COAGULATION OF AN
OIL-WATER EMULSION WASTE

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SCOPE AND CONTENTS:

A small scale continuous unit, jar test unit and zeta meter were used to study the effects of chemical treatment on a stable emulsion waste from a steel rolling mill.

The effect of coagulant dosage and pH of coagulation on colloid mobility and on oil removed was studies. All other system variables were kept constant.

Of the 15 combinations of chemicals tried, an effective coagulant for breaking a fresh stable emulsion and a corresponding pH range for effective separation was obtained.

Colloid mobility measurements indicated the feasibility of using the zeta potential technique in the waste field to determine and control coagulant dosage. However the zeta meter in its present form has limited use with emulsions.

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NOMENCLATURE

A - area of the submerged paddle in a plane perpendicular to the direction of motion (ft^2)

cm. - centimeter

C_d - drag coefficient for the paddles

CSTR - continuous stirred tank reactor

d - rotor diameter (ft.)

d_1 - average diameter of the N_1 particles

d_2 - average diameter of the N_2 particles

D - impeller diameter - (ft.)

D_t - dielectric constant of the suspending liquid

E - potential difference (volts)

EM - electrophoretic mobility (u/sec per E/cm.)

F_d - the drag force (lbs.)

G - velocity gradient at a point (sec^{-1})

G_m - root mean square velocity gradient (sec^{-1})

G_mT - product of velocity gradient and time (dimensionless)

I - current (micro-amps)

K - the ratio of the rotating velocity of fluid to the rotating velocity of the blades

K_1 - electrophoresis cell constant

ml - milliliter

mg/l - milligrams per liter

n - shaft speed (revolutions per second)

N - number of contacts between particles per unit volume and unit time

N_1 - number of suspended particles naturally present in a unit volume of liquid

N_2 - number of floc particles in the fluid

ppm - parts per million

Q - liquid flow rate (ft^3/sec)

r_b - the distance of the center of the rotating paddle from the center of the shaft (ft.)

rpm - revolutions per minute

rps - revolutions per second

Re - Reynolds Number (dimensionless)

Re_I - Impeller Reynolds Number (dimensionless)

S_g - geometric standard deviation

SC - specific conductance (micromhos/cm.)

t - detention time (minutes)

T - detention time (seconds)

Tr - torque (dyne-cm.)

u - microns

v - velocity (fps)

v_{bw} - velocity of the paddle with respect to the water

V - volume (ft^3)

V_t - viscosity of the suspending liquid in poises

W - work of shear per unit of volume per unit of time (ft - lbs/sec - ft^3)

W_1 - energy input by the paddle in the jar tester (dyne-cm/cm^3-sec)

$\bar{x}$ - arithmetic mean

$\bar{x}_g$ - geometric mean

ZP - Zeta Potential (electrostatic volts)

ρ - mass density of fluid - (lbs/ft^3)

μ - absolute viscosity of fluid - (lb/ft-sec)

1. UNDERSTANDING THE OILY WASTE PROBLEM

1.1 Source and Nature of the Waste

The waste used in this experimental work is a typical steel plant waste originating from the cold reversing rolling mill. It is a complex mixture of water, oil, an emulsifier and mill scale, and the resultant mixture is a white color.

The oil - water mixture is fed to the rollers to keep the oxygen from the metal and to lubricate the steel rolling process. When the oil comes in contact with the water it is dispersed into individual small droplets. With the aid of the emulsifier contained in the oil, the result is a stable emulsion of oil in water. The oil droplets in this state will not coalesce and separate from the water phase even after sitting for a prolonged period of time.

The stability of the emulsion can be considered to be due to a number of factors. The small size of the oil droplets, about 5 microns [30], coupled with the large dilution factor of water to oil tend to retard the coalescence of the oil droplets. Collisions between individual drops due to Brownian motion is therefore minimized. Probably the most significant factors are those due to the presence of a surfactant and ionic surface charge for dilute and concentrated emulsions. The surfactant is preferentially attracted to interfaces and therefore the reactive groups of the surfactant are adsorbed to the surface and form a protective film around the oil drop. These reactive groups then attract solution ions of opposite charge which establishes a repulsive force between individual drops. This repulsion is called double layer repulsion [10, 11, 27, 28, 36].

These factors mean that an emulsion type waste can be a problem which requires special attention if successful treatment of the waste is to be effected. Present oil waste treatment techniques are based on the American Petroleum Institute traditional design [31]. This is tailored for the easy-to-separate or the unstable emulsions in which the oil is removed by gravity separation. For difficult to treat stable emulsion wastes such as we have here pretreatment prior to gravity separation is necessary [24, 30, 43]. Some specialized pretreatment methods are addition of chemical coagulants, pH adjustment, application of a strong electrical field and solvent extraction. Now the ultimate aim is to design the treatment works economically. For an emulsion waste there is a necessity to investigate these specialized pretreatment methods to establish design criteria.

1.2 Research Approach

Because specialized pretreatment for the stable emulsion is required, chemical treatment was selected for investigation. It was therefore necessary to take a limited look at optimizing conditions necessary for satisfactory treatment. The experimental approach therefore used a batch unit to obtain a feel for a successful pretreatment a colloid mobility measurement technique as a measure of the double layer repulsion and a small scale continuous unit to identify optimum performance. The former two are suggested techniques for developing continuous flow designs and therefore an evaluation and usefulness of these techniques is required.

The present work is a screening analysis since the optimum conditions depend on achieving optimum overall system variables including mixing, coagulation, separation and optimum chemical dosage. For this waste the greatest unknown is chemical dosage. The scope of the present work is limited to chemical dosage and pH requirements. Inherent in all results then is the assumption that the rapid mix, coagulator and separator are performing at optimum. Specifically preliminary tests indicate that the chemical concentration vary from 50 to 250 mg/l and the pH from 7.0 to 11.8. Therefore for this project the variables considered are chemical concentration and pH with the experimental limits for investigation as stipulated above.

2. LITERATURE REVIEW

2.1 Introduction

Because the approach to the treatment of a stable emulsion is different from the traditional, the literature review deals with the state of the art of chemical coagulation, the use of the batch or jar test and the zeta meter as laboratory techniques to evaluate and optimize treatment methods, and the necessity and relative limitations of these laboratory techniques.

2.2 A Colloidal System

For small particles, surface forces may predominate the body force behavior. Normally this occurs for particles less than one micron in diameter, but this size depends on the surface forces that are present.

The tendency of a surface or particle in a colloidal system to become wetted is referred to as either lyophilic (liquid loving) or lyophobic (liquid hating). Emulsions, characterized as lyophobic are stabilized by a combination of the electrical double layer interaction[27, 28, 33] and the surface active agent which forms a tightly bound protective film around the oil drop.

2.3 Electrical Double Layer Interaction

Colloid particles possess electrical properties that strongly influence their behaviour. These electrical properties are the result of an ionic double layer formation around each colloid which results in the repulsive force between colloid particles.

Charges located on the surface of a colloid, called primary charges, result from one or both of the following[32]:-

(a) dissociation of reactive groups of molecules comprising the particles structure.

(b) preferential adsorption of ions from the bulk solution.

With the emulsion, the primary charge is due to the reactive groups of the surface active agents which can be anionic, cationic or non - ionic depending upron the surfactant used and the ions present in solution. The reactive groups are preferentially adsorbed to the surface of the oil droplet. The primary charge on the oil droplet then attracts solution ions of opposite charge. This forms a compact and stationary layer of counterions, located adjacent to the oil drop known as the fixed or Stern layer. The fixed layer is surrounded by an outer layer of counterions called the Gouy layer which is diffuse and mobile. The concentration of counterions in the diffuse layer varies from the relatively high concentration existing at the boundary between the fixed and diffuse layers to that in the bulk of the solution.

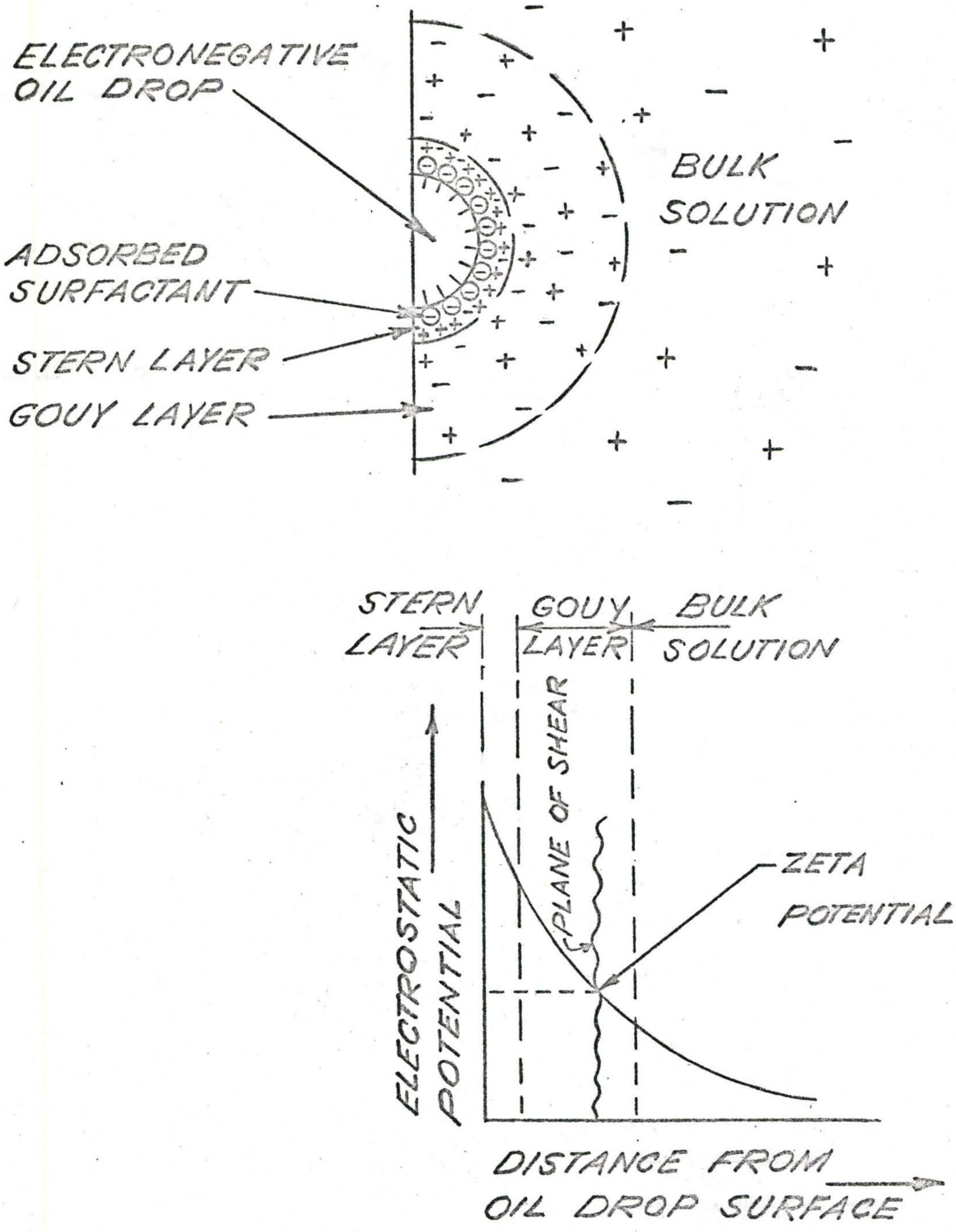
Concentration differences between the cations and anions results in the formation of an electrostatic field surrounding the oil drop. Therefore a difference in potential exists between the surface of the colloid and the bulk solution. The potential of importance is the potential existing at the plane of shear for a colloid and by definition is referred to as Zeta Potential[15, 27, 32, 36]. The plane of shear forms a boundary between the portion of the solution which surrounds the oil droplet and moves integrally with it and the portion which remains a part of the bulk solution and moves independently of the droplet.

A diagram of ionic atmosphere surrounding the oil drop and the corresponding potential gradient through the electrostatic field is shown in Figure 1.

2.4 Measurement of The Electrical Double Layer

When an electrical D-C field is imposed on a liquid containing charged colloidal particles, the particles migrate, the electronegative particles to the positive electrode and vice versa.

The mobility increases with the electrode potential.



ELECTROSTATIC FIELD AROUND THE OIL DROP

FIGURE 1

Friction between the particle and the bulk solution slows down the motion of the colloid toward the electrode. The greater the extent of the diffuse layer the lower the frictional resistance. Therefore the velocity of the particle in a given electric field is proportional to that field (volts per cm.) and to the zeta potential of the colloid. Because the motion of charged colloids in an electric field is dependent mainly on zeta potential and not on particle size or shape, then the measurement of colloid mobility is a rapid and accurate method of determining the magnitude of zeta potential [10, 38].

A few techniques which have been developed to measure colloid mobility in an electric field are micro-electrophoresis, the moving boundary method and zone electrophoresis [27, 28].

Micro-electrophoresis is the commonly used technique and is the one employed in this research project [26,27,29,34,36,39].

Measurement of colloid electrophoretic mobility is a quantitative evaluation of the amount of counterions required for neutralization of the colloid surface charge. This technique can be used very effectively to predict the nature of the charge retained by the colloids in a waste and would then predict the use of either anionic or cationic coagulants. Further research is required to evaluate the extent of the usefulness of this concept in waste treatment. More details of this method are given by Riddick [25, 26].

2.5 Coagulation

The coagulation process involves the addition and thorough mixing of chemicals with the waste to be treated. The function is to effect destabilization, aggregation and binding together of the colloids by the addition of counter-ions to the system [7, 14, 15, 17, 40, 41].

Coagulation can be considered to take place in two separate phases: perikinetic and orthokinetic coagulation [14]. In the initial phase, perikinetic coagulation, the chemicals and the waste are rapidly and thoroughly mixed together and this is considered to be completed in a few seconds [4, 14]. This results in the hydrolysis of the coagulant and neutralization of the colloid surface charge which promotes the formation of microfloc [14]. Once the charge has been neutralized, then the particles need to be brought together. In a stagnant fluid, Brownian motion brings the particles close together and promotes aggregation. When the micro floc, in the initial phase have reached a size at which Brownian motion has little effect then further aggregation of these micro floc is accomplished by orthokinetic coagulation [14]. Mutual collision of the microfloc is effected by velocity gradients which are created by gentle agitation of the fluid [4, 5].

2.6 Flocculation

Flocculation is the process by which the colloid aggregations generated by orthokinetic coagulation are bridged together into a large agglomerated mass of floc. Polymer type flocculants, referred to as coagulant aids, are used for this purpose, and this results in a strengthening of the floc and the creation of a rapid settling mass [35]. Flocculation assists in removing the pin-point, floc-colloid, aggregates which otherwise may not be separated.

2.7 Mechanism of Orthokinetic.Coagulation

The theory of orthokinetic coagulation was developed by Smoluchowski in 1918 and introduced into the literature by Camp [19, 41] who expanded it for use in practical design.

The basic form of this equation characterizing coagulation is given by Equation (1) [5,6,8]

$$N = \frac{1}{6} \; G \, N_1 \, N_2 \, (d_1 + d_2)^3 \quad - - - - - - -(1)$$

From this equation, the number of contacts or collisions is directly porportional to the velocity gradient, G. The magnitude of G is a function of energy input per unit volume and of the absolute viscosity of the fluid. This relationship is expressed by Equation (2) [4].

$$G = \sqrt{\frac{W}{\mu}} \qquad - - - - \qquad (2)$$

Combining equations (1) and (2) yields an expression for N in terms of energy input and viscosity and is given by Equation (3) [6].

$$N = \frac{1}{6}\sqrt{\frac{W}{\mu}}\; N_1 N_2 (d_1 + d_2)^3 \qquad - - \qquad (3)$$

From Equation (3), N, which is equal to the rate of coagulation, increases with number and size of particles and the energy input and decreases with the viscosity of the continuous medium. The floc particle will grow in size until the shearing action of the fluid exceeds the binding energy of the floc and thus overcomes any further tendency for aggregation [4, 41].

The preceding can be related to the design of a rotating paddle by Equation (4) [4, 5]:

$$W = \frac{F_d\, v_{bw}}{V} \qquad - - \qquad (4)$$

This can be expanded to include all the paddles on a rotor as shown in design given in Appendix A. Expanding the preceding equation for F_d and including all the paddles on a rotor yields the expression for rotor design given by Equation (5) [4,5].

$$W = \frac{239\ C_d\ (1-K)^3\ n^3\ \Sigma\ A(r_b)^3}{V} \quad - - - - \quad (5)$$

This equation gives the energy required for orthokinetic coagulation.

2.8 Jar Test Evaluation

The jar test, and its modified versions, has been the laboratory technique used to control full scale coagulation processes since the 1800's [12,13]. The test equipment as now employed is shown in Figure A-2 of the Appendix, and is referred to throughout the text as the batch or jar test. Usually round jars are used instead of the square jars. However the square jars have been found more effective in reducing the amount of bulk rotational flow within the jar.

Hannah [37] states that the jar test simulates the full-scale coagulation process, but there is very little actual similarity between the two. Geometric similarity, mixing characteristics, scale down of floc, hydraulic similarity can not be duplicated [38]. This is also verified by the information given in the literature for plant operations in which the jar test was used as control. For some plants the jar test predicted plant performance, some plants required more coagulant, others required less than the predicted [8,37,38]. Therefore one cannot explicitly expect to determine for a full scale process the exact optimum coagulating rpm., coagulant dosage or the coagulating time with the jar test. There is a lack of standardization in performing the test and in reporting of information on jar test - treatment plant simulation between various researchers and parties concerned. Some researchers have indicated the need for standardization of some aspects of the test [12, 37] but the feasibility of standardization at this time is hampered by the lack of agreement among different researchers on the basic theory of coagulation [12]. The jar test can therefore be used as a guide to establish a floc producing dosage [36] and to evaluate the relative treatment capabilities of different chemicals [7].

Because the information received from the jar test is qualitative, a useable technique still remains to be developed to supply quantitative information for predicting full-scale plant operations.

3. EXPERIMENTAL APPROACH

3.1 Introduction

The experimental work is based on the jar test, zeta meter and a continuous coagulation - separation unit. This section considers the variables in the work, the equipment used to collect data and the testing procedure used in the experimental work.

3.2 Variables

As mentioned in section 1 the operating variables selected for this experimental work were coagulant dosage and pH. The coagulant found to be satisfactory for treatment of the emulsion was ferrous sulphate. pH was varied by the addition of calcium hydroxide and was found to be a necessary requirement to yield a floc that settles at greater than 2 ins./min. Both chemicals were selected by preliminary experimentation on the basis of % oil removed and floc settling velocity. The ranges established for the independent variables were:-

Coagulant dosage - 50 to 250 mg/l

pH - 7 to 11.8

The remaining variables which affect the overall efficiency of the treatment unit were kept constant and these were the oil concentration of the waste sample, rapid mix rpm, detention time (rapid mix), coagulator rpm, coagulator detention time, separator overflow rate, separator detention time and temperature of operation.

The rpm. of the rapid mix chamber was established at 225 rpm. for the continuous unit and 100 rpm. for the batch unit. This double choice in rpm gave a Re_I of 10,000 in both batch and continuous chambers. This was in the turbulent region for data given by Rushton et al [3]. Detention time for rapid mixing was arbitrarily selected as 45 seconds which was within the limits suggested by Rohlich and Murphy [6]:

The coagulator rpm was arbitrarily chosen as 23 rpm in both the continuous and batch units. For the continuous unit this was approximately equal to the calculated optimum rpm. of 24.5. An attempt was made to equate the energy input obtained in the continuous unit to the jar test unit but this resulted in a calculated value of 53 rpm. for the jar test. From the preliminary tests this rpm. was found to be too high for developing proper floc and it was therefore decided to approach the problem by setting the rpm. for the jar test equal to that of the continuous unit. The detention time for orthokinetic coagulation, for both the continuous and batch units was set at 50 minutes from information in the literature [4,6].

The oil concentration of the waste was selected on the basis of a concentration which was amenable to electrophoretic measurement. Oil droplets in the waste could not be distinguished too readily when the oil concentration was in the range of 150 mg/l and greater due to the opacity of the waste. Therefore as the oil concentration of the waste was approximately 400 mg/l, it was diluted with 3 volumes of tap water to one volume of waste to yield a concentration satisfactory for measurement by the zeta meter.

The temperature of the waste was controlled at 24°C which approximated room temperature during the testing period.

Separator overflow rate was arbitrarily established at 500 US gpd/ft^2 and was considered conservative in comparison to reported results. Detention time of 21 minutes in the separator was regulated by the space available within the constant temperature bath.

3.3 Experiment Design

The experimental work was planned on the basis of an octagonal, rotatable, statistical design with center points [20,21]. The design involved a two variable, five level system with a minimum total number of twelve runs which included four runs at the center point (assumed optimum).

For both batch and continuous units the independent variables, coagulant dosage and pH, were varied to determine the effect on the dependent variables, colloid mobility and oil removal.

3.4 Equipment and Procedure

The test equipment can be separated into two units:-

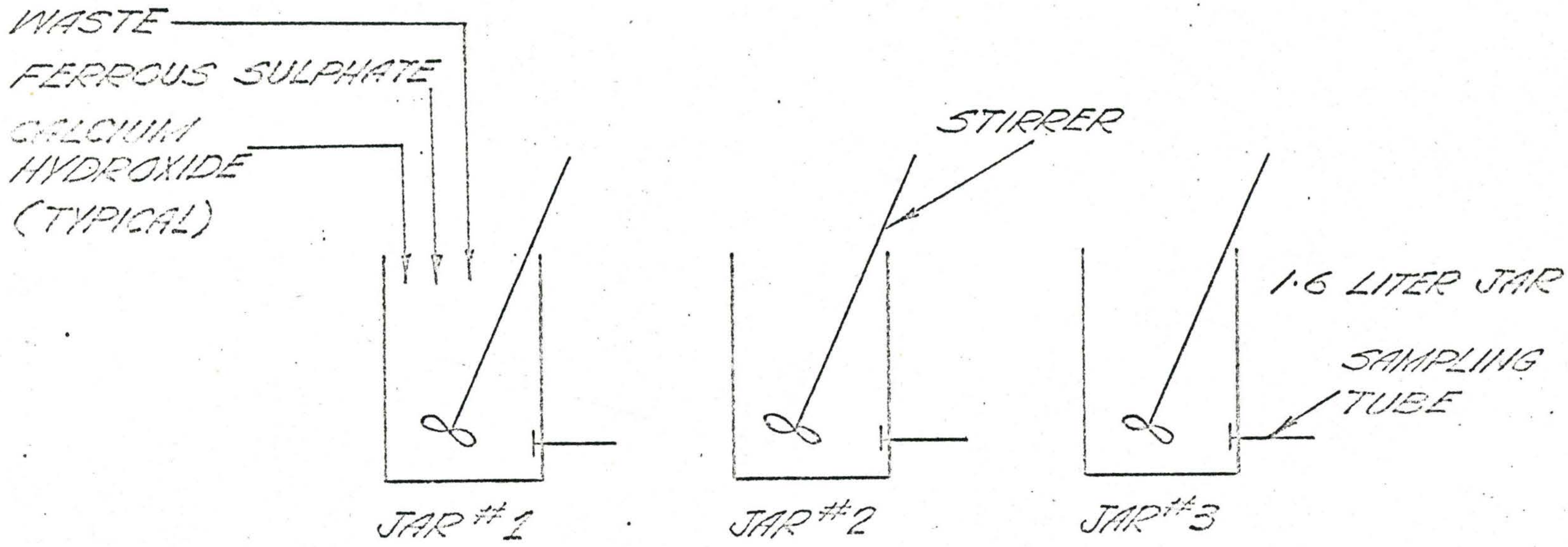
(a) batch

(b) continuous

These units are shown schematically in Figures 2 and 3. Details are given in appendix A.

