3 mm O.D., 1.2 mm I.D.

** 1.5 mm bore

TABLE XXXIV

SERIES L

EXTRACTION VERSUS COLUMN HEIGHT
METHYL ISOBUTYL KETONE-ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter inches	K ft./hr.	C_t lb. moles/cu.ft.	C_r lb. moles/cu.ft.	Ln. Δ C.	C_T lb.moles/cu.ft.
L-1	7.3 mm Cap.*	0.138	1.575	0.01078	0.001535	0.00473	0.000361
L-2	7.3 mm Cap.	0.139	1.61	0.01095	0.00217	0.00542	0.000423
L-3	7.3 mm Cap.	0.141	1.885	0.0109	0.004	0.0069	0.000798
L-4	7.3 mm Cap.	0.149	1.94	0.0109	0.00697	0.00878	0.00116

% Extraction	H ft.	V ft./sec.	L cu.ft./hr.	A sq. ft.	T_c °C	T °C	T_d °C
85.7	3.167	0.372	0.0058	0.0072	14.3	20.0	24
80.1	2.5	0.352	0.0058	0.00595	14.5	20.5	24.5
63.3	1.291	0.345	0.00639	0.00339	14.5	20.0	25.0
36	0.54	0.309	0.00915	0.00211	14.5	20.0	25.5

θ_t seconds	θ_f seconds
8.5	0.492
7.1	0.50
3.75	0.48
1.75	0.40

Note: * 1.5 mm bore.

TABLE XXXV

SERIES M

EXTRACTION VERSUS DROP DIAMETER
WATER-ACETIC ACID (DISPERSED)
CARBON TETRACHLORIDE (CONTINUOUS)

Exp.	Nozzle	Drop Diameter inches	K ft./hr.	C_t lb.moles/cu.ft.	C_r lb.moles/cu.ft.	Ln.ΔC.	C_B lb.moles/cu.ft.	C_T lb.moles/cu.ft.
M-1	6.5 mm Cap.*	0.211	0.0124	0.0108	0.0107	0.01	0.75×10^{-5}	0.75×10^{-5}
M-2	Tube D.D. Small	0.104	0.0172	0.01075	0.01043	0.0101	8.75×10^{-5}	6.23×10^{-5}

% Extraction	H ft.	V ft./sec.	L cu.ft./hr.	A sq. ft.	T_C °C	T °C	T_d °C	θ_t seconds	θ_f seconds
0.933	4.083	0.48	0.0234	0.0188	13.5	19	22.0	8.5	0.44
2.98**	3.958	0.435	0.00253	0.00445	13.5	22.5	26.5	9.1	0.475

NOTE:

* 1.5 mm bore

** Based on total acid present and not amount extractable for equilibrium.

TABLE XXXVI

SERIES N

EXTRACTION VERSUS DROP DIAMETER
WATER (DISPERSED)
CARBON TETRACHLORIDE-ACETIC ACID (CONTINUOUS)

Exp.	Nozzle	Drop Diameter inches	K ft./hr.	C_f lb.moles/cu.ft.	C_r lb.moles/cu.ft.	Ln. ΔC.
N-1	Tube D.D. Med.	0.125	0.0413	0.0	0.01162	0.194
N-2	Tube D.D. Large	0.1825	0.038	0.0	0.00695	0.185
N-3	Tube D.D. Small	0.0973	0.0402	0.0	0.0144	0.178

C_W lb. moles/cu.ft.	C_B lb. moles/cu.ft.	C_T lb. moles/cu.ft.	% Extraction *	H ft.	V ft./sec.	L cu.ft./hr.	A sq. ft.
0.0105	0.0103	0.00985	5.8	4.0	0.444	0.00458	0.00665
0.01015	0.00998	0.00956	3.65	3.98	0.43	0.0154	0.01575
0.00953	0.00904	0.00842	7.78	3.96	0.406	0.00238	0.0048

T_d °C	T °C	T_c °C	θ_t seconds	θ_f seconds
22	19	13	9.0	0.46
27	22	13.5	9.25	0.428
29	23	14	9.75	0.42

Note: * Based on equilibrium.

