EFFECTS OF SECONDARY MIXING IN HELICALLY-COILED TUBULAR REACTORS

By

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EFFECTS OF SECONDARY MIXING IN HELICALLY-COILED TUBULAR REACTORS

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PREFACE

The primary purpose of this study was to investigate the effects of secondary mixing in helically-coiled tubular reactors in the laminar flow regime. Crystal violet dye and sodium hydroxide were used as the reactants in the study.

Secondly, it was intended to design a helical flow tubular reactor for undergraduate laboratory. The operating conditions were based on a 1:1 flow rate ratio of sodium hydroxide and dye solutions and a design operating time of 2-3 hours.

I am especially grateful to my thesis advisor, Dr. Arland H. Johannes, for his valuable and friendly guidance, patience, and cooperation during this study. I am thankful to the other committee members, Dr. Marvin M. Johnson, Dr. Gary L. Foutch and Mr. William Payne for their suggestions and cooperation in the course of this study.

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Finally, I wish to dedicate this achievement to my brother, Dr. John O. Egekeze, for his inspiration and confidence in me, and my entire family for their financial, moral and spiritual support.

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NOMENCLATURE

A_{in} = inlet absorbance

A_{out} = outlet absorbance

Ao = initial absorbance, t=0

A = absorbance at any time

C_A = dye concentration at any time

C_B = sodium hydroxide concentration

C_{Ao} = initial dye concentrations

C_{Bo} = initial sodium hydroxide concentration

D = tube diameter

E = activation energy

E(X) = exponential integral

N_{Re} = Reynolds number

V = reactor volume

vo = volumetric flow rate, ft^3/min

t = time

τ = residence time

K' = pseudo first order rate constant, min^{-1}

K = rate constant, L/mol min

X_A = conversion

ΔP = pressure drop

T_{in} = inlet transmittance

T_{out} = outlet transmittance

L = reactor length

i.d = inside diameter

s.s = steady state time

Q = volumetric flow rate, ml/min

ρ = density

μ = viscosity

T = temperature

r_A = reaction rate

$\bar{v}$,u, = mean velocity

ΔPs = calculated pressure drop for straight tube

ΔPc = calculated pressure drop for helix

Sc = Schmidt number

CHAPTER I

INTRODUCTION

Secondary flow is said to be perpendicular to the main direction of flow and occurs whenever the main flow is caused to change its direction due to constraining walls. In helically-coiled tubes, secondary flow is induced by the action of centrifugal forces.

Studies have shown that this effect inhibits axial dispersion, increases heat transfer coefficients and heat transfer area, and also increases pressure drop in helical tubes as opposed to straight tubes.

In terms of chemical conversion in reactors, there is no clear-cut understanding of the effects of this secondary flow in helical tubes when compared to plug flow reactors and laminar flow reactors.

It is known in the literature that plug flow reactors are expected to have maximum conversion. In helical reactors, it is speculated that there will be secondary mixing as a result of this flow pattern. Hence, it is expected to enhance the performance of the reactor in terms of conversion to approach plug flow.

In recent related work, Asfour (20) predicted that the performance of helically-coiled tubular reactors falls in between plug flow and laminar flow reactors approaching plug flow at very high or very low holding times.

In this study, design changes were made to improve Asfour's experimental set-up. A newtonian fluid in the laminar flow region was considered and the hydrolysis of crystal-violet dye with sodium hydroxide was the reaction employed.

This investigation also considered different orientations of the reactor, different reactor sizes, radius of curvature, coil diameter and pitch.

CHAPTER II

LITERATURE SURVEY

Extensive studies have been done on the flow of fluids through tubular reactors in various areas of interest. A review of the literature will be made in the following subjects:

(1) Diffusion and reaction
(2) Axial dispersion and residence time distribution
(3) Heat transfer and pressure drops
(4) Experimental work and kinetics

Diffusion and Reaction

Cleland and Wilhelm (1) conducted one of the earliest studies on diffusion and reaction in a viscous-flow tubular reactor. They investigated the effect on point and integral conversion of chemical reactions, from radial diffusion and reaction time distribution in viscous-flow tubular reactors. The results were given for a liquid-phase first-order chemical reaction, under isothermal conditions, in a tubular reactor in the laminar-flow region for a range of dimensionless rate and time variables.