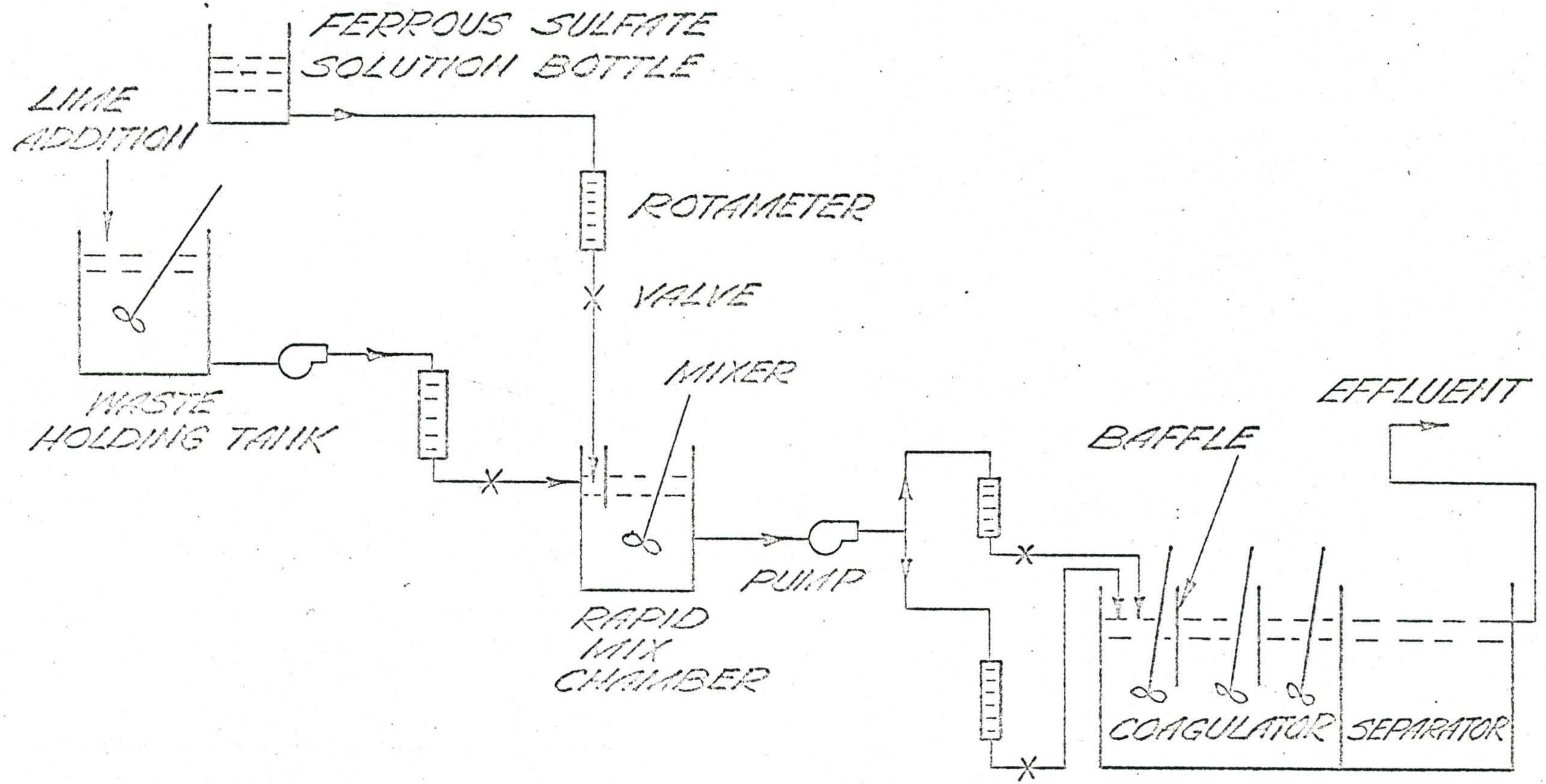
Batch Unit:

Batch experimentation was carried out in three square 1.6 liter jars with a variable speed multiple stirrer. Specified quantities of waste and coagulant were added to the jars and then the pH of the resultant mixture carefully adjusted to the required value by the addition of calcium hydroxide. This mixture was rapidly mixed and the pH of the waste - chemical mixture checked near the end of the rapid mix cycle. Adjustments to pH were made when required. Rapid mixing was followed by a period of gentle mixing to promote orthokinetic coagulation. After a specified length of time



BATCH UNIT SCHEMATIC

FIGURE 2



CONTINUOUS UNIT SCHEMATIC
FIGURE 3

the stirrer was shut off and the coagulated mass of oil and floc allowed to settle under quiescent conditions. Samples of the clarified effluent were taken through the sample tubes located approximately one and half inches above the bottom of the jar.

Continuous Unit:

For the continuous unit the coagulant concentrations were identical to those of the batch runs. The calcium hydroxide quantity required in the holding tank for pH adjustment was based on the batch test requirements.

The oily waste and calcium hydroxide mixture was pumped from the holding tank through a rotameter to the rapid mix chamber. At the same time, ferrous sulphate was fed through a rotameter to the rapid mix chamber by gravity to give the required concentration of coagulant. Here the waste and chemical were rapidly mixed and then pumped to the coagulator through two separate metered lines to obtain identical flows in each of the two basins. Orthokinetic coagulation was promoted by gentle agitation of the rotors. From here the coagulated waste passed through a double vertically slotted baffle to the separator where the floc and entrapped oil settled out and the clarified effluent discharged through an outlet at the top of the separator.

Samples for analysis were collected when the effluent showed no time trend in pH.

Analyses:

Analyses were conducted on the samples collected from the batch and continuous units for:-

(a) oil removal - separatory funnel method

(b) colloid mobility - zeta meter employing the micro electrophoresis technique

(c) pH and alkalinity - pH meter (potentiometric method)

4. RESULTS AND DISCUSSION

4.1 Introduction

This section considers the results of preliminary studies and the experimental runs. Since the number of completed experimental runs were limited the explanation of results are primarily qualitative.

4.2 Feed Consistency

A consistent waste, denoted by a constant oil concentration, was difficult to obtain from the steel plant on a day to day basis because the concentration of oil in the waste was a function of the width of steel sheet being rolled and the overall rolling mill operation. Hence a sample of sufficient size for the entire experimentation was collected and stored at the University.

However, it was difficult to maintain a consistent sample for experimentation with storage at room temperatures of 65 - 70°F. The stored sample degraded biologically with time. As used here, sample degradation means the change in oil concentration, color and pH of the sample and the liberation of gas from the sample due to the presence and activity of the microorganisms. This is summarized in Table 1.

Table 1

Waste Degradation Characteristics

Date	Oil in Diluted Sample (mg/l)	Undiluted Sample		
		pH	Color	Release of Sulphide Gas
12 Oct/68	-	7.1	white	none
16 Oct/68	87	-	white	none
18 Oct/68	82	6.95	dull white	very weak odor
19 Oct/68	70	6.65	light grey	weak odor
20 Oct/68	74	6.65	light grey	pronounced odor
21 Oct/68	65	6.5	dark grey	strong odor

To suppress biological degradation storage of the sample at approximately 4°C is recommended.

4.3 Selecting a Treatment System

Alum was effective as a coagulant in treating an aged waste. An aged waste is a waste that has undergone biological degradation. However alum was not effective in the treatment of a fresh waste, that is a waste which has not undergone biological degradation. A tabulation of the properties of an aged and fresh waste together with their relative sensitivity to alum treatment is given in Table 2.

Table 2

Waste Properties and Sensitivity to Alum Treatment

Item	Aged Waste As Used in March Tests	Fresh Waste As Used in September Tests
pH	6.3	7.3
color	dark grey	white
presence of bacteria	yes	yes
release of Sulphide Gas	strong odor	none
sensitivity to Alum treatment	moderate separation over a limited range of alum dosage	negligible separation

Because the fresh waste could not be effectively treated with alum other coagulants were evaluated by means of the jar test.

Table 3 summarizes the effectiveness of the coagulants. Of all the chemicals tried a ferrous sulphate - calcium hydroxide combination gave the best results for oil separation. This combination was effective in breaking the emulsion over a wide range of ferrous sulphate dosage. For effective separation the pH of the waste should be in the basic range.

The jar test was used to establish qualitatively the

TABLE 3

SUMMARY OF COAGULANT SELECTION

Trial *	Chemicals	Dilution Ratio (Tap water/waste)	Range of Chemical Dosage Selected (mg/l)	Comments
1	Alum	0	50 - 1500	Separation nil to insignificant
2	Alum	19:1 to 2:1	25	Clarification nil to poor
3	Alum	1:1	30 - 250	Some clarification at 50 mg/l. Floc remained in suspension, no separation.
4	Alum NaCl	1:1	Alum: 50-80 NaCl: 25	Some clarification at 50 mg/l. Floc remained in suspension, no separation.
5	Alum NaOH H_2SO_4	1:1	Alum: 50 NaOH) as required H_2SO_4) for pH variation	pH varied from 5.2 to 9.5 Clarification nil at pH 5.2 to poor at pH 7.4. Floc remained in suspension, no separation.
6	Alum	0	55 - 130	Best clarification at 55 mg/l. Some separation but still not satisfactory.

* Trials 1 and 2 - Oil concentration in the undiluted sample was approximately equal to 400 mg/l
Trials 3 to 19 - Oil concentration in the undiluted sample was >> 400 mg/l

TABLE 3 (continued)

Trial *	Chemicals	Dilution Ratio (Tap water/waste)	Range of Chemical Dosage Selected (mg/l)	Comments
7	Alum $CaCO_3$	0	Alum: 55 $CaCO_3$: 10 - 60	No improvement in separation in comparison to the previous test
8	Alum	1 ml Hardtex 999 ml water	35 - 100	Synthetic waste prepared (Hardtex is mixture of oil plus emulsifier) Very little floc formed
9	Dispersonal C-1 (polyelectrolyte)	0 to 1:1	50 - 300	No separation No floc
10	$FeCl_3$	0	50 - 300	Some clarification at 100 mg/l Separation poor
11	$FeCl_3$ $Ca(OH)_2$	0	$FeCl_3$: 0 - 130 $Ca(OH)_2$: 40	Lime added to jar with 110 mg/l of $FeCl_3$ No change in clarification
12	Alchem 550-R (polyelectrolyte)	0	50 - 200	Pin-point floc formed No appreciable clarification

* Trials 1 and 2 - Oil concentration in the undiluted sample was approximately equal to 400 mg/l
Trials 3 to 19 - Oil concentration in the undiluted sample was >> 400 mg/l

TABLE 3 (continued)

Trial *	Chemicals	Dilution Ratio (Tap water/waste)	Range of Chemical Dosage Selected (mg/l)	Comments
13	550-R Alum	1:1	550-R: 10 - 75 Alum: 30 - 130	Best clarification with 25 mg/l of 550-R and 30 mg/l alum Approximately 50% separation after 12 minutes
14	550-R Alum $Ca(OH)_2$	1:1	550-R: 10 - 35 Alum: 50 $Ca(OH)_2$: 75	Jar with 25 mg/l of 550-R gave clear water Separation good over limited range of coagulant dosage only pinpoint floc in suspension after 12 minutes settling Floc settles
15	550-R Alum $Ca(OH)_2$	1:1	550-R: 25 Alum: 50 $Ca(OH)_2$:50 - 200	Best clarification and separation with 100 mg/l of $Ca(OH)_2$ All floc settled to bottom in 2 minutes (pin-point floc negligible)
16	$FeSO_4$	2:1	25 - 200	Best separation (70%) with $FeSO_4$ at 125 mg/l Floc settles

* Trials 1 and 2 - Oil concentration in the undiluted sample was approximately equal to 400 mg/l
Trials 3 to 19 - Oil concentration in the undiluted sample was >> 400 mg/l

TABLE 3 (continued)

Trial *	Chemicals	Dilution Ratio (Tap water/waste)	Range of Chemical Dosage Selected (mg/l)	Comments
17	$FeSO_4$ $Ca(OH)_2$	2:1	$FeSO_4$: 125 $Ca(OH)_2$: 50 - 300	Best separation with $Ca(OH)_2$ at 200 mg/l approx. 95% floc settled in 2 min. Only some pinpoint floc in suspension after 5 min. Effluent-clear water
18	Alum $Ca(OH)_2$	2:1	Alum: 75 $Ca(OH)_2$: 50 - 200	Best clarification with 125 mg/l of $Ca(OH)_2$ Effluent slightly hazy Approximately 10% floc in suspension after 3 minutes.
19	$FeSO_4$ $NaHCO_3$	2:1	$FeSO_4$: 125 $NaHCO_3$: 50 - 450	Best separation with 200 mg/l of $NaHCO_3$ approximately 25% floc in suspension Floc does not settle rapidly

* Trials 1 and 2 - Oil concentration in the undiluted sample was approximately equal to 400 mg/l

Trials 3 to 19 - Oil concentration in the undiluted sample was >> 400 mg/l

optimum ferrous sulphate - calcium hydroxide dosage for setting up the experiment design.

4.4 Discussion of Experimental Results

The results of the experimental runs are summarized in Tables 4, 5, 6 and 7 and in Figures 4 to 10 inclusive for the batch and continuous units.

As there is a direct relationship between colloid travel times and colloid mobilities, the interpretations made for colloid travel times from Figures 4 to 10 therefore apply to colloid mobilities as well. The colloid mobilities for the diluted waste, batch effluent and continuous effluent were log normally distributed. An interesting point about the log normal plot for the samples of any one run and of all runs is that the lines are approximately parallel. The fact that the distributions have approximately the same standard deviations and change only in magnitude indicates that the overall mobilities of the colloids have been reduced uniformly in the treatment units.

The median, geometric mean and standard deviations for each distribution are summarized in Table 4. The relatively large values for the standard deviation of each distribution indicates the need for taking numerous measurements to obtain a realistic median mobility for the sample. The ranges in the

TABLE 4

DATA SUMMARY OF COLLOID MOBILITIES

Run No.	Sample* No.	Mean Sample Temp. °C	Distribution Curve Data For Colloid Travel Time		Median** Time (sec.)	Colloid Mobility at 24°C (u/sec per E/cm.) x 10^{-1**}		
			Mean Time $\bar{x}_g$ (sec.)	Time at $+S_g$ From $\bar{x}_g$ (sec.)		$\bar{x}_g$	S_g	Median
11	11-RW	25.0	0.87	1.05	0.9	-16.89	± 2.9	- 16.35
	11-B	25.0	2.55	3.10	2.6	- 5.76	± 1.01	- 5.65
	11-C	25.25	2.35	2.85	2.3	+ 6.24	± 1.1	+ 6.36
10	10-RW	23.25	0.73	0.845	0.7	-20.85	± 2.86	- 21.75
	10-B	26.4	1.06	1.27	1.0	-13.5	± 2.22	- 14.35
	10-C	25.75	1.35	1.71	1.3	-10.75	± 2.25	- 11.19
8	8-RW	26.5	0.72	0.86	0.7	-19.85	± 3.26	- 20.4
	8-B	26.5	2.90	3.73	2.8	- 4.94	± 1.10	- 5.11
	8-C	26.4	2.88	3.98	2.75	+ 4.95	± 1.37	+ 5.18
2	2-RW	25.6	0.82	0.96	0.8	-17.75	± 2.6	- 18.2
	2-B	25.4	2.2	2.85	2.2	- 6.65	± 1.51	- 6.65
	2-C	24.75	2.91	3.7	2.95	- 5.07	± 1.08	- 5.02
3	3-RW	23.75	0.91	1.1	0.9	-16.57	± 2.89	- 16.73
	3-B	24.25	1.8	2.24	1.75	+ 8.28	± 1.63	+ 8.53
	3-C	24.25	1.79	2.19	1.75	+11.12	± 2.05	+ 11.4
1	1-RW	24.2	0.85	0.985	0.8	-17.60	± 2.42	- 18.72
	1-B	24.2	2.65	3.5	2.7	- 5.64	± 1.37	- 5.55
	1-C	24.0	3.73	5.0	3.7	- 4.03	± 1.02	- 4.06

* Notations RW, B, and C refer to raw diluted waste, batch and continuous samples respectively.

** From Appendix H

Figure H-1 was used to convert the mobilities at any temperature to mobilities at 24°C

$\bar{x}_g$ means Geometric mean

S_g means Standard deviation

*** An $\bar{x}_g$ value of -16.89×10^{-1} means -1.689

TABLE 5

DATA SUMMARY OF EXPERIMENTAL RUNS FOR COLLOID MOBILITY AND OIL

Date	Run	$FeSO_4$ Concentration (mg/l)	pH of Coagulation		Colloid Mobility * (u/sec per E/cm x 10^{-1})			Oil (mg/l)**				
						Effluent			Batch Eff.		Continuous Eff. ***	
			Batch	Continuous	Raw Diluted Waste	Batch	Continuous	Raw Diluted Waste	Remaining	Removed	Remaining	Removed
12 Oct/68	11	150	6.95	6.9	-16.35	-5.65	+6.36	-	3	-	34	-
16 Oct/68	10	50	9.4	9.4	-21.75	-14.35	-11.19	87	16	71	12	75
18 Oct/68	8	150	9.4	9.4	-20.4	-5.11	+5.18	82	18	64	19	63
19 Oct/68	2	79	7.7	7.7	-18.2	-6.65	-5.02	70	8	62	11	59
20 Oct/68	3	221	11.1	11.1	-16.73	+8.53	+11.4	74	10	64	12	62
21 Oct/68	1	150	9.4	9.45	-18.72	-5.55	-4.06	65	8	57	8	57

* Median Mobility at 24°C, minus and positive signs denote electronegative and electropositive respectively.

** Arithmetic mean value
Oct. 12/68 Results are unreliable due to the presence of salt.

*** Eff. means effluent

TABLE 6

DATA SUMMARY OF EXPERIMENTAL RUNS FOR pH AND ALKALINITY

Date	Run	$FeSO_4$ Concentration (mg/l)	pH					Alkalinity as $CaCO_3$ (mg/l)		
			Raw Diluted Waste	Coagulator *		Effluent		Raw Diluted Waste	Effluent	
				Batch	Continuous	Batch	Continuous		Batch	Continuous
12 Oct/68	11	150	7.5	6.95	6.9	6.8	6.5	89	53	37
16 Oct/68	10	50	7.75	9.4	9.4	8.95	9.0	88	58	41
18 Oct/68	8	150	7.5	9.4	9.4	7.9	7.95	89	26	23
19 Oct/68	2	79	7.35	7.7	7.7	7.5	7.5	87	92	51
20 Oct/68	3	221	7.4	11.1	11.1	10.8	10.85	91	96	101
21 Oct/68	1	150	7.15	9.4	9.45	8.1	8.0	84	52	41

* pH taken at influent to coagulator for continuous and upon completion of rapid mix for batch

TABLE 7

QUALITATIVE DESCRIPTION OF FLOC AND EFFLUENT

Run No.	Floc Description (ins.)		Settling Rate				Comments	
	Batch	Continuous	Batch		Continuous		Batch	Continuous
			Floc Size (ins.)	Rate (ins/ min.)	Floc Size (ins.)	Rate * (ins/ min.)		
11	1/16 - pinpoint	1/8 - pinpoint	1/16	1.8	1/8	3.4	Effluent-deep lime	Effluent - deep lime Pinpoint floc in suspension
10	1/16 - pinpoint	1/16 - pinpoint	1/16	1.5	1/16 1/8	1.6 4.0	Floc - amber Effluent - slightly hazy	Effluent - slightly hazy Pinpoint in suspension gives effluent an amber color
8	1/16 - pinpoint	1/8 - pinpoint	1/16	1.5	1/16 1/8	1.65 3.3	Floc - amber Effluent - clear water	Floc - amber Effluent - light lemon Pinpoint floc in suspension
2	1/16 - pinpoint	1/8 - pinpoint	1/16	2.4	1/16 1/8	2.0 3.2	Floc - amber Effluent - lemon	Effluent - light lime

* Difficult to determine due to localized circulatory currents.

TABLE 7 (continued)

Run No.	Floc Description (ins.)		Settling Rate				Comments	
	Batch	Continuous	Batch		Continuous		Batch	Continuous
			Floc Size (ins.)	Rate (ins/ min.)	Floc Size (ins.)	Rate (ins/ min.) *		
3	1/32 - pinpoint	1/16 - pinpoint	1/32	1.4	1/16	3.0	Floc - dark green Effluent - deep lime	Floc - dark green Effluent - deep lime
1	1/16 - pinpoint	1/8 - pinpoint	1/16	2.7	1/16 1/8	4.0	Effluent - light green Floc - amber Some pinpoint floc in suspension	Effluent - deep lime Floc - green Pinpoint floc in suspension

* Difficult to determine due to localized circulatory currents.

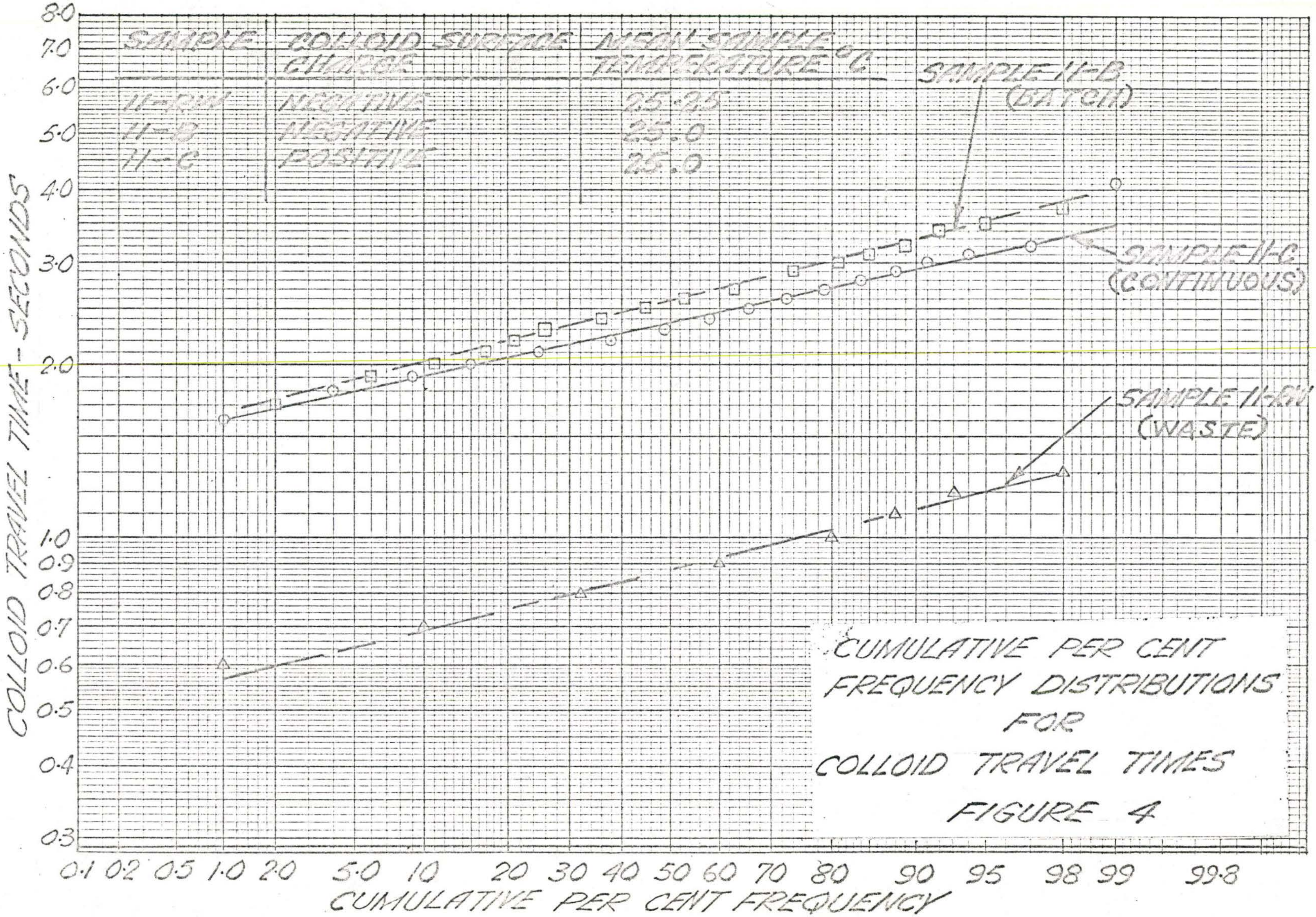
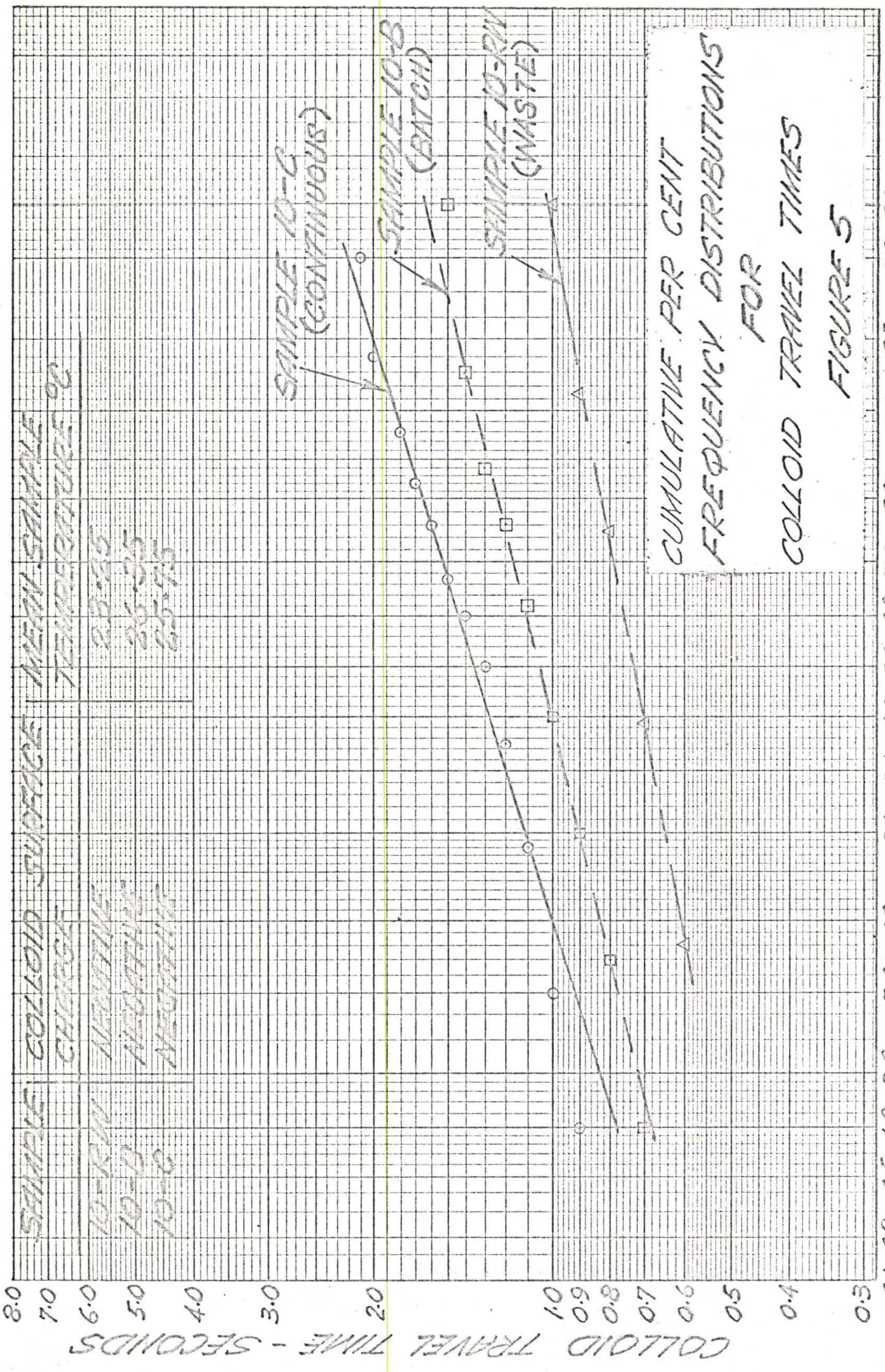
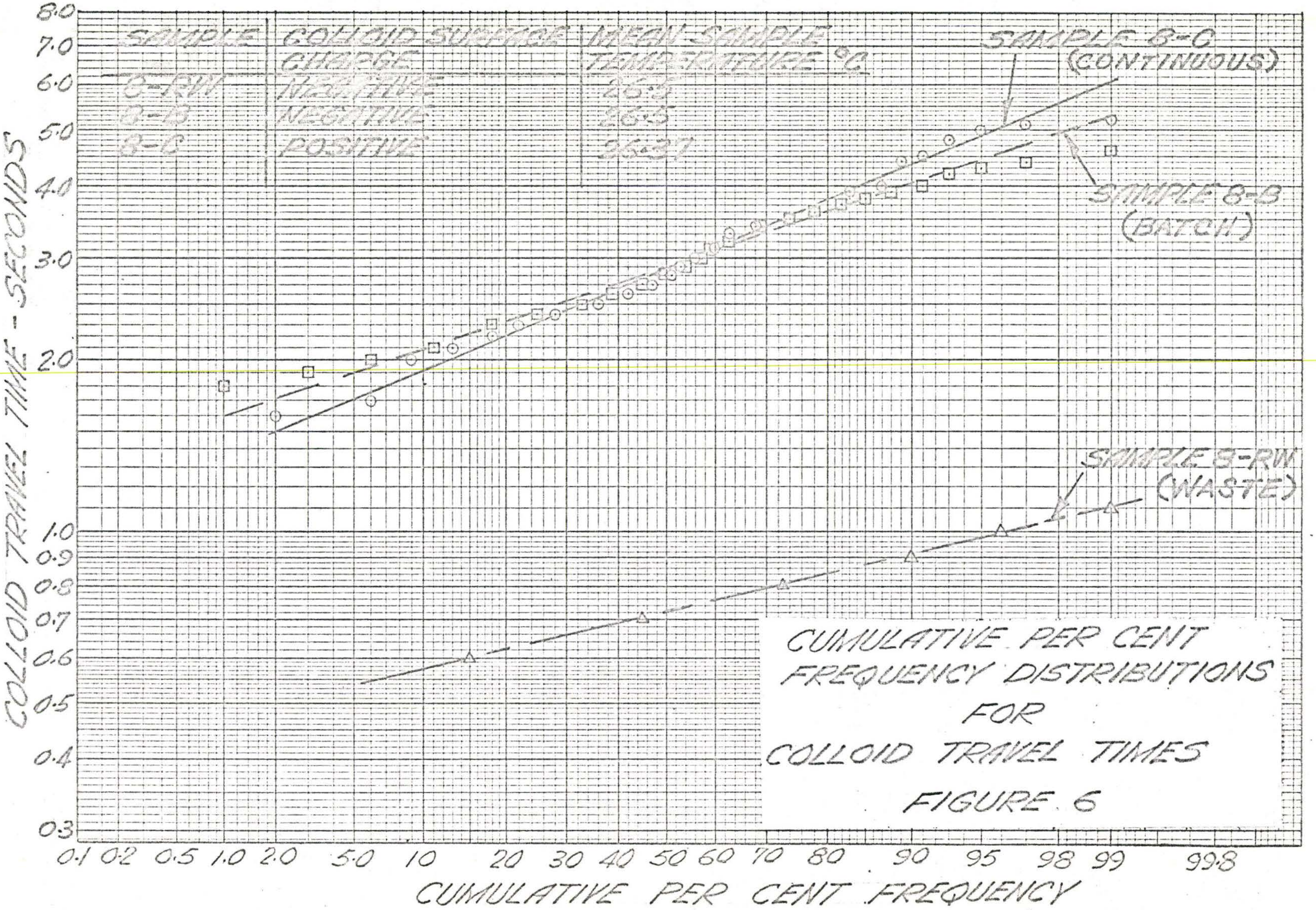