TABLE XXXVII

SERIES O

EXTRACTION VERSUS DROP DIAMETER
BENZENE-ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter inches	K ft./hr.	C_t lb.moles/cu.ft.	C_r lb.moles/cu.ft.	Ln. ΔC.	C_B lb.moles/cu.ft.	C_T lb.moles/cu.ft.
O-1	6.4 mm Cap.*	0.249	0.375	0.00985	0.00706	0.00839	0.000125	0.00035
O-2	5 mm Cap.	0.174	0.299	0.00985	0.00643	0.00804	0.000106	0.0002985
O-3	Tube D.D. Med.	0.16	0.327	0.0104	0.00626	0.00816	0.000112	0.000324
O-4	Tube D.D. Small	0.122	0.275	0.01	0.00560	0.00932	0.000112	0.000249
O-5	6 mm Cap. D.D. #1	0.0813	0.246	0.0102	0.00347	0.00623	0.0000995	0.000324
O-6	6 mm Cap. D.D. #2	0.139	0.29	0.0102	0.00599	0.00792	0.0000995	0.000274
O-7	6 mm Cap. D.D. #1	0.0816	0.257	0.0105	0.00353	0.0064	0.000149	0.000423
O-8	Tube D.D. Small	0.129	0.294	0.0105	0.00572	0.00786	0.0000747	0.000224

% Extraction	H ft.	V ft./sec.	L cu.ft./hr.	A sq.ft.	T_c °C	T °C	T_d °C	θ_t seconds	θ_f seconds
28.4	4.083	0.371	0.029	0.0257	13	17.5	25	11	0.58
34.7	4.083	0.332	0.0114	0.0162	13	20	29	12.3	0.50
39.8	4.028	0.329	0.00788	0.0122	13.5	19	23	12.25	0.56
54	3.917	0.308	0.00394	0.00829	13	19	26	12.75	0.50
66	3.915	0.22	0.00124	0.00541	13	20	29	17.75	0.472
41.3	3.916	0.307	0.0061	0.0112	13	20	26	12.75	0.48
66.4	3.916	0.226	0.00122	0.00516	13	18	26	17.3	0.488
45.5	3.916	0.297	0.00435	0.009	13	18	25	13.2	0.527

NOTE:

* 1.5 mm. bore

TABLE XXXVIII

SERIES P

EXTRACTION VERSUS COLUMN HEIGHT
BENZENE-ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter inches	K ft./hr	C_t lb.moles/cu.ft.	C_r lb.moles/cu.ft.	Ln. Δ C.
P-1	Tube D.D. Med.	0.155	0.364	0.01042	0.0068	0.0085
P-2	Tube D.D. Med.	0.152	0.735	0.01042	0.00744	0.00886
P-3	Tube D.D. Med.	0.155	1.025	0.01068	0.00797	0.00927
P-4	Tube D.D. Med.	0.158	0.488	0.01035	0.007	0.00855
P-5	Tube D.D. Med.	0.158	0.419	0.0104	0.00706	0.00867
P-6	Tube D.D. Med.	0.156	0.214	0.00953	0.00675	0.00808

C_B * lb. moles/cu.ft.	C_T lb. moles/cu.ft.	% Extraction	H ft.	V ft./sec.	L cu.ft./hr.	A sq. ft.
0.0000374	0.000275	34.7	2.875	0.32	0.00837	0.0098
0.0000125	0.000436	28.6	1.042	0.307	0.00749	0.00343
0.00000623	0.000671	25.4	0.541	0.246	0.00747	0.00213
0.0000499	0.000361	32.2	1.896	0.30	0.00795	0.00639
0.0000623	0.000324	32.1	2.417	0.334	0.00795	0.00731
0.0000873	0.0003	29.2	3.95	0.316	0.00765	0.0123

T_c °C	T °C	T_d °C	θ_t seconds	θ_f seconds
13	20	26	9.0	0.48
13	20	27	3.4	0.5
13	19	25	2.2	0.54
13	17.5	26	6.33	0.54
13	18	26	7.25	0.54
13	18	26	12.5	0.54

Note: * Bottom sample point below level of the nozzle in the column.