The liquid-phase hydrolysis of acetic anhydride was chosen as the specific chemical reaction for this study. The dimensionless parameters defined were,

$$\lambda = \frac{K_z}{V_o},\ C = \frac{c}{c_o},\ \alpha = \frac{D}{KR^2},\ U = \frac{r}{R}$$

where, λ = time of contact

α = diffusion parameter, dimensionless

k = reaction-velocity constant

r = radial position in reactor measured from reactor axis

z = distance coordinate measured from reactor inlet = reactor length

V_o = velocity of central streamline within reactor

C = point concentration of reactant, dimensionless

co = concentration of reactant at reactor inlet, M/L^3

c = point concentration of reactant, M/L^3 [illegible]

D = molecular diffusivity = 10^{-5} sq cm/s → [illegible]

R = reactor radius

U = radial coordinate measured from reactor axis

k_z = reaction velocity constant in axial direction

Based on these parameters, they developed models that can be used to predict average concentrations of reactants for both laminar flow (no diffusion) and plug flow (uniform radial concentrations) reactors and also obtained values for first-order reactions that permit quantitative evaluation of the contributions of diffusion and flow profile on mean conversion for any set of circumstances.

Levenspiel and Smith (2) developed a diffusion-type model for the longitudinal mixing of fluids in flow. They characterized this mixing in flow by a single parameter D, the "longitudinal dispersion coefficient". In developing the model they made the assumption that the longitudinal dispersion coefficient D is independent of position and gives a measure of the degree of mixing occurring during flow. As a result, this model was expected to be useful in investigating conversions in homogeneous flow reactors.

In a related work to Levenspiel (2), Aunicky (3), recognized the fact that a coefficient of longitudinal dispersion K, is used to characterize the longitudinal mixing of fluids flowing through straight pipes. He further investigated the case of piping equipped with bends and suggested an extension of the value of K by an expression Δk, which describes the more intense longitudinal mixing that occurs in bend areas. The value of Δk is determined by the secondary flow at these bends and it depends on the Reynolds number (N_{Re}), bend length α, and the proportional bend curvature (R/D) ie,

$$\Delta k = f\ (N_{Re}, R/D, \alpha)$$

While studying the effect of incomplete mixing on homogeneous reactions, Danckwerts (4) investigated the concept of mixing on the molecular scale between elements of fluid that have been in the system for different lengths of time. He proposed a design method that combines a knowledge of the kinetics of the reaction and the hydrodynamic and diffusional behavior of the reactant stream, when calculating the output of different types of reactors under different conditions. He indicated that segregation, which is the existence of different concentrations of a particular reactant in the reactor, leads to undesirable side-reactions and further suggested a measurement for the degree of segregation as,

$$J = \frac{\overline{(\tau - t)^2}}{\bar{\theta}^2}$$

The quantity $\overline{(\tau - t)^2}$ is the mean square deviation of the ages of all points in the system from the mean age, τ, and the value of θ^{-2} can be evaluated if the residence-time distribution for the system is known. This work, however, did not present a complete solution to reactions involving complex kinetics in continuous-flow reactors.

Zwietering (5), extended the concept of degree of mixing and segregations to the case of continuous flow systems with an arbitrary but known residence time distribution, by defining a life-expectation distribution in addition to Danckwerts' age distribution and a condition of maximum mixedness (minimum segregation). He proposed models for calculating the conversion in a reactor in which a chemical reaction of arbitrary order takes place, and contended that two continuous reactors that have identical residence time distributions, can have different degrees of chemical conversion if their state of mixing is different.

Axial Dispersion and Residence Time Distribution

It is generally known that highest conversions are obtained from continuous chemical reactors under plug flow conditions (ie. when axial dispersion is minimized).