CUMULATIVE PER CENT FREQUENCY DISTRIBUTIONS FOR COLLOID TRAVEL TIMES

FIGURE 4



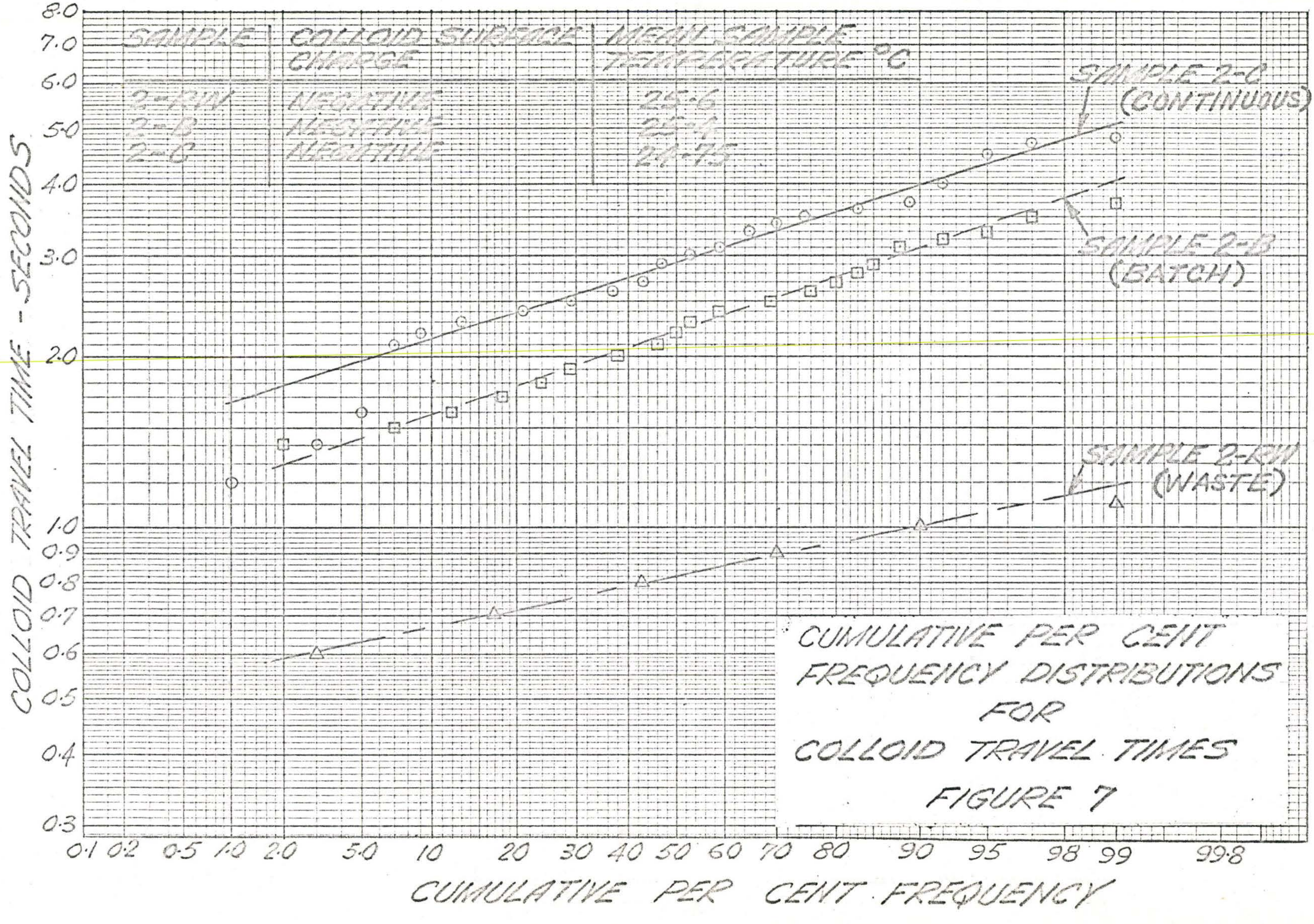
CUMULATIVE PER CENT FREQUENCY DISTRIBUTIONS FOR COLLOID TRAVEL TIMES

FIGURE 5



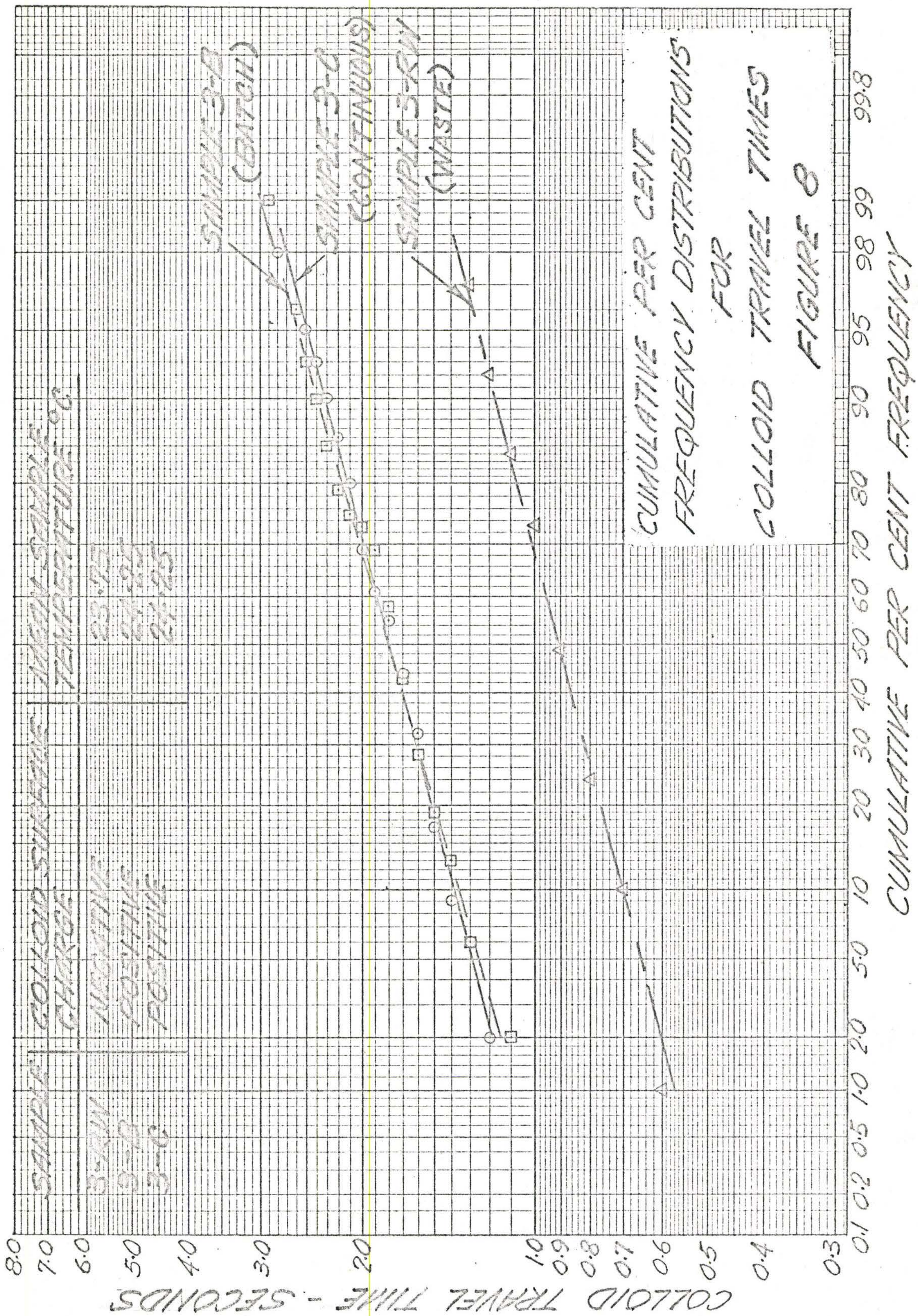
CUMULATIVE PER CENT
FREQUENCY DISTRIBUTIONS
FOR
COLLOID TRAVEL TIMES

FIGURE 6



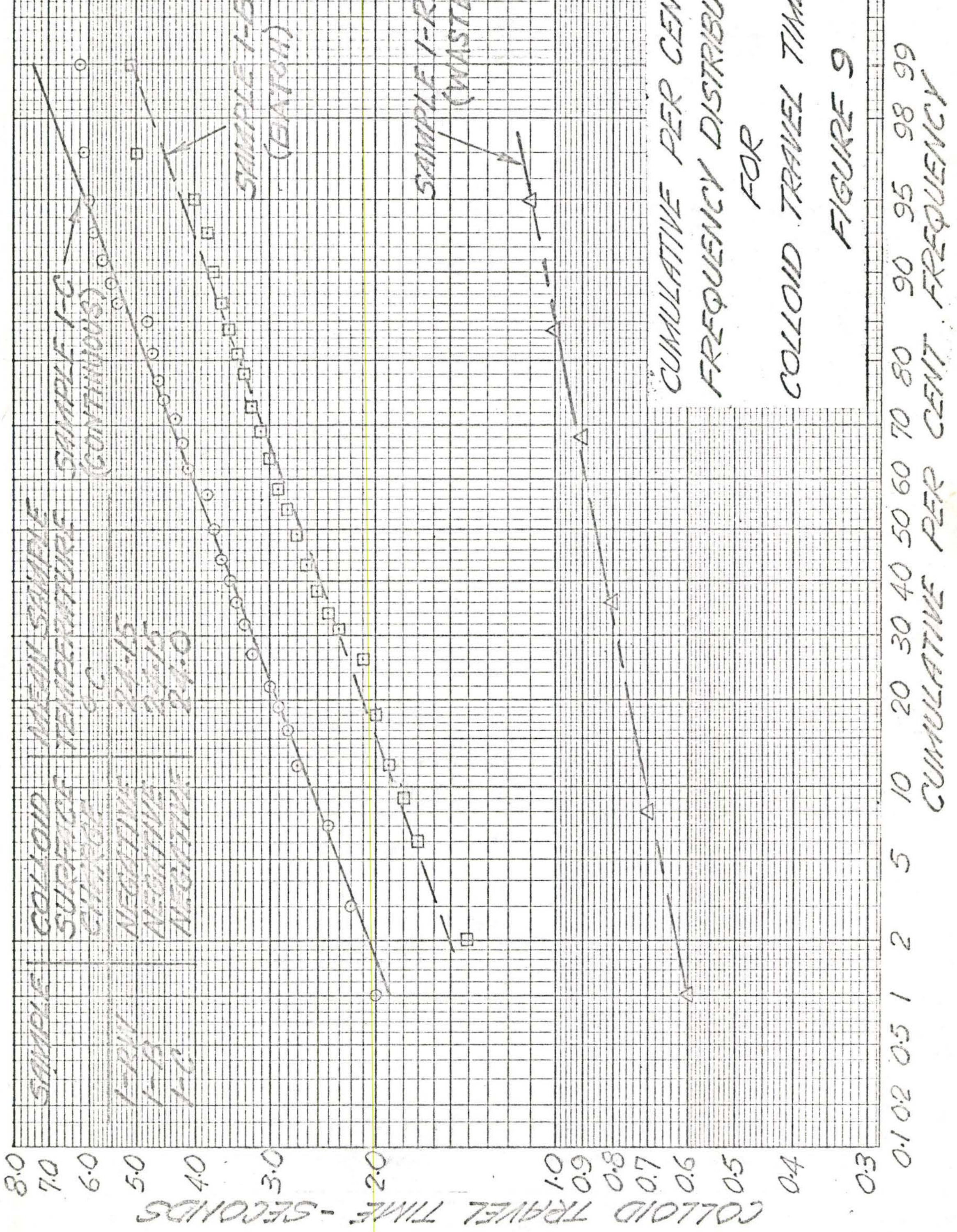
CUMULATIVE PER CENT FREQUENCY DISTRIBUTIONS FOR COLLOID TRAVEL TIMES

FIGURE 7



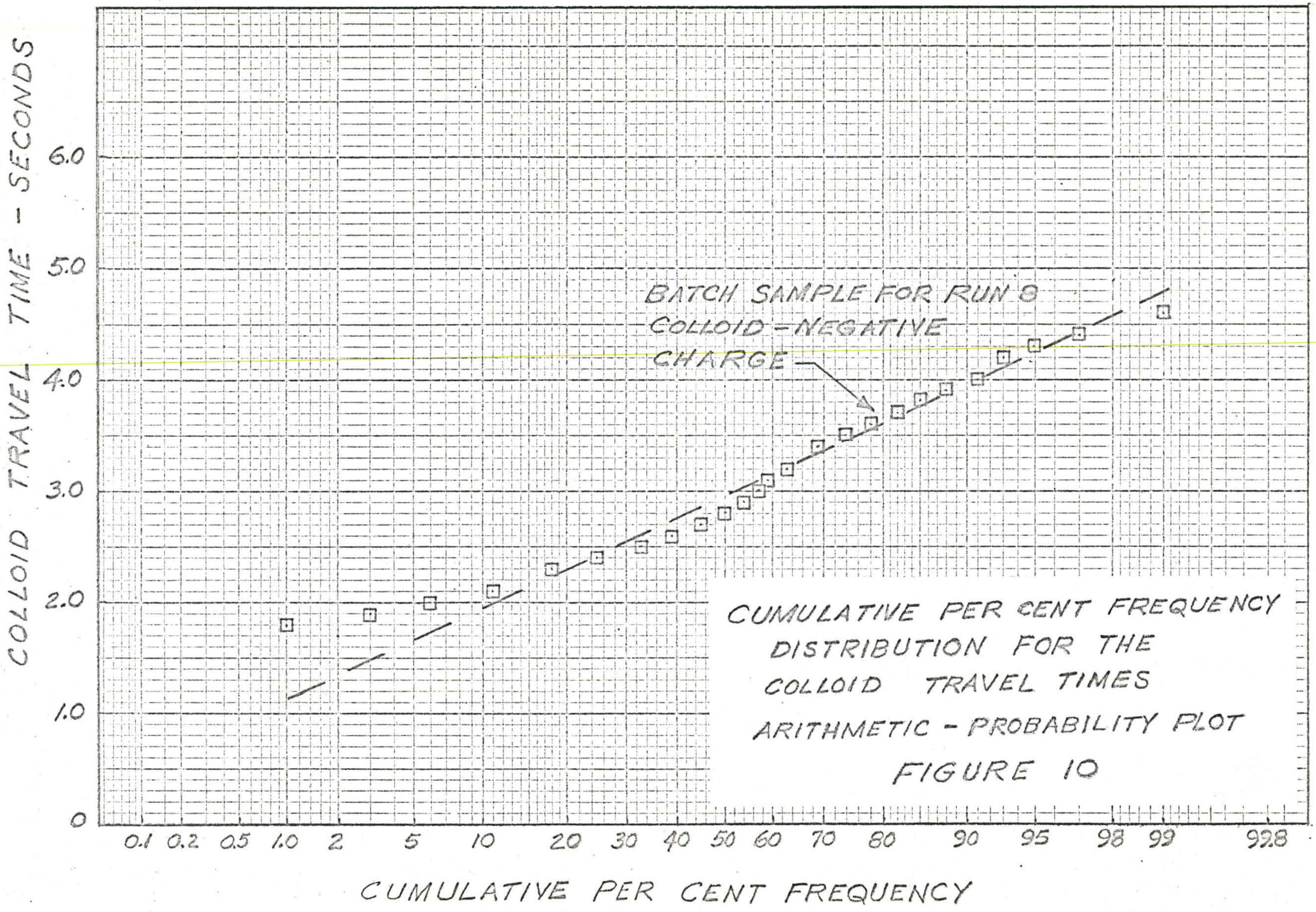
CUMULATIVE PER CENT
FREQUENCY DISTRIBUTIONS
FOR
COLLOID TRAVEL TIMES

FIGURE 8



CUMULATIVE PER CENT
FREQUENCY DISTRIBUTIONS
FOR
COLLOID TRAVEL TIMES

FIGURE 9



CUMULATIVE PER CENT FREQUENCY
DISTRIBUTION FOR THE
COLLOID TRAVEL TIMES
ARITHMETIC - PROBABILITY PLOT

FIGURE 10

geometric standard deviations for all distributions of the diluted waste, batch effluent and continuous effluent samples are 13.7% to 17.5%, 16.4% to 24.3% and 17.6% to 27.7% respectively. There is reasonably close agreement between the median and the geometric mean values for the majority of the samples which indicates the actual distributions are skewed very little and therefore approach a normal distribution but still are better represented by a geometric distribution. This is shown by the plots for Run 8-B in Figure 6 and 10.

The median mobilities for the diluted waste samples fluctuate erratically between -1.635 and -2.175 u/sec per E/cm. No logical explanation can be given for the fluctuations. However, the mobilities of the oil drops might be expected to change progressively lower as the waste deteriorated since the microorganisms were observed to preferentially attach themselves to the colloid and the results indicate the oil concentration of the waste to decrease with time. It is assumed the microorganisms preferentially utilize the oil and the adsorbed reactive groups which would reduce the surface charge and thus the colloid mobility.

The colloid mobility of the raw diluted waste was reduced consistently more in the continuous unit than in the batch. This may be attributed to the increased contact between the counterions and the colloid through more efficient mixing or the lack of precise control over the chemical and waste feed. The positive colloid mobility values indicate an excess of ferrous ions in solution.

The quantity of oil removed by the batch and continuous units is shown in Table 5. There is close agreement between the two but the oil remaining in the effluent of the continuous unit is equal to or slightly greater than the batch unit. This is considered due largely to the oil being carried out with the pinpoint floc aggregations in the effluent. In all of the runs there was considerably more pinpoint floc in the collected sample for the continuous unit than for the batch unit.

Since the waste degraded biologically with time, only relative deductions from any two consecutive days should be attempted. Examining the results for Runs 8 and 2 shows that optimum oil removal did not occur at the optimum ferrous sulphate dosage determined qualitatively by the jar test. Only an increase of 2 mg/l in oil removal was obtained at the jar test optimum of 150 mg/l of ferrous sulphate in comparison to the lower ferrous sulphate dosage of 79 mg/l. The corresponding colloid mobilities were reduced to approximately the same values in each case with the exception of the continuous unit for Run 8 which may be due to lack of precise control over the rate of chemical feed to the coagulator. This suggests that the jar test gives only an estimate for optimum coagulant dosage when effective treatment of the waste is effected over a wide range of coagulant dosage. It is therefore necessary to use a quantitative technique such as zeta potential to determine the optimum dosage.

The pH and alkalinities for the waste and effluent of the various runs are shown in Table 6. The qualitative characteristics of floc size and floc settling rates are given in Table 7. The floc in the continuous unit was generally slightly larger than in the batch. This is probably due to the reduction in velocity gradient along the length of the coagulator in the continuous unit as opposed to maintaining a constant velocity gradient in the batch test. However the overall increase in floc size along the length of the basin in the continuous unit was minimal and may partially be attributed to excessive velocity gradients in the latter stages of coagulation.

Rapid settling rates shown in Table 7 were obtained with the ferrous floc and were in the range of 1.5 to 2.7 and 1.6 to 4.0 inches per minute for the batch and continuous units respectively. The settling rates for the continuous unit were difficult to determine due to localized circulatory currents and therefore the values in Table 7 may be a little high.

4.5 Discussion of Experimental Techniques

Colloid Mobility

Little appears to have been done in utilizing the zeta potential technique in the industrial waste field. Braemar and Hoak [44] suggest that it may be useful in some applications but of little use for industrial wastes due to the variability of the waste.

The writer feels the lack of application may be due to turbidity, color, or opaqueness which would prevent proper observation of the colloid. For this research, colloid observation with the zeta meter was impossible with oil concentrations above 150 mg/l due to the opacity of the emulsion. This places a limitation on the use of this technique. A rectangular cell used by the Ontario Research Foundation is a more satisfactory design for concentrated wastes but its range of applicability should be determined.

Riddick [15,26] states that percipitates and not residual colloids should be tracked. He claims that some researchers have been disillusioned with zeta potential because the mobilities were based on residual colloids. Both residuals and pinpoint floc were tracked for the experimental runs with no noticeable difference in mobility between the two. This aspect of the technique warrants further investigation.

The nature of surface charge retained by a colloid can effectively be determined by the zeta potential technique providing the colloids can be observed with the microscope. The results of this research indicate the feasibility of using the zeta potential technique in conjunction with the jar test to optimize coagulation. The jar test would be used as a guide in establishing coagulant dosage and the zeta meter as a refinement technique.

Feed Control

Positive displacement pumps should be used to control the rate of chemical and waste feed to the continuous unit. Precise control could not be maintained with the rotameters and centrifugal pumps. The pH of the waste during coagulation would be more effectively controlled by feeding the calcium hydroxide as a slurry to the rapid mix chamber.

5. CONCLUSIONS AND RECOMMENDATIONS

Conclusions reached from the coagulation study of an oil-water emulsion waste are:-

1. The emulsion was subject to biological degradation as indicated by a change in oil concentration, change in pH, change in color of the waste, and the release of a sulphide gas.
2. Alum was moderately effective in treating an aged waste over a limited range of coagulant dosage but ineffective for a fresh waste.
3. Of the following chemicals and combinations of chemicals,

   alum; alum and sodium chloride; alum and sulphuric acid; alum and calcium carbonate; dispersonal (polyelectrolyte); ferric chloride; ferric chloride and calcium hydroxide; Alchem 550-R (polyelectrolyte); alum and Alchem 550-R; alum, Alchem 550-R and calcium hydroxide; ferrous sulphate; ferrous sulphate and calcium hydroxide; ferrous sulphate and sodium bicarbonate:

the ferrous sulphate and calcium hydroxide combination gave the best results in breaking the emulsion.