TABLE XXXIX

SERIES Q

EXTRACTION VERSUS DROP DIAMETER
TOLUENE-ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter inches	K ft./hr.	C_t lb.moles/cu.ft.	C_r lb.moles/cu.ft.	Ln. ΔC	C_B lb.moles/cu.ft.	C_T lb.moles/cu.ft.
Q-1	Tube D.D. Small	0.1273	0.328	0.0102	0.00523	0.00745	0.0000998	0.000262
Q-2	6.4 mm Cap.*	0.255	0.655	0.0102	0.0058	0.00777	0.0000872	0.000212
Q-3	6.4 mm Cap.*	0.245	0.355	0.01035	0.00748	0.00883	0.0000997	0.000299
Q-4	5 mm Cap.	0.197	0.307	0.01035	0.00736	0.0088	0.0000997	0.00025
Q-5	Tube D.D. Med.	0.148	0.329	0.01035	0.00606	0.008	0.0000997	0.000287
Q-6	Tube D.D. Small #1	0.116	0.29	0.01037	0.00536	0.00759	0.000125	0.000299
Q-7	Tube D.D. Small #2	0.0962	0.293	0.01037	0.00459	0.00709	0.0000872	0.000287
Q-8	6 mm Cap. D.D.	0.0785	0.265	0.01013	0.00317	0.00598	0.000187	0.000449

% Extraction	H ft.	V ft./sec.	L cu.ft./hr.	A sq. ft.	T_c °C	T °C	T_d °C	θ_t seconds	θ_f seconds
48.7	3.917	0.301	0.00402	0.0082	13	18	27	13	0.56
43	4.083	0.371	0.0307	0.0265	13	18	25	11	0.587
27.8	4.042	0.376	0.032	0.0281	13	18	25	10.75	0.5
28.9	4.042	0.376	0.0176	0.0194	13	19	27	10.75	0.47
41.5	4.029	0.336	0.00725	0.01185	13	19	28	12	0.48
48.2	3.918	0.307	0.00324	0.00738	13	18	29	12.75	0.52
55.7	4.042	0.305	0.00232	0.00645	13	18	28	13.25	0.416
68.7	3.917	0.227	0.00128	0.0056	13	15	25	17.25	0.416

NOTE:

* 1.5 mm. bore

TABLE XL

SERIES R

EXTRACTION VERSUS COLUMN HEIGHT
TOLUENE-ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter inches	K ft./hr.	C_t lb. moles/cu. ft.	C_r lb. moles/cu. ft.	Ln. Δ C.
R-1	Tube D.D. Med.	0.156	0.31	0.01020	0.0066	0.00828
R-2	Tube D.D. Med.	0.157	0.343	0.01020	0.007	0.00854
R-3	Tube D.D. Med.	0.155	0.364	0.01020	0.00727	0.00868
R-4	Tube D.D. Med.	0.153	0.478	0.01020	0.00717	0.0086
R-5	Tube D.D. Med.	0.153	0.627	0.01025	0.00732	0.0087
R-6	Tube D.D. Med.	0.147	1.065	0.01025	0.00766	0.00885

C_B lb. moles/cu. ft.	C_T lb. moles/cu. ft.	% Extraction	H ft.	V ft./sec.	L cu.ft./hr.	A sq. ft.
0.0000499	0.00025	35.3	3.542	0.328	0.00795	0.01115
0.0000249	0.000225	31.4	2.833	0.333	0.00857	0.00937
0.0000374	0.00025	28.7	2.354	0.326	0.0083	0.00771
0.0000249	0.000299	29.7	1.875	0.335	0.00759	0.0056
0.0000125	0.000337	28.6	1.2085	0.295	0.00802	0.00431
0.00000749	0.00071	25.3	0.583	0.292	0.00755	0.00207

T_c °C	T °C	T_d °C	θ_t seconds	θ_f seconds
13	17	27	10.8	0.512
13	16	23	8.5	0.484
13	16	24	7.2	0.488
12	17	22	5.6	0.508
12	17	21	4.1	0.488
12	17	21	2.0	0.456

TABLE XLI

SERIES S

EXTRACTION VERSUS DROP DIAMETER
ISOPROPYL ETHER-ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter inches	K ft./hr.	C_t lb.moles/cu.ft.	C_r lb.moles/cu.ft.	Ln.ΔC.
S-1	7.5 mm Cap. (2 mm I.D.)	0.148	2.81	0.01058	0.000337	0.00297
S-2	7.5 mm Cap. (2 mm I.D.)	0.143	3.0	0.01058	0.000237	0.00272
S-3	7 mm Cap. (I.S. mm I.D.)	0.139	2.8	0.0106	0.00025	0.00276
S-4	6 mm Tube	0.197	2.85	0.0106	0.000542	0.00337
S-5	5.5 mm Cap. (1 mm I.D.)	0.144	3.1	0.01075	0.000218	0.0027
S-6	Tube D.D. Small	0.092	4.46	0.01065	0.000732	0.0037
S-7	Tube D.D. Med.	0.0925	8.08	0.01065	0.002	0.00492