Koutsky and Adler (6) conducted an interesting investigation on the possible attainment of plug flow in continuous flow systems by taking advantage of the geometrical configurations of helically coiled tubes which produces secondary currents. They then made comparisons of helical tubes, straight tubes and packed beds based on tracer distribution tests and pressure drop, in both laminar and turbulent flow regimes. In describing the geometry of helical tubes, they made use of the following four dimensionless variables:

(1) R/Rc = the curvature of the helix where,

R is the equivalent cross sectional radius of the helix

Rc = radius of curvature

(2) a/b = ellipticity, which is the ratio of the major and minor axis of the elliptical cross section of the helix

(3) h/Rc = the pitch where,

h = distance between two adjacent loops of the helix

R_c = radius of curvature

(4) A parameter L/D where, L = length of tube before being formed into a helix

D = 2R, is the equivalent diameter of the helix cross section

Their work was done over specific ranges of these dimensionless variables and over a Reynolds number range of $200 \leq N_{Re} \leq 40,000$. They also used dimensionless coordinates to express the outlet tracer concentrations as a function of time:

$$Y = \frac{CV}{Q}, \quad X = \frac{vt}{V}$$

where,

Y = concentration, dimensionless

X = time, dimensionless

C = exit tracer concentrations as a function of time

V = total volume of tube

Q = total quantity of tracer injected into the system

v = volumetric flow rate

t = time

Though the data from this work does not indicate the exact strength of secondary flow in helices, they showed that pressure drop in helical tubes are up to four times as great as straight tubes and that laminar flow can be maintained up to Reynolds numbers of 8,000. These facts, the authors contended, are a clear indication that there is a substantial strength of secondary flow in helices. In the laminar flow regime, they contend that axial dispersion in helical tubes is moderately higher than that in packed beds and a lot lower than that in straight tubes. Also the pressure drop of helical tubes is intermediate between the low pressure drop of straight tubes and the higher pressure drop of packed beds. The increase in power consumption is said to follow the same order as pressure drop.

Sakra et al. (7) critically reviewed work done by others (Koutsky and Adler (6), Levenspiel and Smith (2)) and contend that their proposed reactor models have some disadvantages in that they consume much space and have difficulties in cleaning.

They proposed a reactor model that would remove these disadvantages based on their theory of double secondary flow. For the calculation of dispersion number D/UL,

where,

D = axial dispersion coefficient

L = reactor length

U = liquid flow rate through reactor

they proposed a relation whereby the dispersion number is a function of eometric parameters of the reactor, magnitude of Reynolds number, and friction factor. However, for laminar flow, dependence on geometric parameters was neglected and they gave the dispersion number as,

$$D/UL = \phi\ (Re,\ Sc)$$

In their results, they asserted that during laminar flow through a helix, D/UL decreases with an increase in Re, while the reverse is true for straight pipe.

Nauman (8) employed Dean's velocity profiles to develop numerical methods that can be used to approximate the residence time distribution for laminar flow in a helically coiled tube. He obtained values for dimensionless residence time and cumulative distribution functions, f (θ), for various streamline parameters and contended that helically coiled tubes have a narrower distribution than straight tubes.

narrower

Heat Transfer and Pressure Drop

Most of the work done on coiled tubes had been in the area of heat transfer. Coiled heat exchangers have been in use for heating and cooling fluids in a wide variety of industries. They are known to have two major advantages over straight tubes. First, heat transfer coefficients in coils are higher than in straight tubes and they allow a greater heat transfer area to be packed into a given space more economically and more conveniently than with straight tubes (9).

Srinivasan et al. (9) critically examined various published correlations on heat transfer and pressure drop in coils and proposed new equations for the critical Reynolds number. They classified coils into those with constant curvature (helices) and those with variable curvature (spirals) and reviewed the experimental and theoretical work on coils under these classifications.

A number of equations were proposed for pressure drop calculations in coils of constant curvature for both laminar and turbulent flow. Most of these equations predict either the dimensionless Fanning friction factor for coils, fc, or its ratio to the fanning friction factor for straight tubes, fs.

Eustice (10) reported the first experimental work on fluids flowing in laminar flow through coils of constant curvature in 1910. He found that volumetric flow-rate through a helix, Qc, was less than that for an equal length of straight tubing, Qs, for a given pressure drop. He correlated his results by the equation,

$$[(Qs - Qc) / Qs]^{x} = [\Delta Q/Qs]^{x} = B/D$$

where, B is a constant, D is the coil diameter, and x is an exponent.