4. The ferrous sulphate - calcium hydroxide combination was effective in breaking the emulsion over a wide range of coagulant dosage.
5. The pH of the waste must be kept in the basic range for effective treatment.
6. The cumulative per cent frequency distribution for colloid mobility of the raw diluted waste, batch effluent and the continuous effluent were log - normal. The range in geometric mean mobilities of the distributions for the raw diluted waste, batch effluent and continuous effluent samples are -1.689 to -2.085, - 1.35 to +0.828 and -1.075 to +1.112 u/sec per E/cm respectively. The standard deviations of the log - normal distributions are approximately the same for all samples.
7. The range in standard deviations for all the distributions of the raw diluted waste, batch effluent and continuous effluent samples were

13.7% to 17.5%, 16.4% to 24.3% and 17.6% to 27.7% respectively and indicates the need to obtain at least 50 measurements of colloid mobility per sample to obtain a reliable median mobility.

8. The colloid mobilities for all the raw diluted waste samples were electronegative. However, the mobilities of the colloids after chemical treatment for the batch sample of Run 3 and for the continuous samples of Runs 11, 8, 3 were electropositive with the mobilities for the remaining samples being electronegative.

9. On the basis of oil removal and colloid mobility the results suggest the jar test might be ineffectual in predicting the optimum coagulant dosage for the continuous unit when the emulsion can be broken over a wide range of coagulant dosage.

10. The circular,electrophoresis cell supplied by Zeta Meter Inc. has limited application for determining colloid mobility of emulsions. Results of this research indicate that 150 mg/l is the approximate permissable maximum oil concentration for colloid observation.

Recommendations proposed as a result of this experimental work are:-

1. Follow up this study with an investigation on a synthetic emulsion waste to obtain correlations of coagulant concentration and pH of coagulation with colloid mobility and oil removal.
   A synthetic sample would eliminate the biological degradation and storage problems.
2. Investigate and propose a suitable electrophoretic mobility technique to permit colloid determinations of emulsions with oil concentrations above 150 mg/l.
3. Use positive displacement pumps for positive waste and chemical feed control in lieu of centrifugal pumps and rotameters.
4. Evaluate the use of ferric sulphate as a coagulant relative to ferrous sulphate for treating a steel rolling mill waste and investigate the use of coagulant aids for removal of pinpoint floc.

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APPENDICES

APPENDIX A: DETAILED EXPERIMENTAL EQUIPMENT DESIGN AND PROCEDURE

A-1 EQUIPMENT DESIGN AND CONSTRUCTION

A-1.1 Introduction

Detailed information presented here is related to the equipment for the continuous unit shown in Figure A-1 and for the batch unit shown in Figure A-2.

The continuous unit consists of feed treatment, coagulation basin and separation units. Appropriate instrumentation was made to all sections. For the batch process, all these functions are satisfied in one vessel through control of the timing cycle.

A-1.2 Rapid Mix Chamber

Design:

The rapid mix chamber was part of the feed treatment. The function of the equipment was to effect adequate contact between the chemicals and the colloids in the waste.

Design criteria suggested by Rich[1] are the Reynolds Number and the detention time, defined as plug flow residence time.

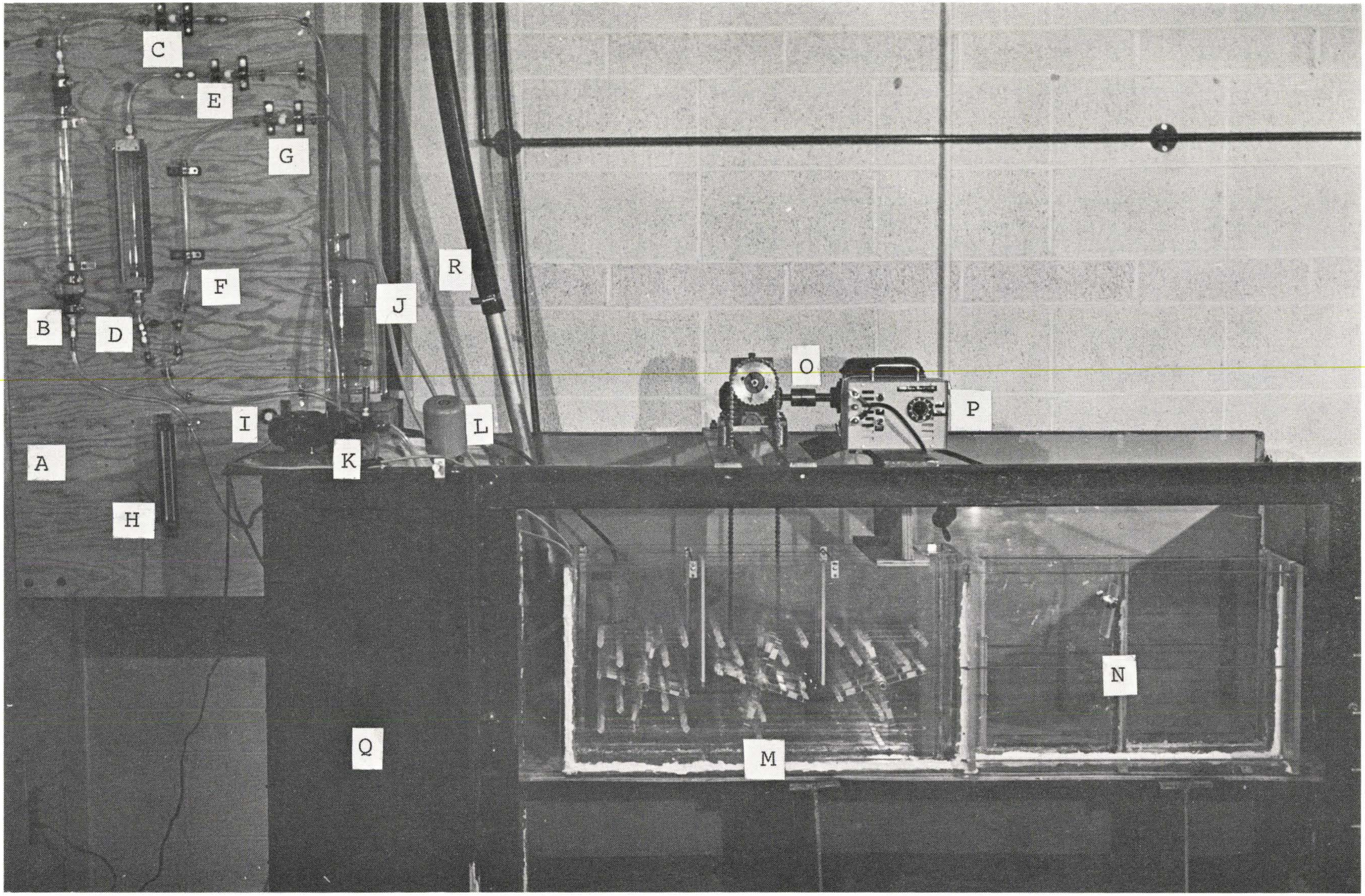
For all experiments the total liquid flow rate was kept constant at 0.174 USgpm.. Tolman[9] suggests that a

FIGURE A-1

PHOTOGRAPH OF THE CONTINUOUS UNIT

LEGEND

A - Rotameter Panel
B - Rotameter Raw Waste Feed
C - Stopcock - Raw Waste Feed Line
D - Rotameter - Coagulator Basin No. 1 Feed
E - Stopcock - Coagulator Basin No. 1 Feed Line
F - Rotameter - Coagulator Basin No. 2 Feed
G - Stopcock - Flocculator Basin No. 2 Feed Line
H - Rotameter - $FeSO_4$ Feed Line
I - Stopcock - $FeSO_4$ Feed Line
J - 8 liter Bottle For $FeSO_4$ Solution
K - Raw Waste Pump
L - Mixer For Rapid Mix
M - Coagulator
N - Separator
O - Coagulator Drive Unit
P - Variable Speed Controller
Q - Constant Temperature Bath
R - Water Line For Constant Temperature Bath



PHOTOGRAPH OF THE CONTINUOUS UNIT

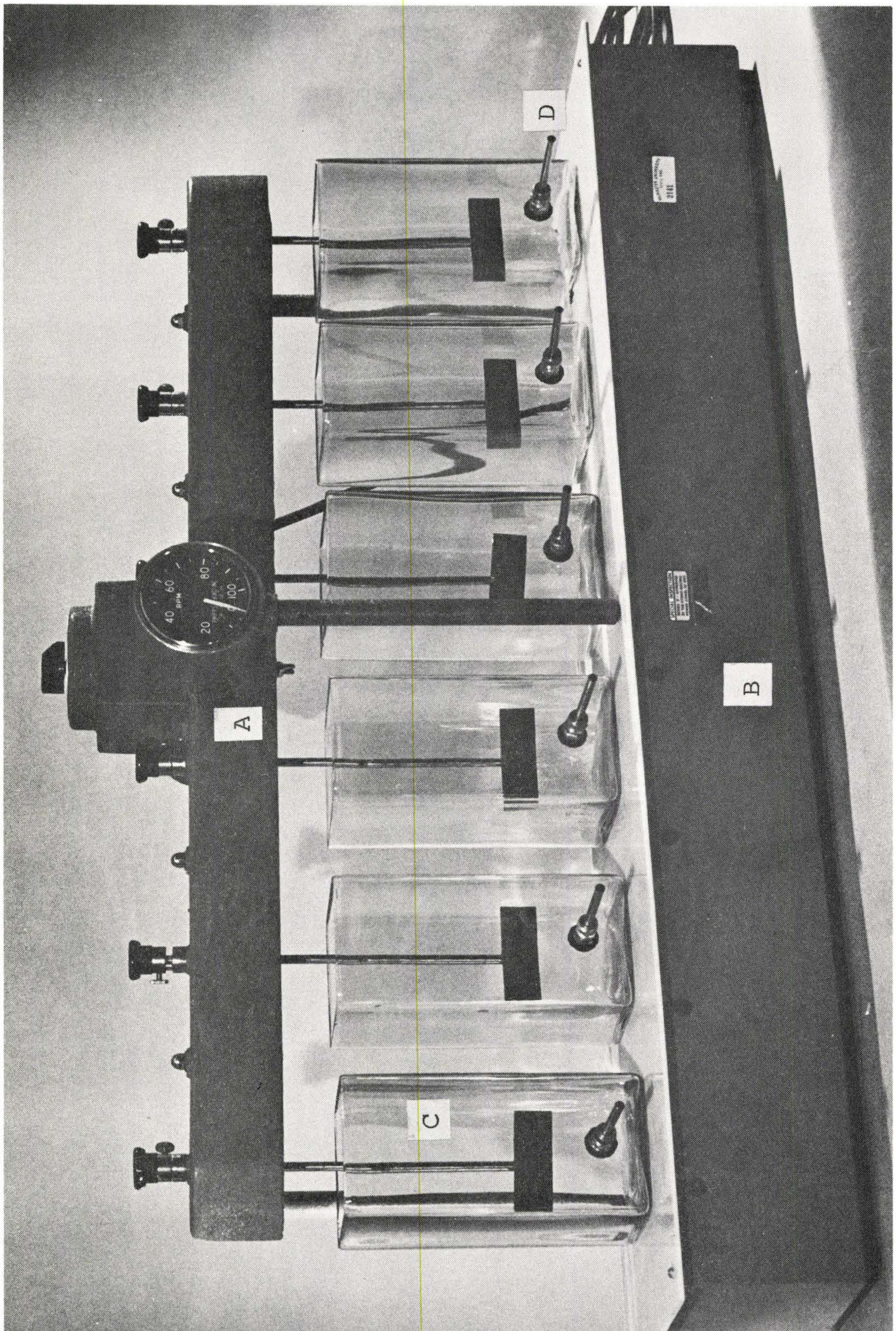
FIGURE A-1

FIGURE A-2

PHOTOGRAPH OF THE BATCH UNIT

LEGEND

A - Variable Speed Multiple Stirrer
B - Stand
C - Typical 1.6 Liter Square Jar
D - Sampling Ports



PHOTOGRAPH OF THE BATCH UNIT

FIGURE A-2

reasonable detention time range from 30 seconds to one minute and therefore 45 seconds was selected. Hence the total volume of the rapid mix chamber was 30.2 in^3. Chamber dimensions were arbitrarily chosen with the internal width and length of 2 3/4 inches, liquid depth of four inches and an overall chamber depth of seven inches with three inches for free board and inlet pipe allowance. Figure A-3 shows chamber details.

Mixer speed was selected on the basis of the following relationship:

$$n = \frac{(Re_I)\mu}{D^2 \rho}$$

The values for the parameters in equation (A-1) are:-

- D = 1.95 inches which is based on criteria of D = 50% to 80% of chamber width as suggested by McCabe and Smith[2].
- $Re_I = 10^4$, based on Rushton's suggestion that for turbulent mixing Re_I equal or exceed 10^4 for a two blade paddle in a baffled chamber[3].
- n = shaft speed, sec^{-1}
- ρ of waste = 62.26 lbs/ft^3 at 24°C
- μ of waste = 1.98×10^{-5} lb - sec/ft^2 at 24°C

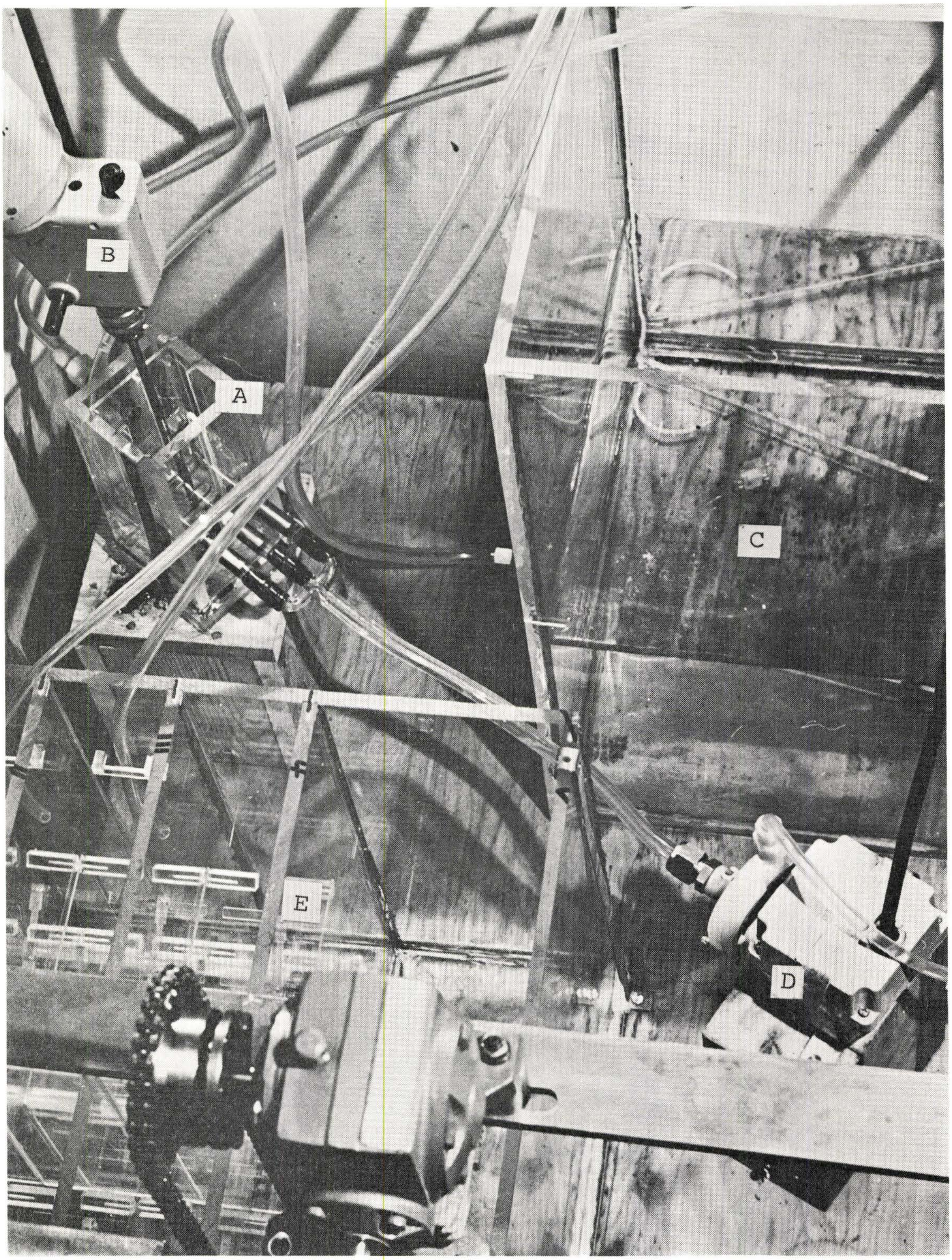
The ρ and μ of waste are assumed equal to ρ and μ of water

FIGURE A-3

PHOTOGRAPH OF RAPID MIX CHAMBER WASTE HOLDING TANK AND COAGULATOR FEED PUMP

LEGEND

A - Rapid Mix Chamber
B - Mixer For Rapid Mix
C - Holding Tank
D - Coagulator Feed Pump
E - Coagulator



PHOTOGRAPH OF RAPID MIX CHAMBER, WASTE HOLDING TANK AND COAGULATOR FEED PUMP

FIGURE A-3

because the quantity of oil in the waste was approximately 90 ppm and the effect of any inorganic colloidal particles present insignificantly affected these two parameters. On this basis the required shaft speed was 225 rpm. Rich[1] and Rushton[3] suggest paddle widths of 1/6 to 1/10 of paddle length and therefore 0.27 inches was selected. The paddle was selected instead of an impeller or turbine for easy construction.

A solid vertical inlet baffle was placed perpendicular to and ½ inch horizontally from the inlet to the chamber and extends vertically from the top of the chamber to two inches below the centerline of the 3/8" diameter inlet. The function of the baffle was to reduce short circuiting between the inlet and outlet of the chamber..

Four chamber baffles were used to prevent bulk rotational flow. The criteria given by Rich and McCabe and Smith suggest a baffle width equal 1/10 - 1/12 of chamber diameter. On this basis a baffle width of 0.28 inches was selected.

The outlets were 3 equally spaced, ¼ inch diameter openings located 2½ inches above the bottom of the chamber.

Construction:

The chamber and baffles, shown in Figure A-3, were constructed of plexiglas and assembled by gluing the component parts together with tensol glue. Stainless steel nipples served as adaptors between the chamber inlet and outlets and tygon waste feed lines.

A-1.3 Coagulator

Basin Desgin:

The coagulator was part of the waste separation phase of the treatment, and its function was promotion of orthokinetic coagulation.

Criteria for design of full scale units are velocity gradient and a dimensionless group which involved the product of velocity gradient and time[4]. The range of values for G_m and G_mT acceptable for design are 10 to 75 sec^{-1} for G_m and and 23,000 to 210,000 for G_mT [4,6,17].

The coagulator was divided into two separate parallel basins with each basin handling one-half of the total flow. Partitioning each basin into three compartments in series by solid vertical baffles reduced short-circuiting. The terms G_m and G_mT are progressively decreased in each successive compartment to promote floc growth. These values are as given in Table A-1.

Table A-1

Design Values for Compartments

Compartment	G_m	G_mT
1	70	70,000
2	47.6	42,800
3	19.13	21,050

Plug flow detention time for design was determined by equation (A-2)[4].

$$t = \Sigma \frac{G_mT}{G_m} \times \frac{1}{60} \quad - - - - \quad (A\text{-}2)$$

$$t = \left(\frac{70,000}{70} + \frac{42,800}{47.6} + \frac{21,050}{19.13} \right) \times \frac{1}{60}$$

$$t = 50 \text{ minutes}$$

This was in agreement with design criteria of 40 to 60 minutes suggested for t [5,6,8]. Therefore the selection of t and Q resulted in a basin volume of 0.588 ft^3 with corresponding values of 3", 30", 11 7/32" and 15" for the width, length, liquid depth and total depth respectively.

Rotor Design:

Each compartment had a rotor whose function was to create fluid shear and therefore keep the chemical in contact with all portions of the fluid mass. Rotor size was based on criteria[9] which suggested rotor diameter equal or exceed

60 per cent of the cross sectional area swept. This gave a rotor diameter of seven inches. To facilitate construction the rotor diameter for each compartment was set at seven inches.

Paddles for the rotors were designed on the basis of the relationship between G_m, W and μ proposed by Camp [4]. Camp's design equations were:-

$$G_m = \sqrt{\frac{W}{\mu}} \quad \text{- - - - - -} \quad \text{(A-3)}$$

$$W = \frac{239\ C_d\ (1-K)^3\ (n)^3\ \Sigma\ A(r_b)^3}{V} \quad \text{- - - -} \quad \text{(A-4)}$$

Substitution of the value for W of equation (A-4) in equation (A-3) and rearranging yields the design equation for the area of all paddles for a rotor as follows:-

$$\Sigma\ A(r_b)^3 = \frac{(G_m)^2\ V\ \mu}{239\ (n)^3\ C_d\ (1-K)^3} \quad \text{- - - -} \quad \text{(A-5)}$$

In equation (A-5) the values for the parameters for the design of the rotor paddles in compartment number one are:-

- C_d = 1.2 which is based on drag coefficient given in Rouse [16] for flat plates
- K = 0.32 on the basis of Camp [4]
- μ = 1.98×10^{-5} lb.-sec/ft^2 at 24°C
- V = 0.195 ft^3 for equation (A-5)

- $G_m = 70 \text{ sec}^{-1}$
- $n = 0.408$ rps by a paddle tip speed of 0.75 fps which was based on suggested criteria [6,8] of 0.75 - 1.5 fps for tip speed. Shafts for all rotors were run at the same speed.

The solution of equation (A-5) resulted in a rotor with four arms and two paddles per arm as shown schematically in Figure A-4. A paddle with length and width of 2½ inches by 9/32 inches respectively was selected for all rotor arms. Stators are used in the design, therefore no limitations are placed on paddle area to prevent bulk rotational flow [4].

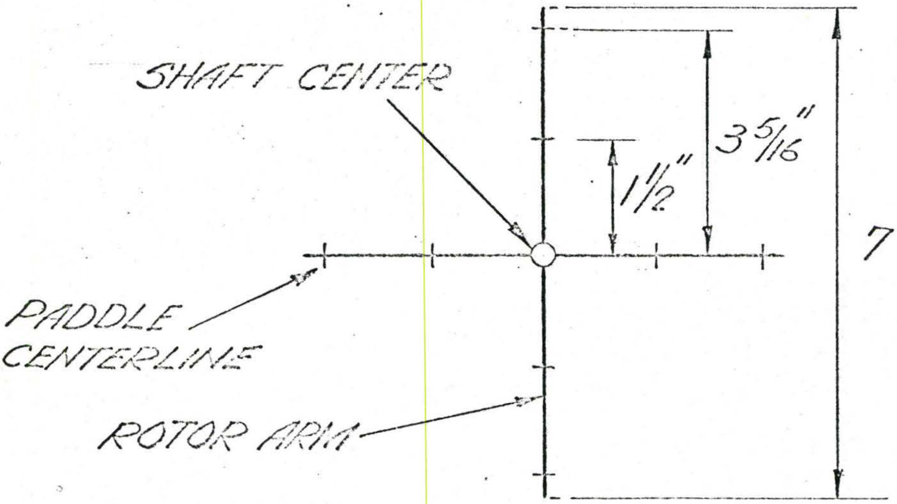
Similarly rotors for compartments two and three were designed on the basis of equation (A-5). The pertinent data for the rotors are summarized in Table A-2.

Table A-2

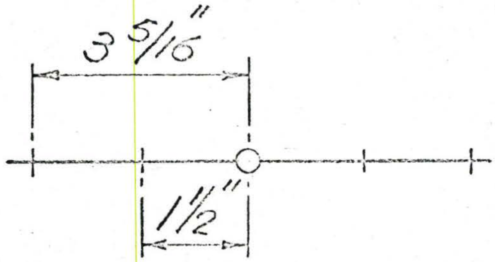
Summary of Rotor Design

Rotor	Rotor Arms	Paddles Per Arm	Paddle Dimensions (inches) Length	Width
1	4	2	2½	9/32
2	2	2	2½	7/16
3	2	1	2½	7/32

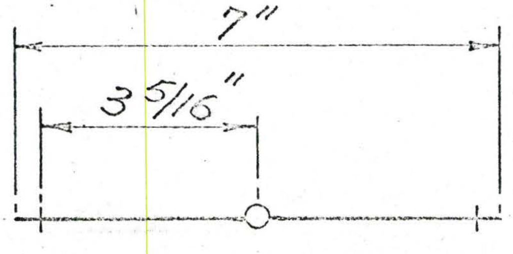
Relative locations of paddles on the rotor arms are illustrated in Figure A-4.



ROTOR #1



ROTOR #2



ROTOR #3

ROTOR SCHEMATICS

FIGURE A-4

Stator Design:

Information on stator design in the literature is meagre. Researchers agree that they are required to prevent bulk rotational flow of the liquid by producing shear in the liquid between the paddle, the movable part, and the stator, the stationary part. At the Cambridge Massachusetts Water Treatment Plant $\Sigma A(r_b)^3$ for the stators were designed approximately equal to $\Sigma A(r_b)^3$ for the paddles. For this design it was decided to supply stators as shown in Table A-3.

Table A-3

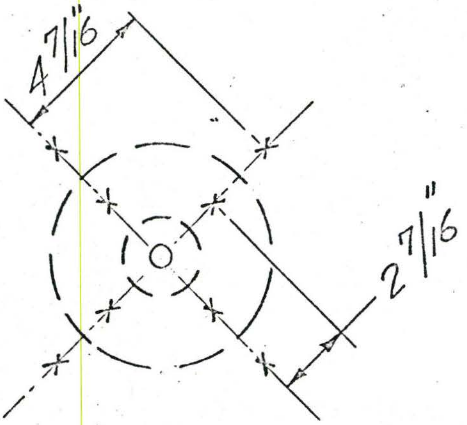
Summary of Stator Design

Rotor Number	Pairs of Stators	Stator Width (inches)
1	8	3/8
2	6	3/8
3	6	3/8

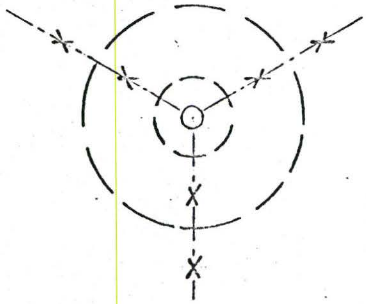
The size was arbitrarily selected to give sufficient contact for gluing to ease construction. Stators were positioned as shown in Figure A-5 for each rotor.