C_B lb.moles/cu.ft.	C_T lb.moles/cu.ft.	% Extraction	H ft.	V ft./sec.	L cu.ft./hr.	A sq. ft.	T_c °C
0.000424	0.000199	96.9	4.0	0.445	0.00783	0.0096	12
0.000312	0.0000997	97.7	4.0	0.445	0.00764	0.00965	12
0.000349	0.0000997	97.6	4.0	0.43	0.00623	0.00833	12
0.000324	0.000199	94.8	4.094	0.401	0.0177	0.0185	12
0.000387	0.000112	97.9	4.083	0.45	0.00722	0.0091	12
0.0000062*	0.000735	93.1	1.0	0.364	0.00208	0.00125	11
0.0000062*	0.0022	81.2	0.292	0.292	0.00205	0.000446	12

T °C	T_d °C	θ_t seconds	θ_f seconds
20	30	9.0	0.448
20	30	9.0	0.412
16	24	9.3	0.47
16	22	10.2	0.468
16	22	9.1	0.452
17	25	2.75	0.408
17	25	1.0	0.42

NOTE:
* Bottom sample taken below the nozzle.

TABLE XLII

SERIES T

EXTRACTION VERSUS COLUMN HEIGHT
ISOPROPYL ETHER-ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter inches	K ft./hr.	C_t lb.moles/cu.ft.	C_r lb.moles/cu.ft.	Ln. Δ C.
T-1	7.5 mm Cap. (2 mm I.D.)	0.181	4.68	0.01065	0.00569	0.00793
T-2	7.0 mm Cap. (1.5 mm I.D.)	0.1445	4.5	0.01065	0.00573	0.0079
T-3	7.0 mm Cap. (1.5 mm I.D.)	0.1445	4.93	0.01065	0.00193	0.0051
T-4	7.0 mm Cap. (1.5 mm I.D.)	0.131	4.18	0.01065	0.00112	0.00424

C_B * lb.moles/cu.ft.	C_T lb.moles/cu.ft.	Column Conc. by Difference	% Extraction	H ft.	V ft./sec.	L cu.ft./hr.
0.0000312	0.0000498	0.000897	46.5	0.333	0.277	0.0136
0.0000312	0.0000312	0.001108	46.3	0.313	0.313	0.00768
0.0000125	0.00143		82	1.021	0.408	0.00747
0.0000375	0.000985		89.3	1.48	0.423	0.00555

A sq. ft.	T_c °C	T °C	T_d °C	θ_t seconds	θ_f seconds
0.00182	12	17	25	1.2	0.472
0.00107	12	17	25	1.0	0.428
0.00259	12	17	25	2.5	0.44
0.00298	12	17	25	3.5	0.44

Note: * Sample tits located outside region being used for column contact.

TABLE XLIII

SERIES U

EXTRACTION VERSUS DROP DIAMETER
CHLOROFORM-ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter inches	K ft./hr.	C_t lb.moles/cu.ft.	C_r	Ln. Δ C.	C_B lb.moles/cu.ft.	C_T
U-1	6.4 mm Cap. (1.5 mm I.D.)	0.127	1.43	0.01078	0.00142	0.00462	0.000312	0.00066
U-2	Tube D.D. Large	0.1695	1.88	0.01078	0.001373	0.00456	0.000187	0.000848
U-3	5.5 mm Cap. (1 mm I.D.)	0.12	1.01	0.01078	0.00243	0.0056	0.000224	0.000499
U-4	Tube D.D. Med.	0.0993	0.595	0.0112	0.00354	0.00665	0.000274	0.000362
U-5	Tube D.D. Small	0.0825	0.452	0.011	0.00347	0.00651	0.000299	0.000386
U-6	6.3 mm Tube (4.5 mm I.D.)	0.199	1.2	0.011	0.00347	0.00651	0.000224	0.000673
U-7	7 mm Cap. (1 mm I.D.)	0.159	1.02	0.01105	0.00347	0.00653	0.0002	0.000536
U-8	7.7 mm Cap. (2 mm I.D.)	0.136	1.035	0.0111	0.00297	0.00615	0.000112	0.000262
U-9	6.4 mm Cap. (1.5 mm I.D.)	0.1275	0.835	0.0111	0.0034	0.00648	0.000112	0.000312
U-10	5.5 mm Cap.	0.1275	1.04	0.0111	0.00252	0.00576	0.0002	0.00045
U-11	6.4 mm Cap.	0.132	0.92	0.111	0.0032	0.00632	0.0000872	0.000461