The first theoretical analysis of the flow of incompressible fluids through helices was made by Dean (11) in 1927. His analysis was based on the assumption that the ratio of the inside diameter of the tube to the diameter of the coil is small. As a result, he expressed the dynamic similarity of fluid flow by the dimensionless group:

$$[2\,(N_{Re})^{2}\,(di/D)]$$

where,

di = tube diameter

D = coil diameter

and also derived the equations

$$Qc/Qs = 1 - (0.03058)[2(N_{Re})^{2}\,(di/D)/597]^{2}$$
$$+ (0.01195)\,[2\,(N_{Re})^{2}\,(di/D)/576]^{4}$$

This equation, however, is known to be of little practical importance, because it is only valid for small values of the dimensionless group given above.

On further investigation of Dean's theoretical analysis, White (12) obtained a relationship for the pressure head loss in a coil as:

$$\Delta H = C\,(8/N_{Re})\,[(4L/di)(u^2/g)]$$

where

L = length of coil

u = mean velocity

$$\frac{1}{C} = \{1 - [1 - (116/N_{Dn})^{0.45}]^{1/0.45}\}$$

and N_{Dn} = Dean's Number = $N_{Re}(di/D)^{0.5}$

Most subsequent papers in this subject were essentially based on Dean's theoretical analysis.

On heat transfer in helices, Mori and Nakayama (13) obtained data experimentally to show the velocity and temperature distributions over a cross section of fluid in a coil for both laminar and turbulent flow. They found both profiles to be asymmetric.

Correlations have also been proposed for calculating the desired length of coil in a helix. Shpringz (14) gave the equation to calculate the length of a helix, Ln, with n turns as:

$$Ln = \pi Dn\,(1 + p^2/4D^2)^{0.5}$$

where, p = pitch and D = coil diameter

Kalb and Seader (15) experimentally investigated the entrance-region heat transfer to gases flowing in a uniform wall-temperature helical coil. They contended that, compared with straight-tube flow, the induced secondary flow in curved tubes causes a higher critical Reynolds number for transitions to turbulent flow and significant variation of transfer coefficients. Their results indicate a rapid transition to laminar flow from turbulent flow conditions at the entrance region.

In a related study, Dravid et al. (21) recommended design values of the heat transfer coefficients for helically coiled tubes operating in the thermal entrance region and at Dean's numbers above 100. Their work was limited to the laminar flow regime.

Owhadi et al. (22,23,24) made a series of investigations on forced convection boiling inside helically coiled tubes and proposed correlations for two phase pressure drop and heat transfer.

Kinetics and Experiment

The investigations made in this work were based on the hydrolysis of crystal violet dye with sodium hydroxide. Corsaro (16) gave a description of the kinetics of this reaction by showing the electron deficiency at the tertiary carbon position where hydroxide ion (OH) is expected to attack to form a colorless carbinol derivative. By measuring the loss of color intensity of the dye at different time intervals, a rate constant can be obtained directly from absorbance readings taken with a spectrophotometer.

In molecular form, the reaction is represented as,

$$(C_6H_4N(CH_3)_2)_3\ CCl + NaOH \rightarrow (C_6H_4N(CH_3)_2)_3\ COH + NaCl$$

This reaction is first order with respect to each reactant and second order overall. If the sodium hydroxide concentration is in great excess initially, its concentration remains approximately constant at all times, hence the overall second-order reaction becomes a pseudo first-order reaction (17), represented by

$$-r_{dye} = K' \, [dye]$$

where,

$$K' = K \, [NaOH]$$

Based on this concept, a few attempts had been made to develop a simple tubular flow reactor experiment for undergraduate laboratory.

Anderson (18) developed one using acetic anhydride and water. The elaborate safety precautions and expensive temperature control requirements of this design made it unacceptable to subsequent investigators.

Hudgins and Cayrol (19) improved on Anderson's (18) design by using a color change as a visual reinforcement of the measured results and developed an experiment which operates at room temperature. They used the hydrolysis of crystal violet dye with sodium hydroxide as their system which remains fairly isothermal during reaction. They also made the reagents very dilute to minimize safety hazards. They compared their experimental results with conversions from PfTR (plug flow tubular reactor) and LfTR (laminar flow tubular reactor) models and concluded that the behavior of their reactor model (helical reactor) lies between the LfTR and PfTR. From their plots, it is interesting to observe that the experimental conversion curve crosses the LfTR curve at short holding times. This suggests that experimental conversions are lower than LfTR at these times.