Construction:

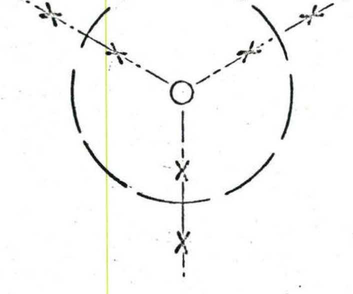
The coagulator, shown in Figure A-6, was constructed of 3/8 inch wide plexiglas and the component pieces were



ROTOR #1



ROTOR #2



ROTOR #3

– LEGEND –

X – STATOR POSITION

◌ – ROTATIONAL PATH OF PADDLE

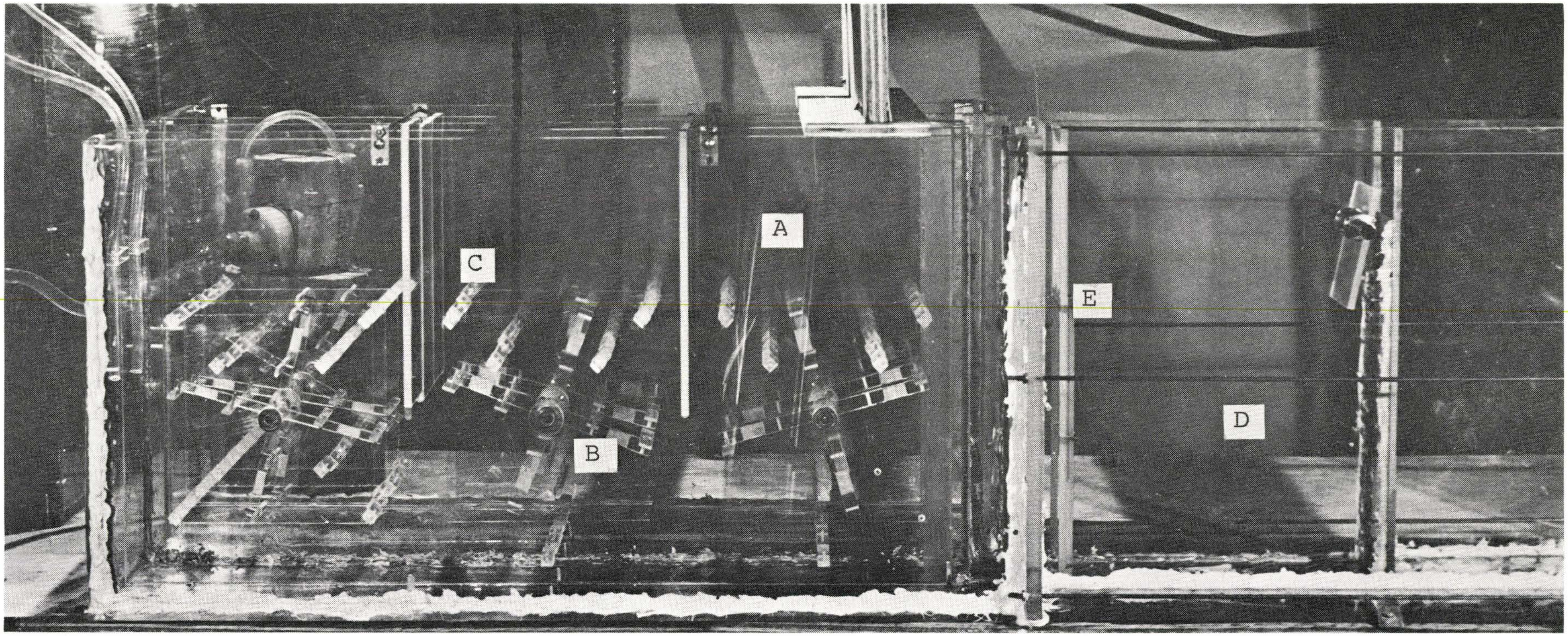
STATOR LOCATION
FIGURE A-5

FIGURE A-6

PHOTOGRAPH OF THE COAGULATOR

LEGEND

A - Coagulation Unit
B - Rotor
C - Stator
D - Separator
E - Vertical Slotted Baffles



PHOTOGRAPH OF THE COAGULATOR

FIGURE A-6

glued together with tensol glue. Joints were sealed with silicone rubber cement to prevent leakage. The rotors made of plexiglas, were secured to the three stainless steel shafts with set screws. Paddles on the rotors were fabricated from plexiglas and attached to the rotor arms with stainless steel screws and spot glued to prevent rotation.

Ball bearing sleeves reduced shaft friction. Leakage from one basin to the next, at the ball bearing sleeves, was prevented by o-ring seals.

Stators were made of plexiglas and were glued to the sides of the basins.

The sprockets on each shaft were chain driven by a variable speed D-C motor - gear reducer combination unit shown in Figure A-6. A flexible mechanical coupling was used to connect the motor to the gear reducer.

A-1.4 Separator

Basin Design:

Criteria for the design of the separator are overflow rate and detention time, defined as the plug flow residence time.

Values for the above parameters, applicable for a coagulated oily waste does not appear to be established in the literature. Indications are that each emulsified waste should be treated separately [22]. Pilot plant studies on coagulation of an emulsified waste used an overflow rate of 865 US gals./ft^2/day [24]. Detention times, from nine to thirty seven minutes for unstable emulsions have been reported for design [23].

The separator for this project was initially designed for a floating alum floc on the basis of preliminary studies in March 1968. However the design was subsequently modified in September 1968 to accomodate a settling iron floc. The criteria for design were arbitrarily selected with the overflow rate at 500 US gals./day/ft^2 and a plug flow detention time of twenty one minutes. The constant temperature bath placed limitations on the size of the separator. Chamber dimensions were 3", 24", 11 9/16" and 15" for the width, length, average liquid depth and an overall basin depth respectively.

Inlet Design:

Two vertically slotted baffles were placed at the separator inlet to distribute the flow uniformly across the separator cross section. The criteria for baffle design of full scale separator units have been developed [18]. An attempt was made to scale down on a minimum velocity of 0.1 fps through the first baffle and an approximate lower limiting value for baffle R_e of 150.

For the first baffle a minimum of three slots, each with a slot width of 1/16 inch and height of 11 9/16 inches, was selected to prevent breakdown of floc size. With a Q per slot of 1.29×10^{-4} cfs., the slot velocity and baffle R_e were 0.026 fps. and 26.4 respectively. Alternative means of attaining a velocity of 0.1 fps through the baffle were reducing the effective height of all three slots to 2 5/8 inches or reducing the number of slots to one or two. However, it was decided to sacrifice some of the basin performance rather than destroy floc size and therefore three slots were selected.

The solid portions of second baffle were ½ inch wide and were placed behind each slot of the first baffle[18]. The distance between baffles was ½ inch.

<u>Outlet Design</u>:

Effluent was discharged through a ½ inch outlet located at the top of the water level.

<u>Construction</u>:

The separator was constructed of plexiglas. Provision was made to alter the size of the separator by assemblying the sections together with tie rods and clamps. Joints were sealed with silicone cement to prevent leakage.

A-1.5 Holding Tank

The waste for an experimental run was placed in a 14¼ inch x 17¼ inch x 22 inch deep plexiglas tank as shown in Figure A-3. The component parts were held together with glue and screws and the joints sealed with the silicone cement.

A-1.6 Constant Temperature Bath

A 3 ft. x 6 ft. - 9 ins. x 3 ft. deep steel frame tank with a plexiglas viewing panel, shown in Figure A-1, was used to maintain the equipment for the continuous unit at a constant temperature. This was achieved by passing a continuous flow of premixed water at a given temperature through the tank. A tele - thermometer was used to indicate the bath temperature continuously.

A-1.7 Batch Process - Jar Modifications

Modification to the standard 1.6 liter jars involved placing outlets at the base of the jars. This is shown in Figure A-2. Swagelok fittings were glued into holes drilled in the glass. To draw off liquid from the settled sample stainless steel tubing blanked and slotted at one end was used.

A-2 EQUIPMENT DESCRIPTION AND OPERATION

A-2.1 Description

The equipment specifications and suppliers are given in Table A-4.

A-2.2 Operation

Both the batch and continuous processes had equal plug flow detention times of 45 seconds for the rapid mix, 50 minutes for the coagulation and 21 minutes for the separation. The operation of the two processes is described more fully as follows.

Continuous Unit

Prior to Startup:

Calcium hydroxide was added to the waste in the holding tank for pH adjustment and kept suspended with a propeller mixer. In addition, calcium hydroxide and ferrous sulphate were added, in the required quantities, to the waste contained in the coagulator and separator. The contents were then thoroughly mixed for approximately five minutes. This batch type operation eliminated the time lag in reaching steady state. Dye tests were conducted on the continuous unit before making the modifications to the separator in September of 1968. These tests revealed that the coagulator

Table A-4

EQUIPMENT SPECIFICATIONS AND SUPPLIERS

EQUIPMENT	WHERE USED	SPECIFICATION DATA	SUPPLIER
Raw Waste Feed Pump	Continuous Unit	Eastern - Model B_1 Centrifugal	Chemical Eng. Dept. Room A314 McMaster University Hamilton, Ontario
Coagulator Feed Pump	Continuous Unit	Cole - Parmer Model 2 E-14NDVR Centrifugal	Chemical Eng. Dept. Room A314 McMaster University Hamilton, Ontario
Holding Tank	Continuous Unit	Plexiglas	Machine Shop Engineering Building McMaster University Hamilton, Ontario
Raw Waste Feed Rotameter	Continuous Unit	Fischer - Porter Flowrator Tube No. B4 27-10/77 Stainless Steel Float	Chemical Eng. Dept. Room C257 McMaster University Hamilton, Ontario
Coagulator Feed Rotameter No. 1	Continuous Unit	Flowrator Tube No. RBM-127-4FBR Stainless Steel Float Flow range: 0.07 - 0.7 USgpm	Brooks Instrument Division 32 A Howden Road Scarborough, Ontario

Table A-4 (continued)

EQUIPMENT	WHERE USED	SPECIFICATION DATA	SUPPLIER
Coagulator Feed Rotameter No. 2	Continuous Unit	Fischer - Porter Flowrator Tube No. 2-L-150/5-51801 Stainless Steel Float	Chemical Eng. Dept. Room C257 McMaster University Hamilton, Ontario
Ferrous Sulphate Feed Rotameter	Continuous Unit	Fischer - Porter Flowrator Tube No. FP-1/8-25-G-5/81 Stainless Steel Float	Chemical Eng. Dept. Room C257 McMaster University Hamilton, Ontario
Rotameter Panel	Continuous Unit	Plywood	Machine Shop Engineering Building McMaster University Hamilton, Ontario
Rapid Mix Chamber	Continuous Unit	Plexiglas panels, Stainless Steel Nipples	Machine Shop Engineering Building McMaster University Hamilton, Ontario
Coagulator - Separator	Continuous Unit	Plexiglas panels, plexiglas rotors, stainless steel shafts, steel sprockets ball bearing sleeves, 0-ring seals, plexiglas baffles	Machine Shop Engineering Building McMaster University Hamilton, Ontario

Table A-4 (continued)

EQUIPMENT	WHERE USED	SPECIFICATION DATA	SUPPLIER
Holding Tank Mixer	Continuous Unit	Palo Laboratory Type V-10, 5000 rpm 115 V AC-D.C.	Chemical Eng. Dept. Room C257 McMaster University Hamilton, Ontario
Flash Mixer	Continuous Unit	Can-Lab Caframo - Type D73-40 115 V A.C.	Chemical Eng. Dept. Room A7 McMaster University Hamilton, Ontario
Coagulator Motor Drive	Continuous Unit	Westinghouse Type FK 120 volt DC. 1/6 H.P. 1725 RPM	Power Service Products 147 Kipling Avenue S. Toronto 18, Ontario
Gear Reducer	Continuous Unit	Catalogue No. T-113-30 Reduction Ratio 30:1 Input RPM 1725 Output Torque 186 in-lbs.	Power Service Products 147 Kipling Avenue S. Toronto 18, Ontario
Coagulator, Drive Chains, Drive sprocket and Flexible Coupling	Continuous Unit	Drive chain - Rex chain RC-35 Drive Sprocket - 35H30	Vickers - Warnick Ltd. 1869 Main St. E. Hamilton, Ontario

Table A-4 (continued)

EQUIPMENT	WHERE USED	SPECIFICATION DATA	SUPPLIER
Tygon Tubing	Continuous Unit	5/16" O.D., 3/8" O.D., ½" O.D., 11/16" O.D.	Chemistry Stores Senior Science Building McMaster University Hamilton, Ontario
Swagelok Fittings	Continuous and Batch Units	Type 316 Stainless Steel and Brass	Niagara Valve 102 Parkdale Avenue N. Hamilton, Ontario.
Stopcocks	Continuous Unit	Waste lines - 6 mm., glass body, teflon plugs Chemical feed - 4 mm., glass body, teflon plug	Canlab 80 Jutland Road, Toronto 18, Ontario
Caulking Cement	Continuous Unit	Dow Corning RTV-733 Silicone	Chemical Eng. Dept. Room A314 McMaster University Hamilton, Ontario
Variable Speed Controller for Coagulator Drive	Continuous Unit	115 V. AC-DC	Electrical Maintenance Department Senior Science Building McMaster University Hamilton, Ontario.

Table A-4 (continued)

EQUIPMENT	WHERE USED	SPECIFICATION DATA	SUPPLIER
Tele- Thermometer	Continuous Unit	Range: 32°F - 122°F 0°C - 50°C	Chemical Eng. Dept. Room C257 McMaster University Hamilton, Ontario
Multiple Stirrer	Batch Unit	Phipps and Bird Speed range - 0-100rpm 115 V A.C.	Chemical Eng. Dept. Room C257 McMaster University Hamilton, Ontario
Square Jars	Batch Unit	glass, 1.6 liters	Canlab 80 Jutland Road Toronto 18, Ontario
pH Meter	Analysis	Beckman Model N	Chemical Eng. Dept. Room C257 McMaster University Hamilton, Ontario
Titration Equipment	Analysis	glass burette - 0-50 ml capacity 0.1 increments Stand and burette holder	Chemical Eng. Dept. Room C257 McMaster University Hamilton, Ontario.

Table A-4 (continued)

EQUIPMENT	WHERE USED	SPECIFICATION DATA	SUPPLIER
Zeta Meter	Analysis	Std. Power and Timer unit, Type 11 UVA Electrophoresis cell. Std. Sterescopic Microscope and Illuminator	Zeta Meter Incorporated 1720 First Avenue New York, N.Y. 10028
Mixer and Variable Speed Controller (Oil Determinations)	Analysis	SMP motor, Model B Controller - 115 V A.C.	Chemical Eng. Dept. Room A314 McMaster University Hamilton, Ontario
Separatory Funnels	Analysis	500 ml. glass body, glass plugs	Chemistry Stores Senior Science Building McMaster University Hamilton, Ontario
Buchner Funnels	Analysis	7.0 cm.	Chemistry Stores Senior Science Building McMaster University Hamilton, Ontario.
Filter Paper	Analysis	Glass Fiber, 7.0 cm.	Fisher Scientific Toronto, Ontario

Table A-4 (continued)

EQUIPMENT	WHERE USED	SPECIFICATION DATA	SUPPLIER
Water Bath	Analysis	Canlab Gallenkamp 5 hole, aluminum alloy bath	Chemistry Stores Senior Science Building McMaster University Hamilton, Ontario
Balance	Analysis	Mettler, Type H15	Chemical Eng. Dept. Room C257 McMaster University Hamilton, Ontario.
Conductivity Meter	Analysis	Type CDM 2d Radiometer Copenhagen	Chemical Eng. Dept. Room A316 McMaster University Hamilton, Ontario.
Helige Turbidimeter	Analysis	115 V AC. - D.C.	Chemical Eng. Dept. Room C257 McMaster University Hamilton, Ontario
Brookfield Viscometer	Analysis	Synchro - Electric Model HBT	Chemical Eng. Dept. Room C257 McMaster University Hamilton, Ontario.

approximated a CSTR and traces of dye appeared in the effluent from the separator after approximately eight minutes had elapsed. The corresponding plug flow detention time for the coagulator - separator unit was 60.5 minutes.

Startup and Run

The procedure was:-

- Start the raw waste pump and remove the plug from the suction line in the waste holding tank.
- Open stopcock on raw waste feed line to approximate setting.
- Open stopcock on ferrous sulphate feed line and adjust to give the correct rotameter setting.
- Start the coagulator feed pump and open stopcocks for the coagulator basin feed lines.
- Adjust stopcocks on the waste feed lines until rotameter settings are correct.
- Observe operation and check effluent pH periodically. Adjust pH, if required, by adding more lime.
- After a time lapse of approximately one to two detention times, a two liter sample was collected for each run.

Batch Unit

Prior To Startup:

A trial run was made for each experimental run with one jar to determine the amount of lime required for pH adjustment. Having conducted the trial run the following procedure was followed for the experimental run:-

- Place one liter of waste in the jar.
- Add required amount of ferrous sulphate to each of the three jars.
- Add quantity of lime as determined by the trial run.
- Rapid mix for 45 seconds.
- Check pH near the end of the rapid mix cycle and adjust if necessary.
- Coagulate for 50 minutes followed by quiescent settling for 21 minutes.
- Collect a sample from each jar through the sampling tubes.

A-3 SAMPLE ANALYSES

A-3.1 Introduction

Analyses were conducted on the raw waste, batch effluent and continuous effluent. The analyses are discussed in detail in the following sections.

A-3.2 Colloid Mobility

Colloid mobility was determined with the zeta meter equipment shown in Figure A-7.

The electrophoresis cell was stoppered and then the chambers were filled with the sample. Air bubbles were eliminated from the cell by easing off the stoppers. After the air bubbles had been released, the anode and cathode were placed in the chambers. The electrophoresis cell was then mounted on the mechanical stage of the microscope, alligator clips connected and the illuminator positioned for proper illumination.

The first step in cell electrophoresis is the determination of the specific conductance of the sample. This was accomplished by :-

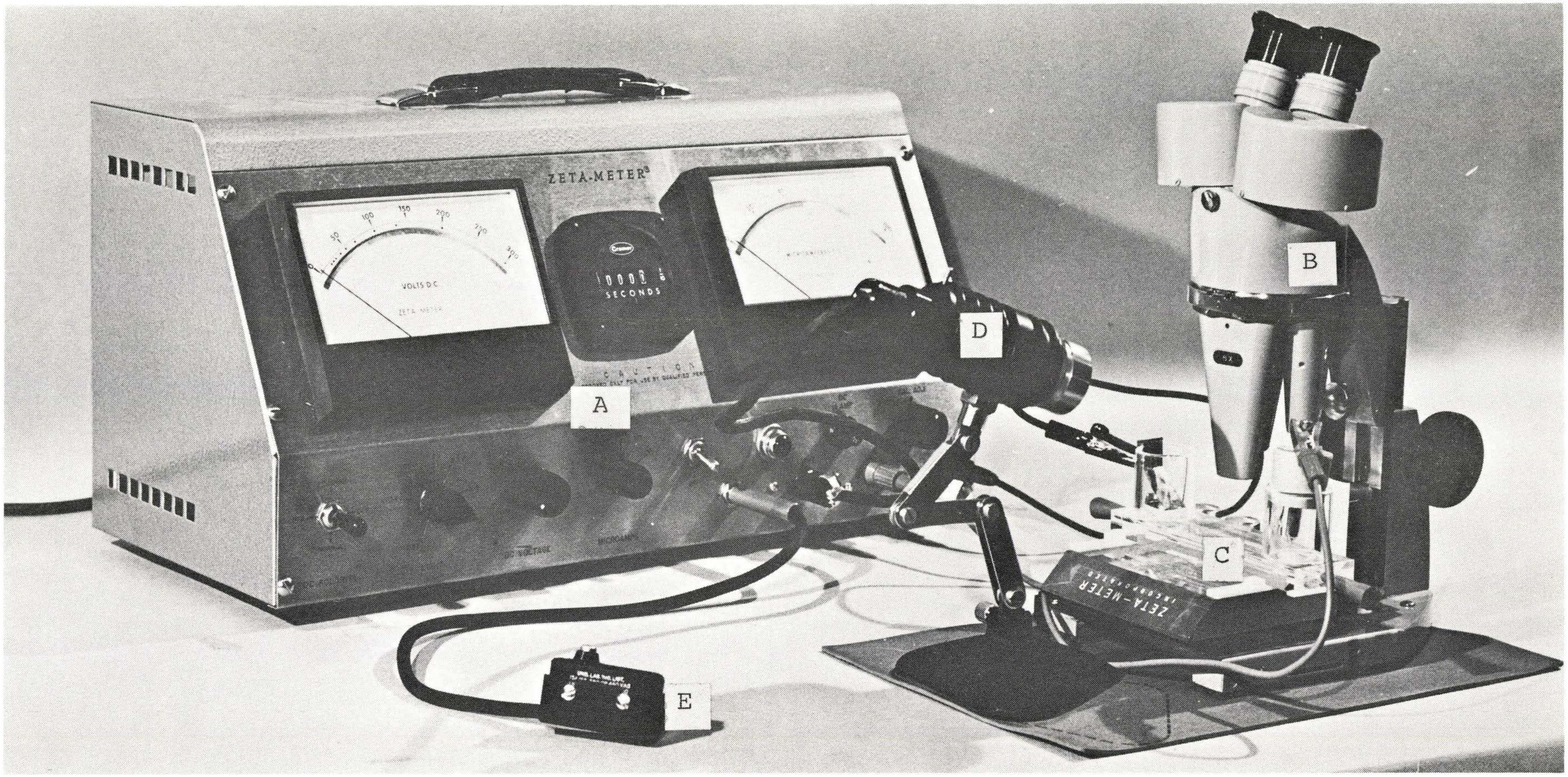
- impressing a voltage across the cell equal to the cell constant

FIGURE A-7

PHOTOGRAPH OF ZETA METER EQUIPMENT

LEGEND

A - Zeta Meter Power Unit
B - Sterescopic Microscope
C - Electrophoresis Cell
D - Microscope Illuminator
E - Timing Switch



PHOTOGRAPH OF ZETA METER EQUIPMENT

FIGURE A-7

- reading the ammeter which in turn gives the specific conductance of the sample directly in micromhos/cm.

Knowing the specific conductance, the voltage for cell electrophoresis can be established. The voltage was set at the determined value, the D.C. polarity, ammeter and illuminator switches turned on and colloid tracking commenced.

A-3.3 pH and Alkalinity

Fifty ml samples were used to conduct the pH and alkalinity measurements. The pH meter was calibrated before use each day with a phosphate buffer solution which had a pH of 6.86. pH and alkalinity determinations were performed on the same sample. The potentiometric titration method, outlined in Standard Methods[42], was used for alkalinity. Samples were titrated to a pH endpoint of 4.5 with 0.02N H_2SO_4.

A-3.4 OIL DETERMINATION

Procedure

Extraction methods outlined in Standard Methods were not applicable for an emulsion type waste and therefore a new procedure was developed. The oil determination procedure is:-

(a) Set up extraction apparatus in fume hood.

(b) Place a 200 ml. sample in a 300 ml. mixing beaker.

(c) Add salt at a concentration of 25 gm/1 and then mix the contents at 2000 rpm with a mixer for two minutes[30].

(d) Add 50 ml. of CCl_4 to the beaker and mix again at 2000 rpm for two minutes.

(e) Place the contents of the beaker in the separatory funnel and allow the CCl_4 and water phase to separate.

(f) After approximately three hours, drain the CCl_4 (lower phase) into a 125 ml. flask.

(g) Place a glass fiber filter paper in a 7.0 cm. Buchner funnel and wet it with CCl_4. This prevents water from passing through the filter paper when filtering.

(h) Pour the flask contents slowly into the Buchner funnel. Filter by gravity into a tared 100 ml. beaker.

(i) Drain the liquid sample phase, in the separatory funnel, into the mixing beaker and repeat steps (b) through (i) until extraction complete.

(j) Place the beaker containing the filtrate on a steam bath and evaporate the CCl_4.

(k) After the final extraction is made, the separatory funnel and mixing beaker are rinsed with 10 ml. of CCl_4 and then placed in the Buchner funnel.

(l) Then the 125 ml. flask and Buchner funnel are rinsed with 10 ml. of CCl_4 The filtrate is collected in the 100 ml. beaker and evaporated as before.

(m) When evaporation of CCl_4 has been completed, wipe the outside of the beaker clean and place in the dessicator to cool.

Difficulties Encountered

Initially toluene was used as the solvent material. Its main drawbacks were:-

(a) a high boiling point of 110.6°C which made evaporation difficult and very time consuming.

(b) specific gravity was less than one and therefore the toluene had to be drawn off last. As a result, water drops retained on the sides of the separatory funnel were drawn off with the toluene. Therefore a trace of salt was present in the evaporated sample and made the oil determination cumbersome and less precise.

(c) an intermediate phase existed with some oil in the emulsified state suspended in toluene.

Hexane was tried next but the difficulties encountered in (b) and (c) for toluene prevailed with hexane as well. Use of Carbon tetrachloride eliminated all the difficulties encountered with the other solvents as it had a low boiling point of 76.8°C and was heavier than water. However, special care had to be taken because of the toxicity of carbon tetrachloride.

For the first two runs the stopcocks of the separatory funnels were greased at the outside edges with a silicone stopcock grease to prevent leakage. It was found that the grease was slightly soluble in the solvent and therefore the remainder of the trials were performed without the grease being applied. To overcome this, separatory funnels with teflon stopcocks are recommended for future experimentation.