% Extraction	H ft.	V ft./sec.	L cu.ft./hr.	A sq. ft.	T_c °C	T °C	T_d °C	θ_t seconds	θ_f seconds
86.9	4.833	0.537	0.00494	0.007	12	22	24	9.0	0.452
87.2	4.833	0.525	0.011	0.0121	12	23	25	9.2	0.48
77.5	4.833	0.542	0.00377	0.0056	12	23	25	8.9	0.5
68.5	4.75	0.5	0.0024	0.00464	12	20	23	9.5	0.44
68.4	4.79	0.456	0.001325	0.00339	10	23	26	10.5	0.46
68.4	4.833	0.508	0.01515	0.01465	11	23	26	9.5	0.56
68.6	4.79	0.532	0.00824	0.0094	10	24	27.5	9.0	0.528
73.2	4.79	0.55	0.00571	0.0073	10	22	26	8.7	0.48
69.4	4.833	0.53	0.00462	0.00659	10	25	29	9.1	0.488
77	4.833	0.537	0.00473	0.00675	10	21.5	25	9.0	0.472
71.2	4.833	0.537	0.00478	0.00647	10	22	25	9.0	0.528

TABLE XLIV

SERIES V

EXTRACTION VERSUS COLUMN HEIGHT
CHLOROFORM-ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter inches	K ft./hr.	C_t lb.moles/cu.ft.	C_r lb.moles/cu.ft.	ln.ΔC	C_B lb.moles/cu.ft.	C_T lb.moles/cu.ft.
V-1	7 mm Cap. (1 mm I.D.)	0.123	1.25	0.0111	0.00262	0.00587	0.000499	*
V-2	7.7 mm Cap. (2 mm I.D.)	0.1415	1.52	0.01087	0.00262	0.00586	0.000336	0.000025
V-3	7.7 mm Cap. (2 mm I.D.)	0.142	1.93	0.01087	0.00257	0.00575	0.000473	0.0000063
V-4	7.7 mm Cap. (2 mm I.D.)	0.14	2.33	0.01087	0.00308	0.00615	0.000723	0.0000063
V-5	7.7 mm Cap. (2 mm I.D.)	0.141	2.93	0.01087	0.00384	0.00675	0.000997	0.0000063
V-6	7.7 mm Cap. (2 mm I.D.)	0.142	3.13	0.01087	0.00556	0.00795	0.000673	0.0000063
V-7	7.7 mm Cap. (2 mm I.D.)	0.141	5.77	0.01087	0.00685	0.00868	0.0000125	0.0000063
V-8	7.7 mm Cap. (2 mm I.D.)	0.14	1.37	0.011	0.00551	0.0116	0.000622	0.0000063
V-9	7.7 mm Cap. (2 mm I.D.)	0.14	1.71	0.01097	0.00366	0.00665	0.000511	0.0000188
V-10	7.7 mm Cap. (2 mm I.D.)	0.14	1.66	0.01097	0.00262	0.00583	0.000399	0.000025
V-11	7.7 mm Cap. (2 mm I.D.)	0.141	1.51	0.01097	0.00166	0.00493	0.0002	0.000648

% Extraction	H ft.	V ft./sec.	L cu.ft./hr.	A sq. ft.	T_c °C	T °C	T_d °C	θ_t seconds	θ_f seconds
76.3	3.625	0.51	0.00404	0.00467	10	22.5	24	7.1	0.50
75.1	3.667	0.565	0.00669	0.00617	11	19	19	6.5	0.46
76.3	2.915	0.55	0.00697	0.00521	11	20	20	5.3	0.448
71.5	2.0	0.526	0.00665	0.00362	11	21	22	3.8	0.448
64.5	1.333	0.532	0.0067	0.00238	11	22	24	2.5	0.456
48.8	0.75	0.5	0.00674	0.00144	11	20	21	1.5	0.46
37	0.292	0.522	0.00667	0.000535	11	21	21	0.56	0.452
50	1.125	0.47	0.00673	0.00233	12	23	24	2.4	0.44
66.5	2.5	0.555	0.0067	0.0043	11	20	20	4.5	0.448
76	3.125	0.521	0.00679	0.00582	11	20	20	6.0	0.44
85	4.77	0.549	0.00668	0.00835	11	21	22	8.7	0.452

NOTE:

* Sample tit above water level.