Asfour (20) suggested design improvements on the work of Hudgins and Cayrol (19), to improve the reproducibility of the results, and to expedite data collection. He studied the effect of residence time on conversion in a tubular flow reactor and compared the experimental conversion with those of plug-flow and laminar-flow models. From the plot of the results of this experiment, it is evident that the experimental conversions fall between those of the plug-flow and laminar-flow models. His experimental conversion curve indicates that there is no intersection with the LFTR but it seems to indicate that at very large or very low holding times, the experimental conversion will approach plug-flow model.

The present investigation was done with few design changes from those of the previous investigators. The hydrolysis of crystal violet dye with sodium hydroxide was employed in the system. (Only newtonian fluids in the laminar flow region are presented in this work.)

In part, this work was aimed at designing a simple helical flow reactor that could be used in undergraduate laboratories. (It is theoretically acceptable that maximum conversions are obtained in plug-flow reactor models and minimum conversions in laminar flow models.) Previous investigators were able to establish that conversion for helical flow models fall somewhere between these two models but there are discrepancies as to where in between these two models the helical flow falls. This present investigation will attempt to predict where the helical flow model falls.

Again, this work was done with the understanding that secondary mixing occurs in helically coiled tubes due to their geometrical configuration. The strength or extent of this mixing is not within the scope of this work. It was intended to study the effect of secondary mixing in helical reactors, at low Reynolds number. Finally, an attempt will be made to propose a model that can predict conversion and power consumption, given Reynolds number, coil diameter, radius of curvature and coil length. Some of the correlations proposed in the literature in other areas of study will be used in this model.

CHAPTER III

KINETICS STUDY

Reaction

The reaction for this investigation is the hydrolysis of crystal-violet dye with sodium hydroxide.

$$(C_6H_4N(CH_3)_2)_3CCl + NaOH ==> (C_6H_4N(CH_3)_2)_3COH + NaCl \quad (1)$$

This reaction is first order with respect to each reactant and second order overall. The overall expression can be put in the form

$$A+B ==> \text{products} \quad (2)$$

with corresponding rate equation,

$$-r_A = -dC_A/dt = KC_AC_B \quad (3)$$

If $C_{B0} >> C_{A0}$, then C_B remains approximately constant at all times, and the above irreversible second order reaction becomes a pseudo-first order reaction represented by,

$$dC_A/dt = -K'C_A \quad (4)$$

where

$$K' = KC_B$$

By separation and integration of equation (4) we obtain,

$$\ln C_A/C_{A0} = -K' t \quad (5)$$

Equation (5) was used in studying the kinetics of the batch reaction for these reactants.

Calibration of Spectrophotometer

The spectronic 20 spectrophotometer, from Milton Roy, was used in this work. The Spec-20 was calibrated over a range of concentrations. Calibration curves are shown in Figure 1 and Figure 2. Before calibration, a performance check was made on the Spec-20.

The technical services section of Aldrich Chemical Company (the dye manufacturers), gave data on the extinction coefficients obtained using 0.003 g/L dye solution over a UV range of 209-591 nm(max). The relation given for the extinction coefficient is,

Extinction Coefficient (E) = Absorbance x Mol.Wt/concentration in g/L.

Based on this data, absorbance values were calculated for the chosen concentrations knowing the molecular weight of the dye.

To check the performance of the Spec-20, 0.003 g/L dye solution was prepared and two runs were made at designated wavelengths. It was found that at 591 nm(max.), the Spec-20 readings were the same as the calculated value and at lower wavelengths, the deviation was about 2%.

As a further check, two spectrophotometers of the same model were used to make runs over the same concentration range and at wavelengths of 591 nm and 590 nm. Again, the readings were the same for the two at 591 and 590 nm.

Hence calibration curves were made at both 590 and 591 nm using one Spec-20 but 591 nm was used for further investigation in this experiment since it is the maximum wavelength for the dye.