A-4 RAW WASTE AND CHEMICAL SOLUTION PREPARATION

A-4.1 Raw Waste

The waste sample from the steel mill was diluted by adding three volumes of tap water to one of waste to have a sufficient quantity on hand for the experimental runs. As oil concentration was held constant during the testing program, it was mandatory that the sample be consistent from day to day. It was found that waste samples obtained from steel plant varied considerly in oil concentration from day to day and therefore this ruled out the possibility of obtaining waste samples daily.

The tap water used as dilution water for the experimental runs was basically obtained from the same source as the water which constitutes a large percentage, by volume, of the oily waste sample.

A sufficient quantity of diluted waste was placed in the holding tank for the experimental run. In addition to this the flocculator and separator were filled with the diluted waste. Note that in the main body of the thesis the diluted oily waste is referred to as raw waste to differentiate between treated and the untreated waste.

A-4.2 Chemical Solutions

Chemical stock solutions for both the batch and continuous process were prepared by weighing out the quantity of chemical, placing it in a volumetric flask and diluting it with distilled water to give the required concentration of stock solution.

Chemicals were transferred from the volumetric flasks to the jars of the batch process by pipette. At the beginning of each run chemical quantities required in the holding tank and in the coagulator and separator were measured out in graduate cylinders. Ferrous sulphate solution, which is fed continuously to the rapid mix chamber, is contained in a 8 liter glass bottle.

APPENDIX B: EFFECT OF DILUTION OF OILY WASTE ON ELECTROPHORETIC MOBILITY

Electrophoretic mobilities for various dilutions of tap water to waste are shown in Table B-1. There appears to be a slight general overall increase in the mobilities of the oil drops from 1.72 at a 2:1 dilution to 1.92 at a dilution of 10:1. The largest change in mobility occurs from the 2:1 to the 3:1 dilution. From 3:1 on, the mobility appears to be affected very little with the exception of the unexplainable variation in the 7:1 dilution.

In view of the slight variations obtained in the colloid mobility measurements of raw waste during the experimental runs and because not all the particles had the same charge, a minimum of 50 measurements were taken for each dilution.

TABLE B-1

EFFECT OF DILUTION ON COLLOID MOBILITY

Objective lens 8X, Volts 250 and Distance traversed = 120 microns

Dilution			Sample Temperature °C		S.C.* of Sample (micromhos)	Colloid Tracking Time			E.M. @ ** 22.5°C u /sec per E/cm.	Comments
Dilution Ratio	Tap Water (ml)	Sample (ml)	Before	After		Cumulative (secs)	Individual (secs)	Median (secs)		
2:1	40	20	22.0	22.5	350	2.8	2.8	2.8	-1.72	Some difficulty in distinguishing colloids plainly
						6.1	3.3			
						8.7	2.6			
						11.5	2.8			
						14.2	2.7			
						17.0	2.8			
						19.8	2.8			
						22.5	2.7			
3:1	60	20	22	23	340	2.4	2.4	2.45	-1.96	
						4.7	2.3			
						7.2	2.5			
						9.8	2.6			
						12.3	2.5			
						14.7	2.4			
						17.3	2.6			
						19.7	2.4			

* means specific conductance
** means electrophoretic mobility

TABLE B-1 continued

Dilution			Sample Temperature °C		S.C.* of Sample (micromhos)	Colloid Tracking Time			E.M. @ ** 22.5°C (u/sec per E/cm.)	Comments
Dilution Ratio	Tap Water (ml)	Sample (ml)	Before	After		Cumulative (secs)	Individual (secs)	Median (secs)		
7:1	105	15	22.0	22.5	330	2.4	2.4			
						4.6	2.2			
						6.9	2.3			
						9.5	2.6			
						11.8	2.3	2.4	-2.0	
						14.4	2.6			
						16.8	2.4			
						19.5	2.7			
10:1	100	10	22.0	22.5	320	2.2	2.2			(a) In all dilutions some threadlike particles were visible (b) These could be: - microrganisms - inorganic contaminants
						4.7	2.5			
						7.6	2.9			
						10.1	2.5	2.5	-1.92	
						12.6	2.5			
						14.9	2.3			
						17.4	2.5			
						19.9	2.5			

* means specific conductance

** means electrophoretic mobility

TABLE B-1 continued

Dilution			Sample Temperature ^{o}C		S.C. * of Sample (micromhos)	Colloid Tracking Time			E.M. @ ** 22.5^{o}C u /sec per E/cm.	Comments
Dilution Ratio	Tap Water (ml)	Sample (ml)	Before	After		Cumulative (secs)	Individual (secs)	Median (secs)		
4:1	40	10	22.0	22.5	330	2.3	2.3			
						4.8	2.5			
						7.3	2.5			
						10.1	2.8			
						12.9	2.8	2.5	-1.92	
						15.1	2.2			
						17.6	2.5			
						19.7	2.1			
						22.3	2.6			
5:1	50	10	22	23	330	2.4	2.4			
						5.3	2.9			
						8.4	3.1			
						11.0	2.6			
						13.4	2.4	2.45	-1.96	
						15.8	2.4			
						18.3	2.5			
						20.7	2.4			

* means specific conductance
** means electrophoretic mobility

APPENDIX C: ZETA METER

C-1 CALIBRATIONS

C-1.1 Determination of Electrophoresis Cell Constant

Calibration was performed as outlined in the Zeta Meter manual [25]. A 0.001 N KCl solution with a specific conductance of 141 micromhos at 25°C was used in the determination of the cell constant. The specific conductance of the KCl solution was determined with a conductivity meter.

The cell constant was determined by the following relationship:-

$$\text{cell constant} = K_1 = \frac{SC \times E}{I} \qquad ---- \text{(C-1)}$$

The procedure for determining the cell constant was:-

- fill the cell with the standard KCl solution at 25°C
- place the electrodes in the cell chambers
- impress a voltage across the cell between electrodes in order to give an ammeter reading of 300 microamps.
- record voltage and current

Data collected for the determination of K_1 is given in Table C-1.

Table C-1

K_1 Determination for Electrophoresis Cell

SC of Standard KCl Solution (micromhos)	Temperature Standard KCl Solution	Voltmeter Reading (volts)	Ammeter Reading (micro-amps)	Cell Constant = K_1 (Dimensionless)
141	25	132	300	62.1
141	25	132	300	62.1
141	25	131	300	61.6
141	25	131	300	61.6

Mean value for K_1 from the above data is 61.85.

C-1.2 Adjustment of Ammeter Shunts

This adjustment was carried out in accordance with the procedure outlined in the Zeta Meter manual [25].

Three standard resistors, 300K, 30K and 3K supplied with the Zeta Meter were employed to adjust the ammeter shunts.

C-1.3 Microscopic Adjustments

Procedures set out in the Zeta Meter manual [25] were followed in making the following adjustments for the eyepiece width, synchronizing the focal lengths of eyepieces and eliminating collimation of objective lens. The first two adjustments should be made by every microscope operator because these settings vary with each individual.

C-2 DIFFICULTIES WITH THE ZETA METER

C-2.1 Viewing the Emulsion

Colloids in the undiluted waste sample could not be seen due to the opacity of the emulsion. It was found that colloids for a 2:1 dilution could be seen without any great difficulty but with a 1:1 dilution they could not be seen. To utilize the zeta meter the waste had to be diluted.

C-2.2 Locating the Positioning Line

The right eyepiece contains an occular micrometer. Above the tracking lines is the OX - - OX or positioning line. This positioning line must be positioned over the optical front wall of the electrophoresis cell at mid-depth in order to properly track the colloid [25]. Now there are two requirements which must be met to establish the proper conditions for locating the positioning line:-

(a) the electrophoresis cell has to be positioned so that the front wall is in the viewing field of the objective lens.

(b) the illuminator must also be properly positioned and adjusted to have the light pass through the front half of the cell.

If the above manipulations have been properly carried out then the light bands and two yellow - white lines, which distinguish the front wall of the cell, become visible.

It is recommended that the individual acquaint himself with this technique before proceeding with the experimental work.

C-2.3 Gasification of the Electrodes

Two anodes are furnished with the Zeta meter:

(a) platinum - irridium - suggested for use with samples having a specific conductance of 1000 micromhos or less.

(b) a low carbon molybdenum cylinder suggested for samples with a SC exceeding 1000 micromhos.

In cell electrophoresis one volume of oxygen forms at the anode (+) for two volume of hydrogen at the cathode (-). When gasification occurs rapidly and becomes a critical factor, then the molybdenum anode should be used as the molybdenum combines instantly with the gaseous oxygen as it evolves.

It was found that with the waste samples and the effluent samples, the specific conductance was generally between 300 and 500 micromhos/cm. The platinum - irridium anode was used and gasification was found to occur in approximately 1.5 minutes. This necessitated filling the electrophoresis cell a few times to obtain 50 colloid mobility measurements.

The molybdenum anode should have been used to eliminate the number of times the cell had to be filled but it was decided that the procedure would not be changed for the balance of the runs.

C-3 Colloid Mobility - Zeta Potential Relationship

Electrophoretic mobility is expressed in terms of u/sec per E/cm. This represents the rate of travel of the colloid on the basis of a drop in potential of one volt per cm. through the cell.

Now Zeta potential is related to electrophoretic mobility by Equation C-2.

$$ZP = EM \times (X) \frac{V_t}{D_t} \quad - - - - \quad (C-2)$$

As "V_t" and "D_t" are properties of the fluid that can be determined, it is evident that the greatest difficulty here is in deciding on the value to assign to "X" in equation (C-2). Values for the "X" factor proposed by the different researchers are given in table C-2 [25,28].

Tables C-2

Values For "X" Factor

Authority	Value of "X"	Colloid Properties
Helmholtz-Smoluchowski	4π	- relatively large, non-conducting particles. - thickness of double layer << the radius of curvature of particle.
Debye - Hickel	6π	- relatively small, non-conducting particle. - thickness of double layer >> the radius of the particle. - spheres
	4π to 8π	- rod shaped particles, - value dependent upon orientation of particle with relation to direction of migration
Henry	$(4\pi)\ f\ (k_d\ a)$	- $f\ (K_d\ a)$ is Henry's rationalization of the Helmholtz-Smoluchowski and the Debye-Hickel equations. - applicable for large, small and intermediate particles.

Therefore the individual researcher must identify the particle as to shape, size, particle radius and other related characteristics.

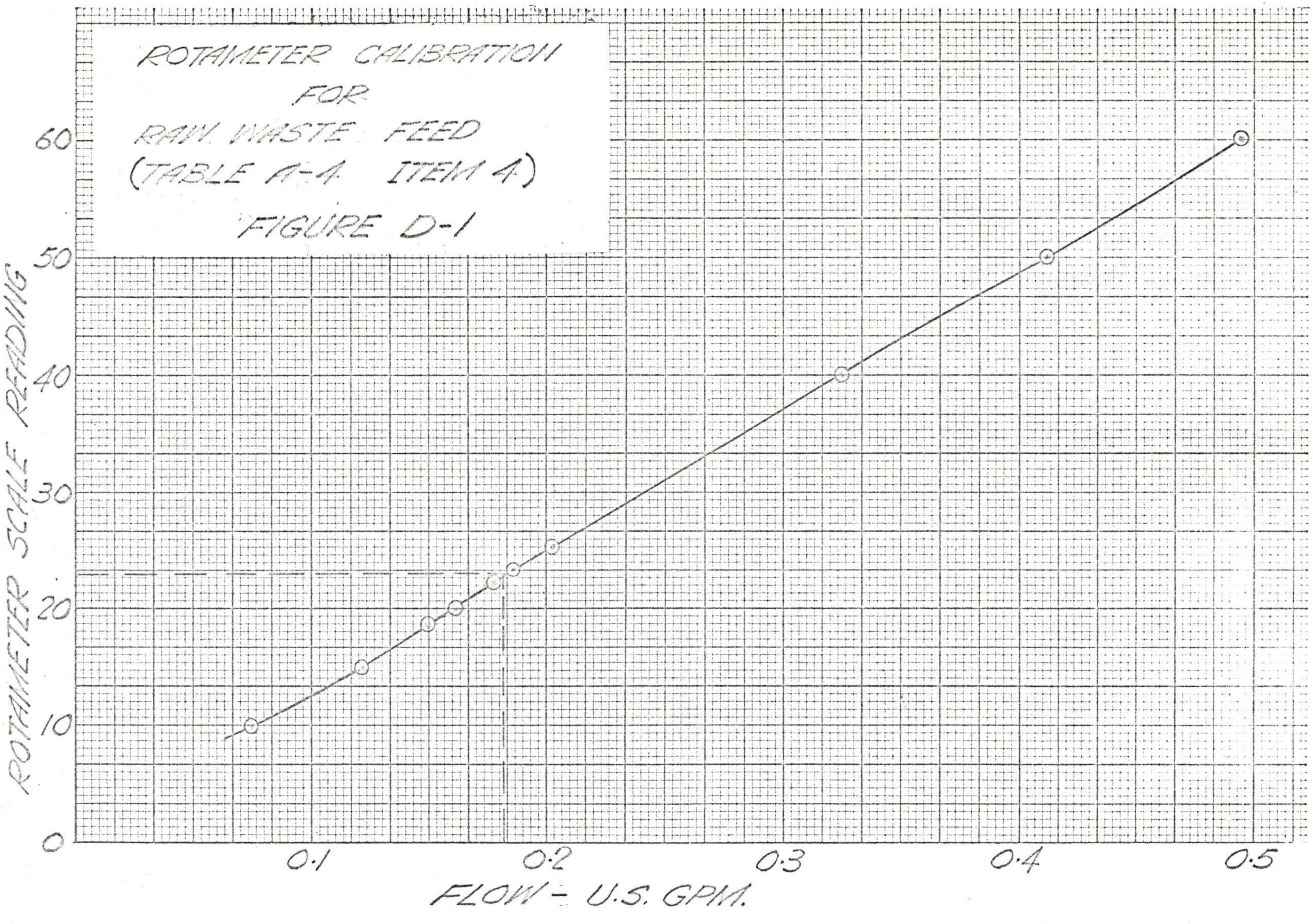
For this experimental work colloid mobility was recorded rather than Zeta Potential as this eliminated the need to characterize the particles in detail.

APPENDIX D: ROTAMETER CALIBRATIONS AND PRELIMINARY SCREENING

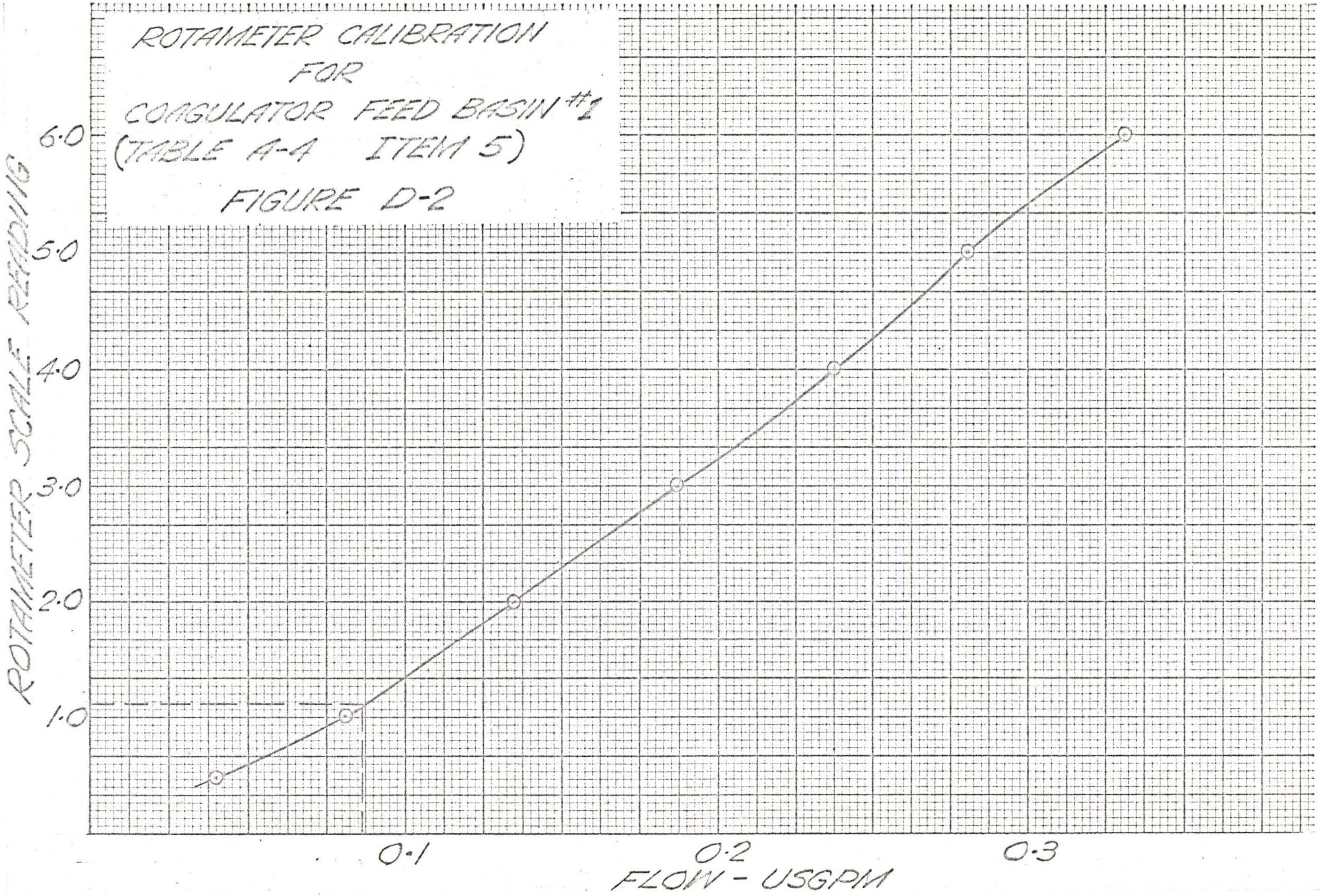
D-1 Rotameter Calibrations

The rotameters were calibrated by using volumetric flasks as the measurement basis and water as the calibrating fluid. As the concentration of oil was approximately 100 ppm in the diluted raw waste sample, the error introduced by using water was considered to be insignificant.

Calibration curves are shown in Figures D-1, D-2, D-3 and D-4.

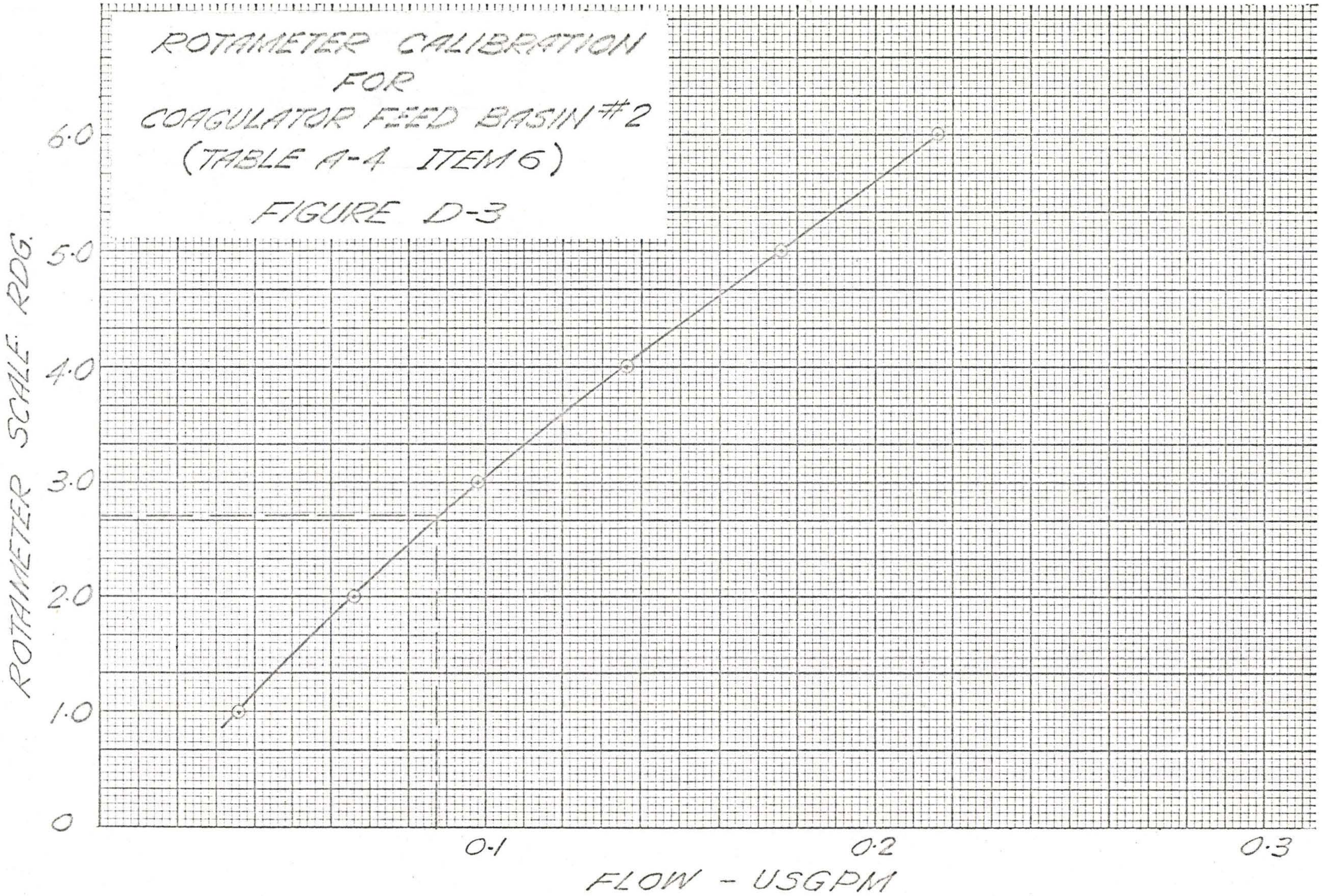


ROTAMETER CALIBRATION
FOR
RAW WASTE FEED
(TABLE A-4 ITEM 4)
FIGURE D-1



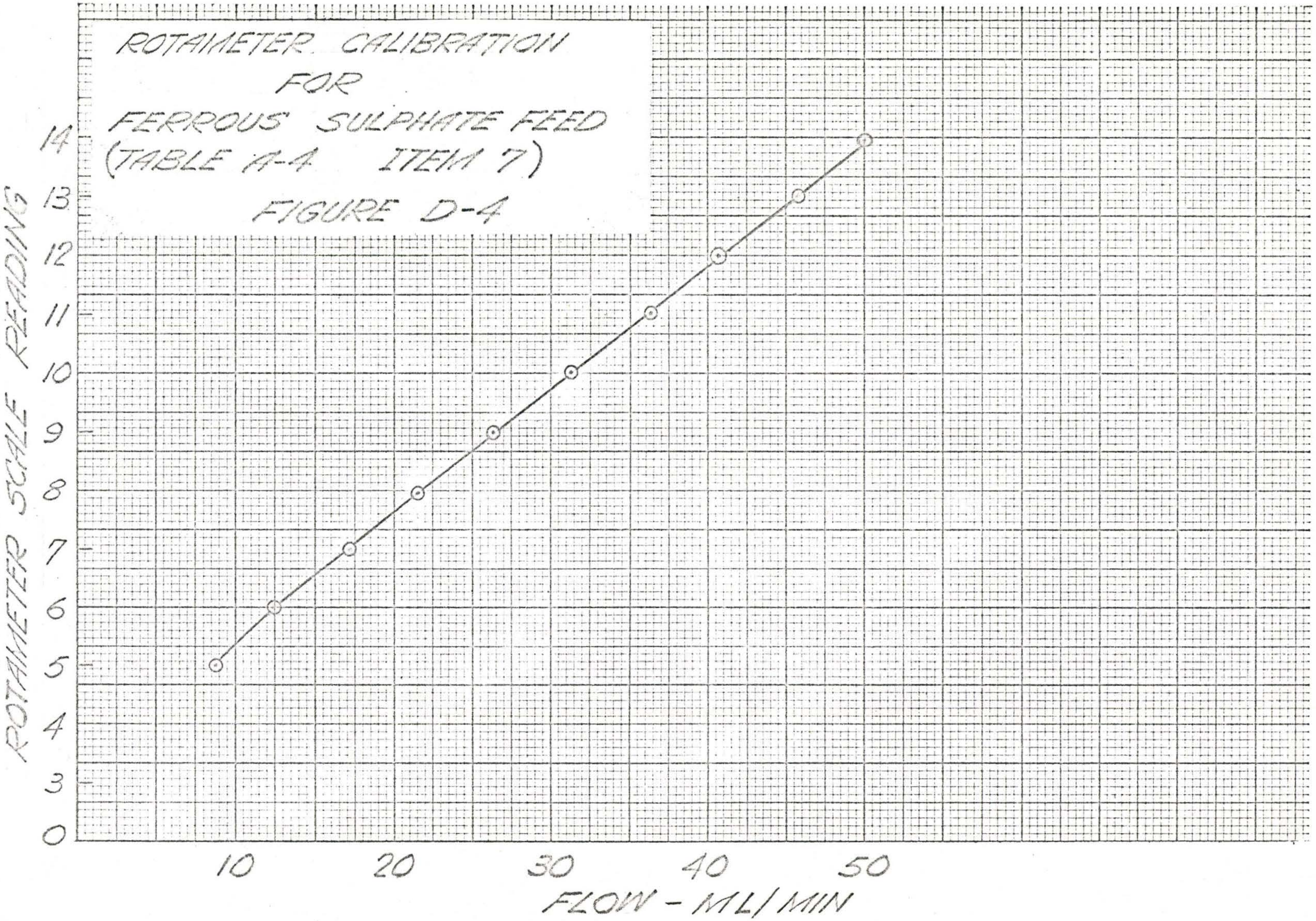
ROTAMETER CALIBRATION
FOR
COAGULATOR FEED BASIN #1
(TABLE A-4 ITEM 5)

FIGURE D-2



ROTAMETER CALIBRATION
FOR
COAGULATOR FEED BASIN #2
(TABLE A-4 ITEM 6)

FIGURE D-3



ROTAMETER CALIBRATION
FOR
FERROUS SULPHATE FEED
(TABLE A-4 ITEM 7)
FIGURE D-4

D-2 Dye Test

This was a qualitative test performed to determine the mixing characteristics and to learn something about the detention time in the coagulator and separator of the continuous unit.