TABLE XLV

SERIES W

EXTRACTION VERSUS DROP DIAMETER (END EFFECT)
CARBON TETRACHLORIDE-ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter inches	K ft./hr.	C_t lb.moles/cu.ft.	C_r lb.moles/cu.ft.	Ln. Δ C.	C_w lb.moles/cu.ft.
W-1	Tube D. D. Large	0.153	1.06	0.01023	0.0089	0.00952	0.000276
W-2	Tube D. D. Medium	0.0973	1.17	0.01023	0.00793	0.00904	0.000326
W-3	5.5 mm O.D., 1 mm I.D.	0.106	1.275	0.0103	0.00809	0.00906	0.000313
W-4	7 mm O.D., O.D. 1 mm I.D.	0.117	1.1	0.0103	0.00853	0.00936	0.000276
W-5	7.5 mm O.D., 2 mm I.D.	0.14	0.965	0.0103	0.00885	0.0095	0.000251
W-6	Cap. D. D. Small	0.102	1.37	0.0103	0.00787	0.009	0.000351
W-7	6 mm O.D., 4.5 mm I.D.	0.195	0.865	0.01037	0.00937	0.0098	0.000276
W-8	7 mm O.D., 5 mm I.D.	0.21	1.05	0.01037	0.0092	0.00972	0.000338
W-9	8 mm O.D., 6 mm I.D.	0.224	0.944	0.01037	0.0093	0.00968	0.000326

% Extraction	H ft.	V ft./sec.	L cu.ft./hr.	A sq. ft.	T_c °C	T_d °C	θ_t seconds	θ_f seconds
13	0.411	0.411	0.0078	0.001028	20	23	1.0	0.50
22.5	0.438	0.417	0.00206	0.000448	19.5	24.5	1.05	0.486
21.5	0.427	0.427	0.00266	0.00051	18.5	25	1.0	0.48
17.2	0.416	0.416	0.00355	0.0006115	18	23.5	1.0	0.489
14.1	0.416	0.378	0.00609	0.000963	19	23	1.1	0.489
23.6	0.406	0.406	0.00246	0.000484	19	25	1.0	0.47
9.64	0.443	0.385	0.018	0.00213	18.5	21	1.15	0.448
11.3	0.4685	0.391	0.0204	0.00235	18	21.5	1.2	0.492
10.3	0.416	0.32	0.0243	0.00285	18	21.5	1.3	0.5

TABLE XLVI

SERIES X

EXTRACTION VERSUS DROP FORMATION TIME (END EFFECT)
CARBON TETRACHLORIDE-ACETIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter inches	K ft./hr.	C_t lb.moles/cu.ft.	C_r lb.moles/cu.ft.	Ln. ΔC.	C_w lb.moles/cu.ft.
X-1	7.5 mm O.D. 2 mm I.D.	0.141	0.97	0.01037	0.00885	0.00959	0.000263
X-2	7.5 mm O.D. 2 mm I.D.	0.141	1.2	0.01037	0.00854	0.00949	0.00030
X-3	7.5 mm O.D. 2 mm I.D.	0.141	0.686	0.01037	0.00932	0.0098	0.000226
X-4	7.5 mm O.D. 2 mm I.D.	0.148	0.846	0.01037	0.00915	0.00975	0.000232
X-5	7.5 mm O.D. 2 mm I.D.	0.144	0.93	0.01048	0.009	0.00968	0.000226

% Extraction	H ft.	V ft./sec.	L cu.ft./hr.	A sq. ft.	T_c °C	T_d °C	θ_t seconds	θ_f seconds
14.65	0.427	0.371	0.00463	0.000755	17.5	21	1.15	0.66
17.65	0.427	0.388	0.00212	0.000341	17	21.5	1.1	1.4
10.1	0.427	0.388	0.01845	0.00288	18	21	1.1	0.166
11.75	0.427	0.388	0.00744	0.0011	17	20	1.1	0.48
13.2	0.427	0.388	0.0102	0.00156	19	23	1.1	0.32

TABLE XLVII

SERIES YA

EXTRACTION VERSUS DROP DIAMETER (END EFFECT)
CARBON TETRACHLORIDE-PROPIONIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter inches	K ft./hr.	C_t lb.moles/cu.ft.	C_r lb.moles/cu.ft.	Ln. ΔC.	C_W lb.moles/cu.ft.
YA-1	7.5 mm O.D. 2 mm I.D.	0.141	0.743	0.0079	0.0071	0.00752	0.000192
YA-2	6 mm O.D. 4.5 mm I.D.	0.1885	1.0	0.0079	0.00702	0.00751	0.000182
YA-3	Tube D. D. small	0.1	0.707	0.00785	0.00663	0.00718	0.000273