Initially a concentrated methylene blue dye was pumped into the coagulator under the same conditions for flow rate and mixing rpm as required for the experimental runs. When the water in the coagulator and separator was colored, the dye was then flushed out with clear tap water.

Qualitatively it was observed that the coagulator was completely and uniformly mixed a deep blue after approximately 2.5 minutes had elapsed and appeared to approximate a CSTR.

First visible signs of dye appeared in the separator effluent approximately 4 minutes. The dye test was run prior to modifying the detention time for the separator. The plug flow detention time for the test was 10.5 minutes. The dye preferentially appeared to flow through the top quarter of the separator indicating a short circuiting and influent distribution problem.

It took seven plug flow detention times to flush all the dye out of the coagulator and separator with tap water.

A film of this test can be obtained from the McMaster Chemical Engineering Department.

APPENDIX E: CHEMICALS USED IN EXPERIMENTATION

This information is presented in tabular form in Table E-1.

TABLE E-1

CHEMICALS

Chemical		Supplier	Comments
Name	Formula		
Alchem 550-R	Not available	Alchem Ltd., Burlington, Ontario.	Cationic polyelectrolyte
Aluminum Sulphate	$Al_2(SO_4)_3.18H_2O$	Fisher Scientific 184 Railside Rd., Don Mills, Ont.	
Sodium Bicarbonate	N_aHCO_3	Fisher Scientific 184 Railside Rd., Don Mills, Ont.	
Calcium Carbonate	$CaCO_3$	Fisher Scientific 184 Railside Rd., Don Mills, Ont.	
Dispersonal C-1	Not available	Canadian Aniline & Extract Co. Ltd. 162 Ward Avenue, Hamilton, Ontario	Polyelectrolyte
n-Hexane	C_6H_{14}	Eastern Chemical Corp. Industrial Rd., Pequannce N.J. U.S.A. 07440	
Toluene (Toluol)	$C_6H_5-CH_3$	McArthur Chemical Co. Ltd., 4 Battlefield Drive Stoney Creek, Ont.	

TABLE E-1 continued

Chemical Name	Chemical Formula	Supplier	Comments
Carbon Tetrachloride	CCl_4	Mallinckrodt Chemical Works Ltd 3320 American Dr., Malton, Ontario.	
Calcium Hydroxide	$Ca(OH)_2$	Allied Chemical Canada Ltd., 100 North Queen St. Toronto 14, Ont.	Powder
Ferrous Sulphate	$FeSO_4 . 7H_2O$	Allied Chemical Canada Ltd., 100 North Queen St. Toronto 14, Ont.	
Sodium Chloride	NaCl	Fisher Scientific, 184 Railside Rd., Don Mills, Ont.	
Sulphuric Acid	H_2SO_4	British Drug Houses (Canada) Ltd., Barclay Avenue, Toronto, Ontario.	

NOTE: 1. All chemicals are reagent grade
2. The first 6 chemicals were used in preliminary experimentation.

APPENDIX F: ENERGY INPUT RELATIONSHIP BETWEEN COAGULATOR AND JAR TEST

Prior to commencing the experimental runs, the calculated energy input for the coagulator of the continuous unit was related to the measured energy input of the paddle for the jar test. This was done to establish a coagulating rpm. in the jar test based on the optimum design conditions for the coagulator. Using the rpm. versus torque curve, Figure F-1, and the calculated energy input for the coagulator the corresponding coagulating rpm. determined for the jar test was 53. Preliminary investigations for an iron floc indicated that the shear was too excessive at this rpm. The square jars that are used in the batch test do not have baffles to prevent bulk rotational flow and this would partially explain why the rpm. of the paddle must be correspondingly high to achieve the same energy input. Therefore the decision was made to keep the rpm. in both the batch and continuous units identical.

The data and calculations used as a basis for the preceding discussion are given in Table F-1 and Figure F-1 respectively.

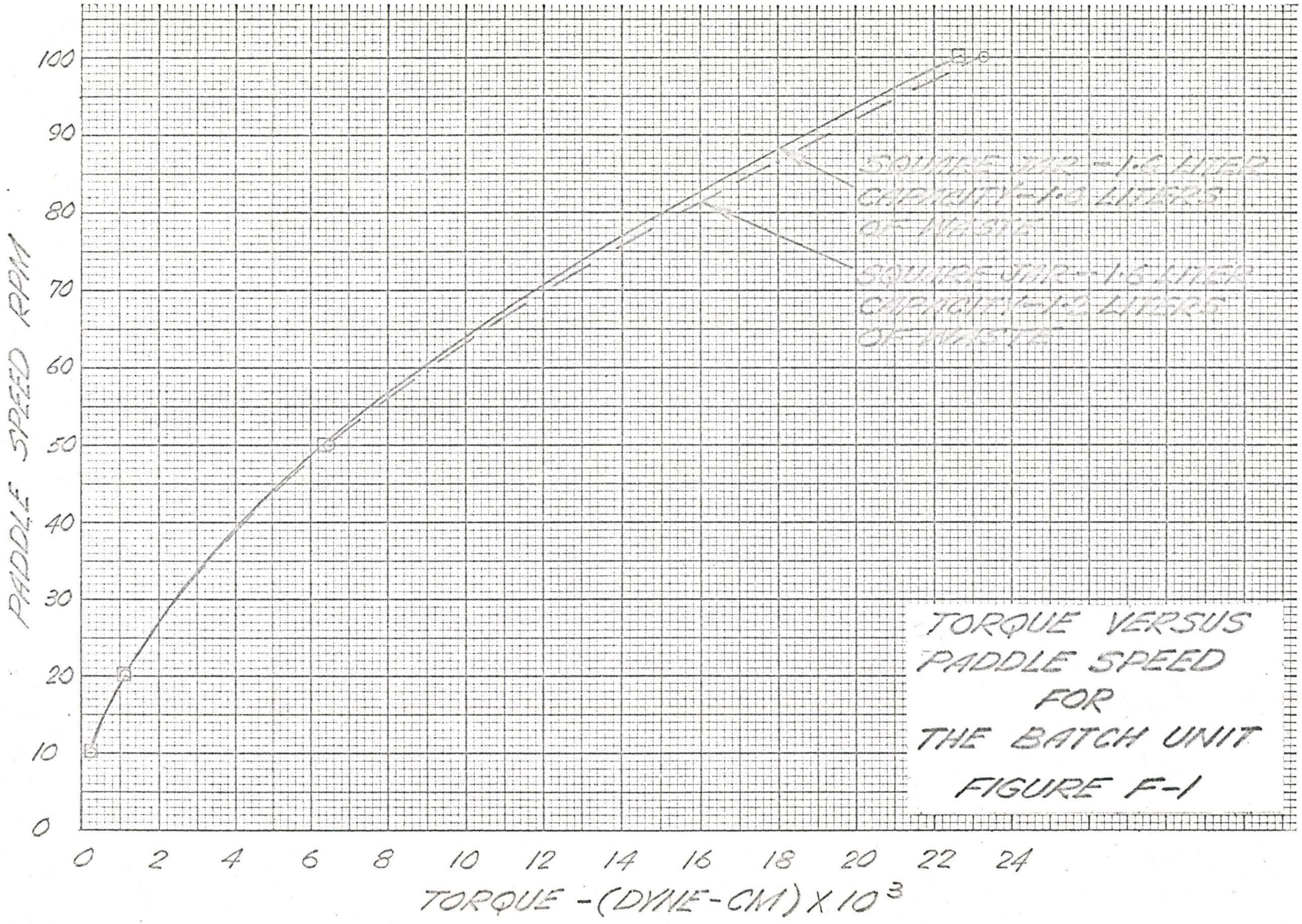
TABLE F-1

Torque Determinations - Jar Test

RPM	Viscometer Scale Reading (%)		Average Scale Reading (%)		Torque (dyne-cm.) **	
	1.0 liters *	1.2 liters *	1.0 liters *	1.2 liters *	1.0 liters*	1.2 liters*
10	0.4	0.4				
10	0.4	0.4	0.4	0.4	230	230
10	0.4	0.4				
20	2.0	2.1				
20	1.9	1.9	1.9	2.0	1092	1150
20	1.9	2.0				
50	10.9	11.5				
50	10.4	11.4	10.8	11.3	6210	6497
50	11.2	11.2				
100	39.4	40.8				
100	39.5	40.4	39.4	40.4	22653	23228
100	39.3	40.0				

* refers to the volume of waste used.

** 100% scale is equal to 57,496 dyne-cm. with linear relationship between 0 - 100% of scale.



TORQUE VERSUS PADDLE SPEED FOR THE BATCH UNIT

FIGURE F-1

DATA

The data in Table F-1 was obtained with the Brookfield Viscometer and the results have been plotted as shown in Figure F-1.

Calculations:

Energy input for the coagulator in the continuous unit based on a G_m for the basin of 44.6 sec^{-1} was 19.2 dyne - cm/cm^3 - sec. For the jar test energy input for the paddle is given by equation (F-1)

$$W_1 = (T_r) \ \pi \ (\text{rps}) \quad \times \ \frac{1}{V} \quad - - - - \qquad (\text{F-1})$$

By trial and error the torque-rpm. measurements given in Figure F-1 were related to the energy input of the continuous unit with equation (F-1). The corresponding rpm. in the jar test which gave an energy input of 19.2 dyne - cm/cm^3 - sec was 53.

APPENDIX G: STATISTICAL DESIGN

By employing statistical design of experiments the researcher is able to set experimental variables at levels that will yield a maximum of information with the minimum number of experimental runs.

The statistical design for this experiment involved two independent variables at five levels. A rotatable octagonal design with center points was used. The two independent variables were:- ferrous sulphate concentration and pH. The design is shown in Table G-1 for both coded and uncoded levels [21].

The dependent variables are colloid mobility and oil concentration.

TABLE G-1

STATISTICAL DESIGN FOR TWO INDEPENDENT VARIABLES AT FIVE LEVELS

RUN	CODED		UNCODED	
	X_1	X_2	X_1 = mg/l $FeSO_4$	X_2 = pH
1	0	0	150	9.4
6	0	0	150	9.4
2	-1	-1	79	7.7
4	1	-1	221	7.7
5	-1	1	79	11.1
3	1	1	221	11.1
12	0	0	150	9.4
8	0	0	150	9.4
7	$\sqrt{2}$	0	250	9.4
10	$-\sqrt{2}$	0	50	9.4
9	0	$\sqrt{2}$	150	11.8
11	0	$-\sqrt{2}$	150	7.0

APPENDIX H: EXPERIMENTAL DATA

The experimental data is presented in tables H-1 to H-7 inclusive. The relationship between temperature and colloid mobility is given by Figure H-1.

TABLE H-1

CHEMICAL CONCENTRATIONS USED IN EXPERIMENTAL RUNS

Run No.	$FeSO_4$ (mg/l)	$Ca(OH)_2$ mg/l	pH			Comments
			Raw Diluted Waste	Coagulation Batch	Coagulation Continuous	
11	150	54	7.5	6.95	6.9	
10	50	62.5	7.75	9.4	9.4	Additional 350 ml of 10 gm/l $Ca(OH)_2$ and 10 ml of 0.1 N H_2SO_4 added to holding tank to maintain pH at 9.4 in the coagulator of the continuous unit. Corresponding volume of waste unknown when additions made.
8	150	152	7.5	9.4	9.4	Additional 150 ml of 20 gm/l $Ca(OH)_2$ and 22 ml of 0.1 N H_2SO_4 added to holding tank to maintain pH at 9.4 in the coagulator of the continuous unit. Corresponding volume of waste unknown when additions made.
2	79	44	7.35	7.7	7.7	

TABLE H-1 (continued)

Run No.	$FeSO_4$ (mg/l)	$Ca(OH)_2$ mg/l	pH			Comments
			Raw Diluted Waste	Coagulation		
				Batch	Continuous	
3	221	290	7.4	11.1	11.1	
1	150	112	7.15	9.4	9.4	Additional 74 ml of 20 gm/l $Ca(OH)_2$ added to holding tank to maintian pH at 9.4 in the coagulator of the continuous unit. Corresponding volume of waste unknown when the additions are made.

TABLE H-2

OIL DETERMINATIONS

Sample * No.	Beaker Wt. (gms.)			Oil		Comments
	Oil + Residue + Tare	Tare	Oil + Residue	gms	(mg/l)	
11-RW$_1$	94.2891	94.2557	0.0334			All evaporated samples for the batch, continuous and the raw diluted waste contained considerable amounts of salt
11-RW$_2$	91.4585	91.4317	0.0268			
11-RW$_3$	98.6189	98.5952	0.237			
11-B$_1$	96.7033	96.6495	0.0538			
11-B$_2$	101.2391	101.1480	0.0911			
11-B$_3$	92.0731	92.0389	0.0392			
11-C$_1$	97.9628	97.8901	0.0727			
11-C$_2$	97.3995	97.2755	0.1241			
11-C$_3$	99.0600	99.0068	0.0532			
11-B$_{1.1}$	49.3330	49.3255	0.0005	0.0005	3	Oil extracted from the above evaporated samples with toluene and re-evaporated
11-B$_{2.1}$	46.1302	46.1212	0.0090	0.0090	45	
11-B$_{3.1}$	45.2195	45,2078	0.0117	0.0117	59	
11-C$_{1.1}$	49.0722	49.0647	0.0075	0.0075	38	Toluene was the solvent used in the extraction for all samples of Run 11. Results for Run 11 unreliable
11-C$_{2.1}$	49.3737	49.3674	0.0063	0.0063	32	
11-C$_{3.1}$	48.1340	48.1235	0.0105	0.0105	53	

The sample volume for each determination was 200 ml.

* Notations RW, B and C refer to the raw diluted waste, batch and continuous samples respectively.

Blanks run on the solvents showed no residue. NaCl was added to each 200 ml sample at concentration of 25 gms/l

TABLE H-2 (continued)

Sample * No.	Beaker Wt. (gms.)			Oil		Comments **
	Oil + Residue + Tare	Tare	Oil + Residue	gms	(mg/l)	
10-RW$_1$	1.4549	1.4343	0.0206	0.0206	103	
10-RW$_2$	1.4647	1.4309	0.0338	0.0338	169	Reject
10-RW$_3$	1.4540	1.4398	0.0142	0.0142	71	
10-B$_1$	1.4404	1.4328	0.0076	0.0076	38	Reject
10-B$_2$	1.4525	1.4404	0.0121	0.0121	61	Reject
10-B$_3$	1.4379	1.4343	0.0036	0.0036	18	
10-B$_4$	1.4385	1.4305	0.0080	0.0080	40	Reject
10-B$_5$	1.4370	1.4340	0.0030	0.0030	15	
10-B$_6$	1.4340	1.4329	0.0011	0.0011	6	
10-B$_7$	1.4439	1.4418	0.0021	0.0021	11	
10-B$_8$	1.4443	1.4392	0.0051	0.0051	26	
10-B$_9$	1.4429	1.4384	0.0045	0.0045	23	
10-C$_1$	1.4321	1.4297	0.0024	0.0024	12	
10-C$_2$	1.4338	1.4315	0.0023	0.0023	12	
10-C$_3$	1.4394	1.4317	0.0077	0.0077	38	Reject

The sample volume for each determination was 200 ml.

* Notations RW, B and C refer to the raw diluted waste, batch and continuous samples respectively.

Blanks run on the solvents showed no residue. NaCl was added to each 200 ml sample at concentration of 25 gms/l

** Samples are rejected because of stopcock grease interferences in the final weight

TABLE H-2 (continued)

Sample * No.	Beaker Wt. (gms.)			Oil		Comments
	Oil + Residue + Tare	Tare	Oil + Residue	gms	(mg/l)	
8-RW$_1$	1.4516	1.4299	0.0217	0.0217	109	
8-RW$_2$	1.4490	1.4339	0.0151	0.0151	76	
8-RW$_3$	1.4500	1.4325	0.0175	0.0175	88	
8-B$_1$	98.9677	98.9647	0.0030	0.0030	15	
8-B$_2$	99.0055	99.0021	0.0034	0.0034	17	
8-B$_3$	95.6370	95.6329	0.0041	0.0041	21	
8-C$_1$	99.9988	99.9952	0.0036	0.0036	18	
8-C$_2$	93.1748	93.1708	0.0040	0.0040	20	
8-C$_3$	100.5400	100.5364	0.0036	0.0036	18	
2-RW$_1$	94.7300	94.7175	0.0125	0.0125	63	
2-RW$_2$	100.0127	99.9986	0.0141	0.0141	71	
2-RW$_3$	98.9827	98.9677	0.0150	0.0150	75	
2-B$_1$	93.1749	93.1739	0.0010	0.0010	5	
2-B$_2$	94.7197	94.7183	0.0014	0.0014	7	
2-B$_3$	100.5428	100.5404	0.0024	0.0024	12	

The sample volume for each determination was 200 ml.

* Notations RW, B and C refer to the raw diluted waste, batch and continuous samples respectively.

Blanks run on the solvents showed no residue. NaCl was added to each 200 ml sample at concentration of 25 gms/l

TABLE H-2 (continued)

Sample *	Beaker Wt. (gms.)			Oil		Comments
No.	Oil + Residue + Tare	Tare	Oil + Residue	gms	(mg/l)	
2-C_1	91.4341	91.4320	0.0021	0.0021	11	
2-C_2	99.0081	99.0063	0.0018	0.0018	9	
2-C_3	95.6394	95.6371	0.0023	0.0023	12	
3-RW_1	94.7293	94.7155	0.0138	0.0138	69	
3-RW_2	91.4475	91.4287	0.0188	0.0188	94	
3-RW_3	98.6085	98.5929	0.0156	0.0156	78	
3-B_1	91.4331	91.4310	0.0021	0.0021	11	
3-B_2	95.6390	95.6366	0.0024	0.0024	12	
3-B_3	99.0070	99.0056	0.0014	0.0014	7	
3-C_1	100.5421	100.5394	0.0027	0.0027	14	
3-C_2	98.5971	98.5950	0.0021	0.0021	11	
3-C_3	93.1751	93.1729	0.0022	0.0022	11	
1-RW_1	100.0124	99.9990	0.0134	0.0134	67	
1-RW_2	98.9813	98.9681	0.0132	0.0132	66	
1-RW_3	98.6070	98.5951	0.0121	0.0121	61	

The sample volume for each determination was 200 ml.

* Notations RW, B and C refer to the raw diluted waste, batch and continuous samples respectively.

Blanks run on the solvents showed no residue. NaCl was added to each 200 ml sample at concentration of 25 gms/l

TABLE H-2 (continued)

Sample * No.	Beaker Wt. (gms.)			Oil		Comments
	Oil + Residue + Tare	Tare	Oil + Residue	gms	(mg/l)	
1-B_1	98.9693	98.9676	0.0017	0.0017	9	
1-B_2	100.5404	100.5390	0.0014	0.0014	7	
1-B_3	95.6381	95.6363	0.0018	0.0018	9	
1-C_1	94.7179	94.7163	0.0016	0.0016	8	
1-C_2	99.0074	99.0056	0.0018	0.0018	9	
1-C_3	99.9999	99.9986	0.0013	0.0013	7	

The sample volume for each determination was 200 ml.

* Notations RW, B and C refer to the raw diluted waste, batch and continuous samples respectively.

Blanks run on the solvents showed no residue. NaCl was added to each 200 ml sample at concentration of 25 gms/l

TABLE H-2 (continued)

Blank	Weight (gms.)			Comments
	Residue + Tare	Tare	Residue	
Tolune - 1	1.3850	1.3850	0	Sample - 200 ml. of
Tolune - 2	1.3927	1.3927	0	distilled water
Tolune - 3	1.3932	1.3932	0	Solvent - 100 ml.
CCl_4 - 1	1.3863	1.3863	0	Sample - 200 ml. of
CCl_4 - 2	1.3837	1.3837	0	distilled water
CCl_4 - 3	1.3808	1.3808	0	Solvent - 100 ml.

TABLE H-3

ZETA METER READINGS FOR COLLOID MOBILITY DETERMINATIONS

Date	Run No.	Sample No.	S.C. (micromhos/cm)	Volts	Micro-Amps	Direction * of Travel	Temperature °C	
							Start	Finish
12 Oct/68	11	11-RW	350	200	1150	C to A	24.5	25.5
		11-B	480	200	1550	C to A	24.5	25.5
		11-C	430	200	1400	A to C	25.0	25.5
16 Oct/68	10	10-RW	345	200	1200	C to A	22.5	24.0
		10-B	365	200	1200	C to A	26.2	26.5
		10-C	350	200	1150	C to A	25.5	26.0
18 Oct/68	8	8-RW	355	200	1200	C to A	26.25	26.75
		8-B	440	200	1425	C to A	26.0	27.0
		8-C	440	200	1425	A to C	25.75	27.0
19 Oct/68	2	2-RW	342	200	1150	C to A	25.2	26.0
		2-B	450	200	1500	C to A	25.0	25.8
		2-C	375	200	1220	C to A	24.0	25.5
20 Oct/68	3	3-RW	330	200	1100	C to A	23.5	24.0
		3-B	735	200	2450	A to C	23.5	25.0
		3-C	720	150	1750	A to C	23.5	25.0
21 Oct/68	1	1-RW	330	200	1100	C to A	23.8	24.5
		1-B	425	200	1450	C to A	23.8	24.5
		1-C	415	200	1350	C to A	23.5	24.5

* Notations C and A refer to Cathode and Anode respectively.
Objective lens = 8X
Tracking distance for each colloid = 30 microns
Distance between electrodes = 10 cm.

TABLE H-4

COLLOID MOBILITY - TRAVEL TIMES

Sample	Time	Seconds
11-RW	Cum.	0.8 1.5 2.2 3.2 3.8 4.5 5.3 6.1 7.1 8.1 9.1 9.9 10.8 11.6 12.5 13.2 13.9 14.8 15.7 16.6 17.6 18.3 19.4 20.2 21.0 21.7 22.5 23.4 24.5 25.4 26.7 27.9 28.8 30.0 30.8 31.6 32.5 33.4 34.6 35.6 36.6 37.5 38.8 39.5 40.3 41.1 41.9 42.7 43.7 44.6
	Ind.	0.8 0.7 0.7 1.0 0.6 0.7 0.8 0.8 0.9 1.1 1.0 0.8 0.9 0.8 0.9 0.7 0.7 0.9 0.9 0.9 1.0 0.7 0.9 0.8 0.8 0.7 0.8 0.9 1.1 0.9 1.3 1.2 0.9 1.2 0.8 0.8 0.9 0.9 1.2 1.0 1.0 0.9 1.3 0.7 0.8 0.8 0.8 0.8 1.0 0.9
11-B	Cum.	2.9 5.8 9.5 15.3* 17.3 21.0 23.9 26.5 29.2 31.5 34.4 37.8 40.4 42.7 45.8 48.7 51.1 53.6 56.1 58.5 60.9 62.9 64.6 67.3 68.9* 72.1 ; 2.0 4.1 6.4 8.1 10.8 13.3 16.8 19.2 21.1 23.8 26.2 29.4* 32.3 34.2 36.3 38.9 41.1 43.8 45.9 48.2 51.3 53.9 57.3 60.4 62.8 65.4 68.4
	Ind.	2.9 2.9 3.7 2.0 3.7 2.9 2.6 2.7 2.3 2.9 3.4 2.6 2.3 3.1 2.9 2.4 2.5 2.5 2.4 2.4 2.0 1.7 2.7 3.2 2.0 2.1 2.3 1.7 2.7 2.5 3.5 2.4 1.9 2.7 2.4 2.9 1.9 2.1 2.6 2.2 2.7 2.1 2.3 3.1 2.6 3.4 3.1 2.4 2.6 3.0
11-C	Cum.	2.0 6.1 8.5 10.1 13.1 15.3 17.4 19.9 23.0 26.5* 29.4 32.0* 33.9* 36.4 38.7 39.2* 41.9 44.1 46.7 48.8 51.7 54.5 56.9 59.4 62.5 65.7 67.8 70.5 72.9 75.1 77.4 79.5 81.6 84.2 86.5 89.3 91.9 94.3 96.0* 98.3 100.3 102.8 105.1 107.3 109.4 110.8* 112.6 115.3* 117.5 119.4 121.6 123.7 126.0 126.4* 128.2 130.1 132.0 134.0
	Ind.	2.0 4.1 2.4 1.6 3.0 2.2 2.1 2.5 3.1 2.9 2.5 2.3 2.7 2.2 2.6 2.1 2.9 2.8 2.4 2.5 3.1 3.2 2.1 2.7 2.4 2.2 2.3 2.1 2.1 2.6 2.3 2.8 2.6 2.4 2.7 2.0 2.5 2.3 2.2 2.1 1.8 2.2 1.9 2.2 2.1 2.3 1.8 1.9 1.9 2.0

TABLE H-4 (continued)