% Extraction	H ft.	V ft./sec.	L cu.ft./hr.	A sq. ft.	T_C °C	T_d °C	θ_t seconds	θ_f seconds
10.1%	0.416	0.416	0.00687	0.000985	14	19.5	1.0	0.44
11.1%	0.416	0.378	0.0153	0.00179	14	20	1.1	0.476
15.55%	0.416	0.347	0.00218	0.000524	14	20	1.2	0.50

TABLE XLVIII

SERIES YB

EXTRACTION VERSUS DROP DIAMETER
CARBON TETRACHLORIDE (DISPERSED)
WATER-ACETIC ACID (CONTINUOUS

Exp.	Nozzle	Drop Diameter inches	K ft./hr.	C_t lb.moles/cu.ft.	C_r lb.moles/cu.ft.	Ln. Δ C.	C_W lb.moles/cu.ft.
YB-1**	Tube D.D. small	0.0957		0.0	0.00028		0.0107
YB-2	6 mm O.D. 4.5 mm I.D.	0.191	7.3	0.0	0.000093	0.0000622	0.0108
YB-3	7.5 mm O.D. 2 mm I.D.	0.154	5.05	0.0	0.0000867	0.0000677	0.0108
YB-4	7.5 mm O.D. 2 mm I.D.	0.152	14.6	0.0	0.0000743	0.0000773	0.0108

% Extraction*	H ft.	V ft./sec.	L cu.ft./hr.	A sq.ft.	T_c °C	T_d °C	θ_t seconds	θ_f seconds
100	0.855	0.45	0.001995	0.000792	15	24	1.9	0.48
77.5	0.843	0.432	0.0152	0.00311	15	24	1.95	0.50
72.2	0.855	0.438	0.00765	0.00194	15	24	1.95	0.52
61.8	0.219	0.438	0.00752	0.000495	15	24	1.95	0.508

NOTE:

** Reached equilibrium at undetermined column height.

* Based on equilibrium.

TABLE XLIX

SERIES Z

EXTRACTION VERSUS DROP DIAMETER
CARBON TETRACHLORIDE-PROPIONIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	Nozzle	Drop Diameter inches	K ft./hr.	C_t lb.moles/cu.ft.	C_r lb.moles/cu.ft.	Ln.ΔC.	C_T lb.moles/cu.ft.	C_B lb.moles/cu.ft.
Z-1*	7.5 mm O.D. 2 mm I.D.	0.169	0.495	0.00781	0.00475	0.00615	0.000192	0.000131
Z-2	7.5 mm O.D. 2 mm I.D.	0.148	0.372	0.00781	0.00511	0.00635	0.000121	0.000081
Z-3	7 mm O.D. 1 mm I.D.	0.114	0.209	0.00784	0.00573	0.0067	0.000081**	0.000081
Z-4	Cap. D. D. Small	0.0962	0.236	0.00784	0.00502	0.00635	0.000101	0.000091
Z-5	Tube D. D. Medium	0.103	0.246	0.00784	0.0051	0.00633	0.000101	0.000091
Z-6	6 mm O.D. 1.5 mm I.D.	0.1305	0.314	0.00788	0.00526	0.00647	0.000101	0.000071
Z-7	Tube D. D. Large	0.150	0.33	0.00785	0.00546	0.00657	0.000121	0.000071
Z-8	6 mm O. D. 4.5 mm I.D.	0.191	0.53	0.00785	0.00492	0.00627	0.000152	0.000091
Z-9	7 mm O. D. 5 mm I. D. (P.)	0.190	0.514	0.00785	0.00494	0.00627	0.000152	0.000081
Z-10	8 mm O.D. 5.5. mm I.D.	0.205	0.638	0.00792	0.00462	0.00611	0.000222	0.000121
ZA-1***	6 mm O.D. 4.5 mm I.D. (P.)	0.196	0.403	0.0104	0.00733	0.00875	0.000175	0.000137
ZA-2***	6.5 mm O.D. 1.5 mm I.D.	0.1355	0.303	0.0104	0.0073	0.00874	0.00015	0.000125
ZA-3***	Tube D.D. Medium	0.1043	0.271	0.0104	0.00668	0.00837	0.000125	0.000125

TABLE XLIX (CONT'D)
SERIES Z

EXTRACTION VERSUS DROP DIAMETER
CARBON TETRACHLORIDE-PROPIONIC ACID (DISPERSED)
WATER (CONTINUOUS)

Exp.	% Extraction	H ft.	V ft./sec.	L cu.ft./hr.	A sq. ft.	T_c °C	T_d °C	θ_t seconds	θ_f seconds
Z-1*	39.2	4.77	0.569	0.01135	0.0114	16	23.5	8.4	0.46
Z-2	34.6	4.77	0.569	0.00731	0.00837	17.5	26	8.4	0.48
Z-3	27	4.77	0.555	0.00367	0.00554	15	23	8.6	0.44
Z-4	36	4.77	0.53	0.002155	0.00406	16	24	9.0	0.448
Z-5	35	4.77	0.53	0.00262	0.0046	16.5	25	9.0	0.452
Z-6	33.3	4.77	0.568	0.00539	0.00695	17	26	8.4	0.449
Z-7	30.5	4.77	0.581	0.00768	0.00848	14	22	8.2	0.476
Z-8	37.3	4.77	0.568	0.0153	0.0135	14	21	8.4	0.496
Z-9	37.1	4.77	00.561	0.0155	0.014	14	22	8.5	0.48
Z-10	41.7	4.77	0.50	0.01935	0.0164	14	24	8.7	0.488
ZA-1***	29.5	4.77	0.561	0.0158	0.01375	14	24	8.5	0.52
ZA-2***	29.8	4.77	0.605	0.0058	0.0068	14	24	7.9	0.466
ZA-3***	35.8	4.77	0.561	0.00256	0.00421	14	25	8.5	0.48

* Nozzle showed some drop adherence.
** Top liquid purged during run.
*** ZA Series - HAC replaced propionic acid in these runs.

NOMENCLATURE

Term		Meaning	Units
a	-	major axis of an ellipsoid	length
A	-	interfacial or surface area	$(\text{length})^2$
b	-	minor axis of an ellipsoid	length
C	-	concentration	$\text{Mass}/(\text{length})^2$
c	-	drag coefficient	none
D	-	drop rate	drops/time
d	-	drop diameter	length
D_i	-	molecular diffusivity	$(\text{length})^2/\text{time}$
D_e	-	eddy diffusivity	$(\text{length})^2/\text{time}$
f	-	fraction of terminal velocity	none
F	-	force	force
g	-	gravitational constant	$\text{length}/(\text{time})^2$
H	-	column height	length
I.T.	-	interfacial tension	force/length
K_o	-	overall mass transfer coefficient	length/time
K	-	distribution ratio (C_w/C_s)	none
K'	-	inverse distribution ratio	none
k	-	film mass transfer coefficient	length/time
L	-	flow rate	$(\text{length})^3/\text{time}$
n	-	association	molecules
M	-	molecular weight	none
P	-	pressure	$\text{mass}/(\text{length})(\text{time})^2$

Term		Meaning	Units
Q	-	mass	mass
r	-	drop radius	length
S	-	single molecules	none
D_o	-	double molecules	none
T	-	temperature	temperature
V	-	velocity	length/time
V'	-	volume	$(\text{length})^3$
"NuE"	-	mass transfer number $= \frac{Kd}{D_i}$	none
Re	-	Reynolds number $= \frac{d\rho V}{\mu}$	none
We	-	Weber's capillary group $= \frac{d\rho V^2}{I.T.}$	none
Sc	-	Schmidt number $= \frac{\nu}{D_i}$	none
α	-	degree of ionization	none
θ	-	time	time
μ	-	absolute viscosity	mass/(length)(time)
ν	-	kinematic viscosity $= \mu/\rho$	$(\text{length})^2$/time
ρ	-	density	$\text{mass}/(\text{length})^3$

Subscript

w	-	at the wall (wetted wall columns), otherwise water
c	-	in the core (wetted wall columns), otherwise it designates the continuous phase
s	-	solvent or sphere

Subscript

o	-	outer or overall
d	-	dispersed phase
b	-	solvent
a	-	solute
f	-	feed when referred to C and formation when referred to Θ
r	-	product
T	-	top sample
B	-	bottom sample
t	-	contact
i	-	interface
e	-	equilibrium and also ellipsoid
D	-	drag
B_o	-	bouyancy
G	-	gravity
K	-	critical or maximum

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