Sample	Time	Seconds
10-RW	Cum.	0.6 1.3 2.3 3.0 3.8 4.5 5.3 6.0 6.8 7.6 8.2 8.9 9.5 10.2 10.9 11.7 12.4 13.0 13.6 14.2* 14.8 15.7 16.2* 16.9 17.7 18.4 19.3 20.1 20.9 21.6 22.5 23.2 24.0 24.8 25.5 26.3 27.0 27.7 28.4 29.3 30.0 30.6* 31.4 32.1 32.7 33.4 34.3 35.0 35.6 36.3 37.0 37.7 38.3* 39.1
	Ind.	0.6 0.7 1.0 0.7 0.8 0.7 0.8 0.7 0.8 0.8 0.6 0.7 0.6 0.7 0.7 0.8 0.7 0.6 0.6 0.6 0.9 0.7 0.8 0.7 0.9 0.8 0.8 0.7 0.9 0.7 0.8 0.8 0.7 0.8 0.7 0.7 0.7 0.9 0.7 0.8 0.7 0.6 0.7 0.9 0.7 0.6 0.7 0.7 0.7 0.8
10-B	Cum.	0.6* 1.6 2.5 3.4 4.3 5.2 6.5 7.2 8.0 9.0 10.1 11.1 12.1 13.1 14.1 15.2 16.0 17.2 18.3 19.5 20.6 22.0 23.0 23.8 24.8 25.8 26.8 27.8 28.6 29.9 31.4 32.8 33.7 34.9 35.8 36.6 37.9 39.0 40.1 41.2 42.3 43.7 45.1 46.2 47.3 48.6 49.5 50.9 51.8 52.8 54.0
	Ind.	1.0 0.9 0.9 0.9 0.9 1.3 0.7 0.8 1.0 1.1 1.0 1.0 1.0 1.0 1.1 0.8 1.2 1.1 1.2 1.1 1.4 1.0 0.8 1.0 1.0 1.0 1.0 0.8 1.3 1.5 1.4 0.9 1.2 0.9 0.8 1.3 1.1 1.1 1.1 1.1 1.4 1.4 1.1 1.1 1.3 0.9 1.4 0.9 1.0 1.2
10-C	Cum.	1.6 2.9 4.2 5.7 7.1 8.7 10.1 11.0 12.3 13.6 15.1 16.9 18.6 19.9 21.7 23.8 25.6 27.6 29.3 30.9 32.0 33.3 34.4 36.0 38.1 39.3 41.3 42.4 44.6* 46.1 47.3 49.1 50.1 51.6 52.8 54.1 55.6 56.7* 58.0 59.1 60.2 61.3 62.4 63.5 64.7 65.9 67.1 68.2 69.4 70.4 71.4 72.5
	Ind.	1.6 1.3 1.3 1.5 1.4 1.6 1.4 0.9 1.3 1.3 1.5 1.8 1.7 1.3 1.8 2.1 1.8 2.0 1.7 1.6 1.1 1.3 1.1 1.6 2.1 1.2 2.0 1.1 1.5 1.2 1.8 1.0 1.5 1.2 1.3 1.5 1.3 1.1 1.1 1.1 1.1 1.1 1.2 1.2 1.2 1.1 1.2 1.0 1.0 1.1

TABLE H-4 (continued)

Sample	Time	Seconds
8-RW	Cum.	0.6 1.2 1.7* 2.2* 2.8 3.5 4.1 4.7 5.3 6.6* 6.7 7.2* 8.0 8.8 9.5 10.2 11.0 11.7 12.4 13.1 13.8 14.5 15.1 15.7 16.5 17.4 18.3 18.9 19.6 20.4 21.0 21.8 22.8 23.7 24.6 25.4 26.1 27.1 27.9 28.6 29.4 30.0 30.9 31.7 32.6 33.2 34.0 34.7 35.3 35.9 36.5* 37.0* 37.6 38.3 39.0 39.7
	Ind.	0.6 0.6 0.6 0.7 0.6 0.6 0.6 1.1 0.8 0.8 0.7 0.7 0.8 0.7 0.7 0.7 0.7 0.7 0.6 0.6 0.8 0.9 0.9 0.6 0.7 0.8 0.6 0.8 1.0 0.9 0.9 0.8 0.7 1.0 0.8 0.7 0.8 0.6 0.9 0.8 0.9 0.6 0.8 0.7 0.6 0.6 0.6 0.7 0.7 0.7
8-B	Cum.	2.0 6.3 8.7 12.5 15.7 19.2 22.6 25.7 28.4 30.5 36.6* 39.0 43.4 46.9 49.6 51.2 55.1 58.7 61.9 64.1 66.4 70.1 72.1* 76.3 79.5 80.5* 82.1* 86.0 88.8 91.8 93.9 : 1.8 4.4 6.8 14.1* 18.1 20.6 23.3 25.9 28.3 30.9 33.7 36.1 38.1* 40.1 42.2 44.2* 46.7 49.2 51.5 55.2 58.6 61.5 64.9 69.5 73.1 76.0 77.9
	Ind.	2.0 4.3 2.4 3.8 3.2 3.5 3.4 3.1 2.7 2.1 2.4 4.4 3.5 2.7 3.9 3.6 3.2 2.2 2.3 3.7 4.2 3.2 3.9 2.8 3.0 2.1 1.8 2.6 2.4 4.0 2.5 2.7 2.6 2.4 2.6 2.8 2.4 2.0 2.1 2.5 2.5 2.3 3.7 3.4 2.9 3.4 4.6 3.6 2.9 1.9
8-C	Cum.	2.4 6.4 8.2* 13.4 18.5 23.5 26.2 31.0 34.4 38.3 40.9 44.3 48.7 53.2 56.7 59.0 63.5* 65.1 67.6 70.1 73.7 75.9 77.6 80.6 83.7 86.1 89.2 91.5 93.2 95.3 98.8 100.4 103.8 106.2 108.3 111.3 114.1 116.8* 119.5 122.4 124.9 127.5 130.8 132.8 135.2 137.3 139.8 143.4 147.3 151.2 153.4 156.1 159.5
	Ind.	2.4 4.0 5.2 5.1 5.0 2.7 4.8 3.4 3.9 2.6 3.4 4.4 4.5 3.5 2.3 1.6 2.5 2.5 3.6 2.2 1.7 3.0 3.1 2.4 3.1 2.3 1.7 2.1 3.5 1.6 3.4 2.4 2.1 3.0 2.8 2.7 2.9 2.5 2.6 3.3 2.0 2.4 2.1 2.5 3.6 3.9 3.9 2.2 2.7 3.4

TABLE H-4 (continued)

Sample	Time	Seconds
2-RW	Cum.	0.8 1.7 2.4 3.0 3.8 4.8 5.6 6.2 7.2 8.0 8.7 9.5 10.4 11.1 12.0 12.7 13.7 14.3 15.2 15.9 16.8 17.7 18.4 19.1 19.9 20.7 21.5 22.3 23.2 24.2 25.2 26.0 26.8 27.5 28.3 29.1 30.1 31.1 32.0 33.0 33.9 34.8 35.5 36.7* 37.8 38.7 39.5 40.2 40.9 41.8 42.6
	Ind.	0.8 0.9 0.7 0.6 0.8 1.0 0.8 0.6 1.0 0.8 0.7 0.8 0.9 0.7 0.9 0.7 1.0 0.6 0.9 0.7 0.9 0.9 0.7 0.7 0.8 0.8 0.8 0.8 0.9 1.0 1.0 0.8 0.8 0.7 0.8 0.8 1.0 1.0 0.9 1.0 0.9 0.9 0.7 1.1 0.9 0.8 0.7 0.7 0.9 0.8
2-B	Cum.	1.8 3.5 5.9 7.9 10.4 13.3 16.5 18.4 20.4 22.6 25.2 28.9 30.8 33.3 35.5 37.5 39.8 41.3 43.7 46.1 47.8 49.3 51.3 52.7 54.5 56.2 58.3 60.3 63.5 66.8 69.5 71.0 72.6 75.4 77.8 81.3 82.9 85.5 88.6 90.6 93.1 95.5 98.2 99.6 102.1 104.2 106.7 109.8 111.7 113.4
	Ind.	1.8 1.7 2.4 2.0 2.5 2.9 3.2 1.9 2.0 2.2 2.6 3.7 1.9 2.5 2.2 2.0 2.3 1.5 2.4 2.4 1.7 1.5 2.0 1.4 1.8 1.7 2.1 2.0 3.2 3.3 2.7 1.5 1.6 2.8 2.4 3.5 1.6 2.6 3.1 2.0 2.5 2.4 2.7 1.4 2.5 2.1 2.5 3.1 1.9 1.7
2-C	Cum.	2.3 3.5 4.9 6.5 9.4 11.7 14.2 16.6 18.8 21.2 23.6 26.3 28.7 31.3 34.9 38.0 41.5 44.5 47.0 50.4 53.0 55.4 60.2 63.8 67.2 70.3 73.6 76.9 80.0 84.5 87.8 90.7 93.3 96.3 99.8 102.7 106.3 108.4 111.0 115.0 118.5 121.1 124.7 127.2 131.9 134.9 138.5 142.6 146.3 148.6
	Ind.	2.3 1.2 1.4 1.6 2.9 2.3 2.5 2.4 2.2 2.4 2.4 2.7 2.4 2.6 3.6 3.1 3.5 3.0 2.5 3.4 2.6 2.4 4.8 3.6 3.4 3.1 3.3 3.3 3.1 4.5 3.3 2.9 2.6 3.0 3.5 2.9 3.6 2.1 2.6 4.0 3.5 2.6 3.6 2.5 4.7 3.0 3.6 4.1 3.7 2.3

TABLE H-4 (continued)

Sample	Time	Seconds
3-RW	Cum.	0.7 1.5 2.4 3.1 4.0 4.9 5.8 6.5 7.4 8.0 8.7 9.8 10.7 11.4 12.3 13.1 14.0 14.9 15.8 16.7 17.7 18.4 19.3 20.4 21.3 22.1 23.0 24.3 25.1 26.2 27.5 28.7 29.6 30.3 31.3 32.4 33.5 34.7 35.7 36.7 37.6 38.7 39.7 40.8 42.1 42.9 43.8 44.7 45.5 46.2
	Ind.	0.7 0.8 0.9 0.7 0.9 0.9 0.9 0.7 0.9 0.6 0.7 1.1 0.9 0.7 0.9 0.8 0.9 0.9 0.9 0.9 1.0 0.7 0.9 1.1 0.9 0.8 0.9 1.3 0.8 0.9 1.3 1.2 0.9 0.7 1.0 1.1 1.1 1.2 1.0 1.0 0.9 1.1 1.0 1.1 1.3 0.8 0.9 0.9 0.8 0.7
3-B	Cum.	1.5 2.9 4.6 6.4 8.0 10.2 12.2 14.0 15.4 17.8 19.2 20.8 22.7 24.9 25.9* 27.8 29.5 31.3 33.8 35.6 36.7 37.8 39.5 41.1 42.9 44.2 46.0 47.7 49.5 51.3 53.6 56.0 58.6 60.3 62.5 64.6 66.9 68.5 70.4 71.7 73.1 74.7 76.3 77.7 79.3 81.9 84.8 87.1 88.8 90.4 92.1
	Ind.	1.5 1.4 1.7 1.8 1.6 2.2 2.0 1.8 1.4 2.4 1.4 1.6 1.9 2.2 1.9 1.7 1.8 2.5 1.8 1.1 1.1 1.7 1.6 1.8 1.3 1.8 1.7 1.8 1.8 2.3 2.4 2.6 1.7 2.2 2.1 2.3 1.6 1.9 1.3 1.4 1.6 1.6 1.4 1.6 2.6 2.9 2.3 1.7 1.6 1.7
3-C	Cum.	1.6 4.4 6.9 10.0* 12.8 15.1 16.9 19.3 21.3 23.3 25.6 27.6 29.1 31.1 32.8 34.8 36.9 38.9 40.6 42.4 43.9 45.6 47.2 48.6 50.4 51.9 53.2 55.0 56.5 58.7 60.4 62.1 64.2 66.2 67.8 69.3 71.0 72.8 74.4 76.3 78.4 80.0 81.5 83.0 84.3 86.5 88.1 89.3 90.5 92.1 94.2
	Ind.	1.6 2.8 2.5 2.8 2.3 1.8 2.4 2.0 2.0 2.3 2.0 1.5 2.0 1.7 2.0 2.1 2.0 1.7 1.8 1.5 1.7 1.6 1.4 1.8 1.5 1.3 1.8 1.5 2.2 1.7 1.7 2.1 2.0 1.6 1.5 1.7 1.8 1.6 1.9 2.1 1.6 1.5 1.5 1.3 2.2 1.6 1.2 1.2 1.6 2.1

TABLE H-4 (continued)

Sample	Time	Seconds
1-RW	Cum.	0.8 1.5 2.3 3.1 3.9 4.9 5.6 6.4 7.2 7.8 8.7 9.5 10.2 11.1 12.2 13.0 13.8 14.7 15.6 16.6 17.4 18.2 19.4 20.3 21.2 22.3 23.1 24.1 25.1 25.8 26.6 27.3 28.3 29.4 30.5 31.4 32.2 33.0 33.8 34.6 35.4 36.4 37.7* 38.4 39.2 40.0 41.1 42.0 42.9 44.0 44.8
	Ind.	0.8 0.7 0.8 0.8 0.8 1.0 0.7 0.8 0.8 0.6 0.9 0.8 0.7 0.9 1.1 0.8 0.8 0.9 0.9 1.0 0.8 0.8 0.8 0.9 0.9 1.1 0.8 1.0 1.0 0.7 0.8 0.7 1.0 1.1 1.1 0.9 0.8 0.8 0.8 0.8 0.8 1.0 0.7 0.8 0.8 0.9 0.9 0.9 1.1 0.8
1-B	Cum.	1.4 3.9 6.7 9.9 11.9 15.6 18.7 20.8 23.8 26.6 29.5 33.6 37.1 42.2 45.2 46.9 48.8 51.4 54.4 56.4 61.4 64.7 68.5 71.0 73.4 75.1 77.8 80.0 82.7 85.0 86.4 88.2 91.2 93.2 95.3 97.2 100.6 103.0 105.6 109.1 111.1 113.2 116.8 120.0 121.8 125.1 128.8 131.5 134.1 137.0
	Ind.	1.4 2.5 2.8 3.2 2.0 3.7 3.1 2.1 3.0 2.8 2.9 4.1 3.5 5.1 3.0 1.7 1.9 2.6 3.0 2.0 5.0 3.3 3.8 2.5 2.4 1.7 2.7 2.1 2.7 2.3 1.4 3.2 3.0 2.0 2.1 1.9 3.4 2.4 2.6 3.5 2.0 2.1 3.6 3.2 1.8 3.3 3.7 2.7 2.6 2.9
1-C	Cum.	2.9* 6.2 9.4 13.5 20.9 26.8 32.8 36.0 42.2 46.3 52.4 55.8 60.0 63.7 66.1 68.3 71.0 75.7 79.9 85.4 90.8 94.2 99.9 102.8 105.6 108.3 112.8 116.1 119.9 123.7 126.9 131.2 135.9 139.4 144.1 147.7 150.9 155.1 158.1 162.6 167.2 170.6 175.4 179.1 182.7 185.1 188.8 190.8 194.5 198.3 201.8 204.6
	Ind.	3.3 3.2 4.1 5.9 6.0 3.2 6.2 4.1 6.1 2.4 4.2 3.7 2.4 2.2 2.7 4.7 4.2 5.5 5.4 3.4 5.7 2.9 2.8 2.7 4.5 3.3 3.8 3.8 3.2 4.3 4.7 3.5 4.7 3.6 3.2 4.2 3.0 4.5 4.6 3.4 4.8 3.7 3.6 2.4 3.7 2.0 3.7 3.8 3.5 2.8

TABLE H-4 (continued)

NOTE:

Cum. means cumulative travel time
Ind. means individual travel time
* Determination rejected because of poor tracking
42.7 underlined figure means that floc was tracked

$$\text{Colloid Mobility} = \text{EM} = \frac{\text{Tracking distance (microns)}}{\text{Colloid Tracking Time (Secs.)}} \times \frac{\text{Distance between electrodes (cm.)}}{\text{volts across electrodes}}$$

TABLE H-5

SAMPLE TABULATION FOR DETERMINING THE CUMULATIVE DISTRIBUTION OF COLLOID MOBILITIES

Run 11-C

Individual Travel Time (secs.)	Ranked Time (secs.)	Frequency	Cumulative Frequency	Cumulative % Frequency
2.0	1.6	1	1	1
4.1	1.8			
2.4	1.8	2	3	4
1.6	1.9			
3.0	1.9			
2.2	1.9	3	6	9
2.1	2.0			
2.5	2.0			
3.1	2.0	3	9	15
2.9	2.1			
2.5	2.1			
2.3	2.1			
2.7	2.1			
2.2	2.1			
2.6	2.1			
2.1	2.1	7	16	25
2.9	2.2			
2.8	2.2			
2.4	2.2			
2.5	2.2			
3.1	2.2			
3.2	2.2	6	22	38
2.1	2.3			
2.7	2.3			
2.4	2.3			

TABLE H-5 (continued)

Individual Travel Time (secs.)	Ranked Time (secs.)	Frequency	Cumulative Frequency	Cumulative % Frequency
2.2	2.3			
2.3	2.3	5	27	49
2.1	2.4			
2.1	2.4			
2.6	2.4			
2.3	2.4	4	31	58
2.8	2.5			
2.6	2.5			
2.4	2.5			
2.7	2.5	4	35	66
2.0	2.6			
2.5	2.6			
2.3	2.6	3	38	73
2.2	2.7			
2.1	2.7			
1.8	2.7	3	41	79
2.2	2.8			
1.9	2.8	2	43	84
2.2	2.9			
2.1	2.9	2	45	88
2.3	3.0	1	46	91
1.8	3.1			
1.9	3.1	2	48	94
1.9	3.2	1	49	97
2.0	4.1	1	50	99

TABLE H-6

ALKALINITY DETERMINATIONS

Run.	Sample No. *	Titrant (ml) **			
		Reading at end	Reading at start	Difference	Alkalinity (mg/l)
11	11-RW$_1$	21.60	17.10	4.50	90
	11-RW$_2$	25.85	21.60	4.25	85
	11-RW$_3$	30.45	25.85	4.60	92
	11-B$_1$	5.50	2.85	2.65	53
	11-B$_2$	7.75	5.50	2.25	45
	11-B$_3$	10.85	7.75	3.10	62
	11-C$_1$	12.55	10.85	1.70	34
	11-C$_2$	14.35	12.65	1.70	34
	11-C$_3$	16.45	14.35	2.10	42
10	10-RW$_1$	31.60	27.20	4.40	88
	10-RW$_2$	36.05	31.70	4.35	87
	10-RW$_3$	40.50	36.05	4.45	89
	10-B$_1$	16.90	14.15	2.75	55
	10-B$_2$	10.90	8.20	2.70	54
	10-B$_3$	14.15	10.90	3.25	65

* Notations RW, B and C refer to the raw diluted waste, batch and continuous samples respectively.

** Titration endpoint - pH of 4.5
The sample volume for each determination was 50 ml.

TABLE H-6 (continued)

Run	Sample No. *	Titrant (ml) ** Reading at end	Reading at start	Difference	Alkalinity (mg/l)
10	10-C_1	19.0	16.9	2.1	42
	10-C_2	21.0	19.0	2.0	40
	10-C_3	23.0	21.0	2.0	40
8	8-RW_1	18.5	13.9	4.6	92
	8-RW_2	22.8	18.5	4.3	86
	8-RW_3	27.2	22.8	4.4	88
	8-B_1	6.10	4.85	1.25	25
	8-B_2	7.40	6.15	1.25	25
	8-B_3	8.80	7.40	1.4	28
	8-C_1	10.05	8.95	1.10	22
	8-C_2	12.70	11.55	1.15	23
	8-C_3	13.90	12.70	1.20	24
2	2-RW_1	5.5	0.9	4.6	92
	2-RW_2	9.7	5.5	4.2	84
	2-RW_3	14.0	9.7	4.3	86

* Notations RW, B and C refer to the raw diluted waste, batch and continuous samples respectively.

** Titration endpoint - pH of 4.5
The sample volume for each determination was 50 ml.

TABLE H-6 (continued)

Run	Sample No. *	Titrant (ml) **			Alkalinity (mg/l)
		Reading at end	Reading at start	Difference	
2	2-B$_1$	18.55	14.0	4.55	91
	2-B$_2$	23.10	18.55	4.55	91
	2-B$_3$	27.85	23.10	4.75	95
	2-C$_1$	30.65	28.1	2.55	51
	2-C$_2$	33.15	30.65	2.50	50
	2-C$_3$	35.75	33.15	2.60	52
3	3-RW$_1$	7.45	2.80	4.65	93
	3-RW$_2$	11.90	7.45	4.45	89
	3-RW$_3$	16.40	11.90	4.50	90
	3-B$_1$	37.30	32.30	5.0	100
	3-B$_2$	27.25	22.50	4.75	95
	3-B$_3$	32.30	27.50	4.60	92
	3-C$_1$	42.40	37.30	5.1	102
	3-C$_2$	47.40	42.40	5.0	100
	3-C$_3$	46.25	41.15	5.1	102

* Notations RW, B and C refer to the raw diluted waste, batch and continuous samples respectively.

** Titration endpoint - pH of 4.5

The sample volume for each determination was 50 ml.

TABLE H-6 (continued)

Run	Sample No. *	Titrant (ml) **			Alkalinity (mg/l)
		Reading at end	Reading at start	Difference	
1	1-RW_1	9.60	5.65	3.95	79
	1-RW_2	13.85	9.60	4.25	85
	1-RW_3	23.15	18.80	4.35	87
	1-B_1	25.80	23.15	2.75	55
	1-B_2	28.30	25.80	2.50	50
	1-B_3	30.90	28.30	2.60	52
	1-C_1	32.80	30.95	2.05	41
	1-C_2	34.80	32.80	2.0	40
	1-C_3	36.90	34.80	2.10	42

* Notations RW, B and C refer to the raw diluted waste, batch and continuous samples respectively.

** Titration endpoint - pH of 4.5

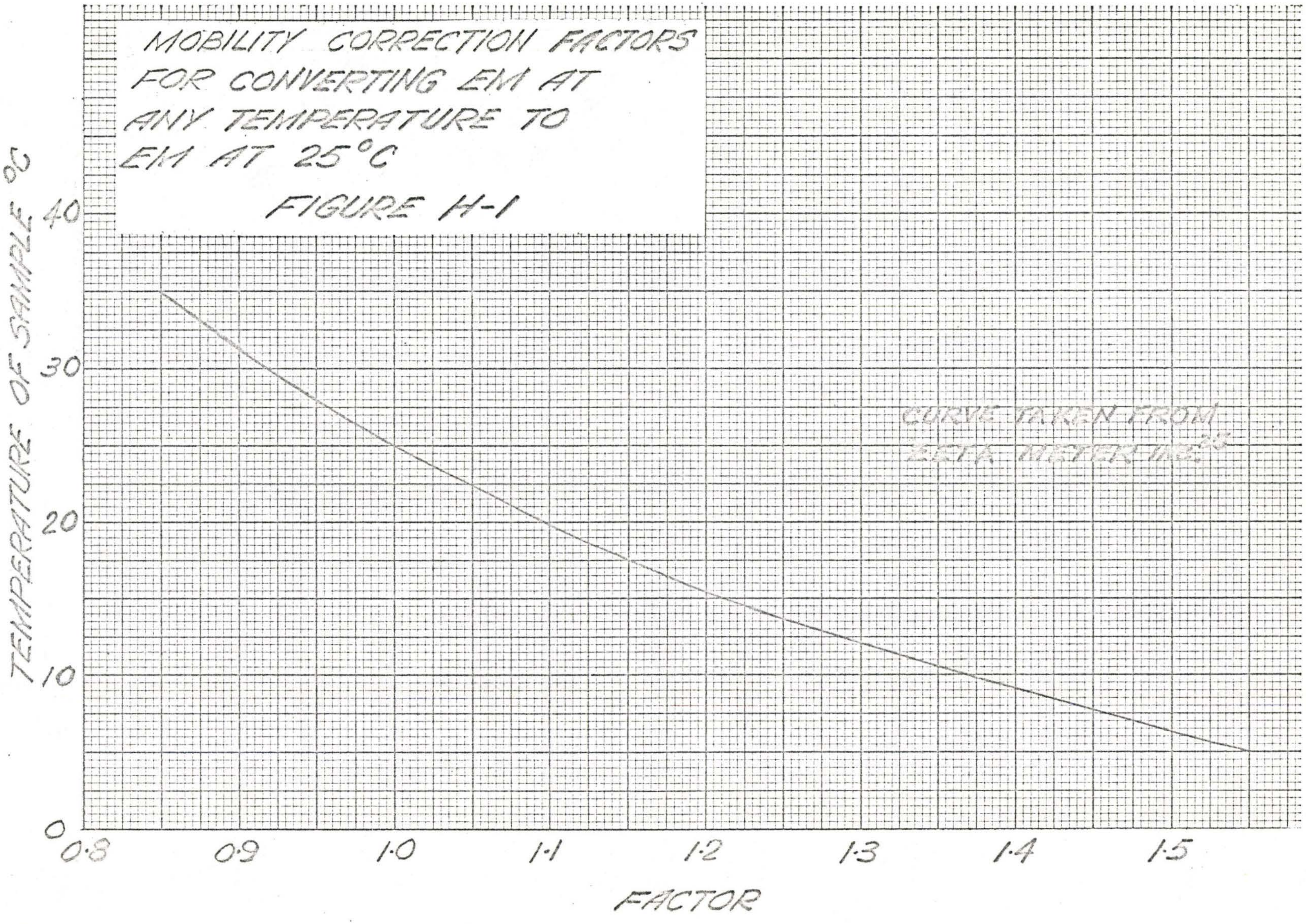
The sample volume for each determination was 50 ml.

TABLE H-7

MICROORGANISM PLATE COUNT

Date	Dilution (Waste/Agar Medium)	Plate Count * No. of Colonies	Microorganisms/ml.
18 Oct/68	$1:10^5$	few	
	$1:10^4$	135 145	1.4×10^6
	$1:10^3$	1125	
	Control	none	
22 Oct/68	$1:10^4$	0 2	
	$1:10^5$	few	
	$1:10^3$	130 70	1.0×10^5
	Control	1	

48 hour - Incubation Period
20°C - Incubation Temperature
* Duplicate plates were prepared.
Plate counts performed by Mrs. A. Latoszek, Environmental Engineering - McMaster University.



MOBILITY CORRECTION FACTORS FOR CONVERTING EM AT ANY TEMPERATURE TO EM AT 25°C

FIGURE H-1

CURVE TAKEN FROM ZETA METER INC.[26]