To calibrate the Spec-20, 0.05 g of dye was dissolved in 1 litre of deionized water (conductivity 80 micro-ohm/cm; 2 ppm). With a pipette, various volumes of this solution were diluted to obtain a concentration range of 0.5×10^{-6} g/mL to 10×10^{-6} g/mL.

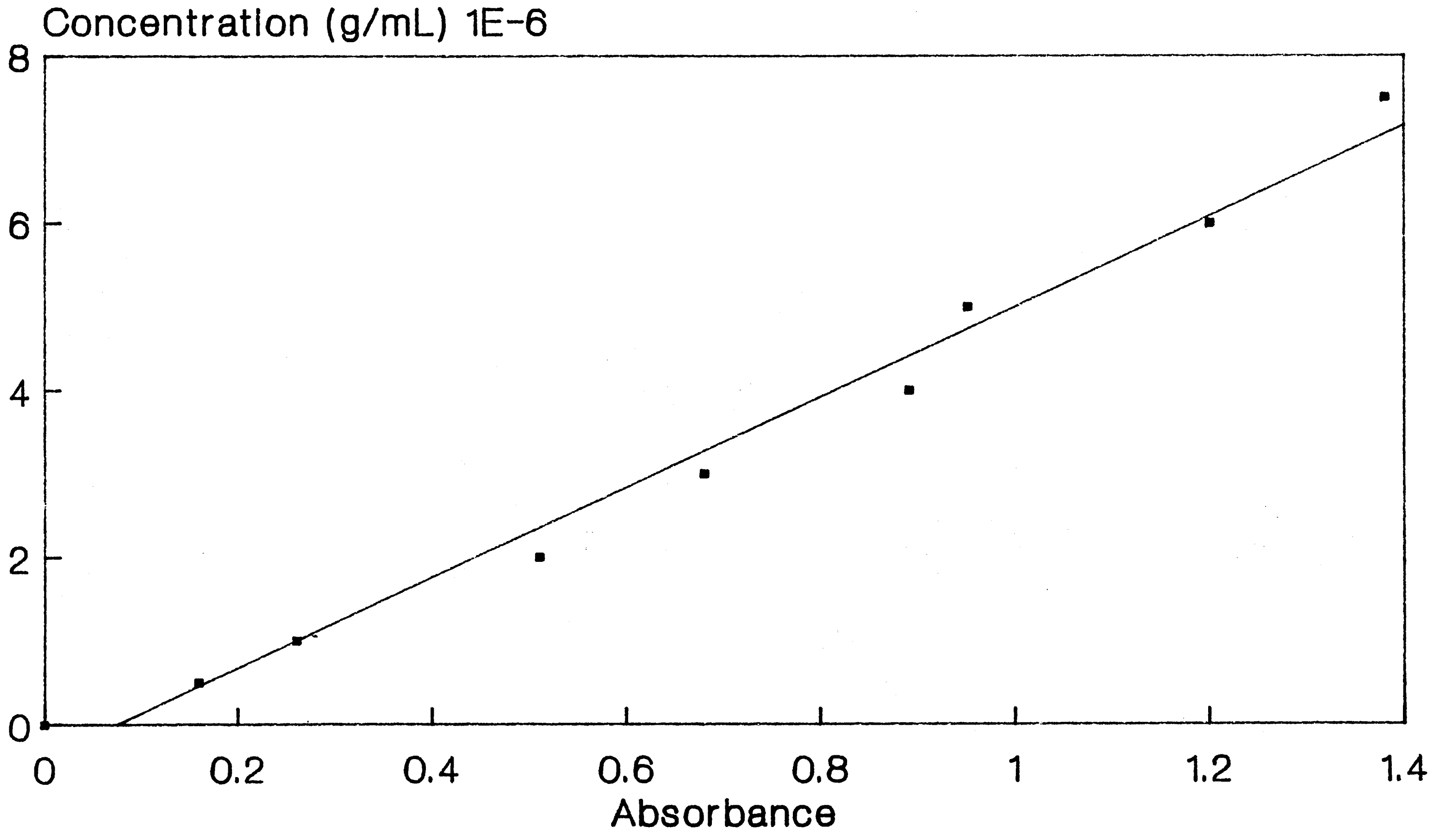

Figure 1. Spec-20 calibration at 591 nm.

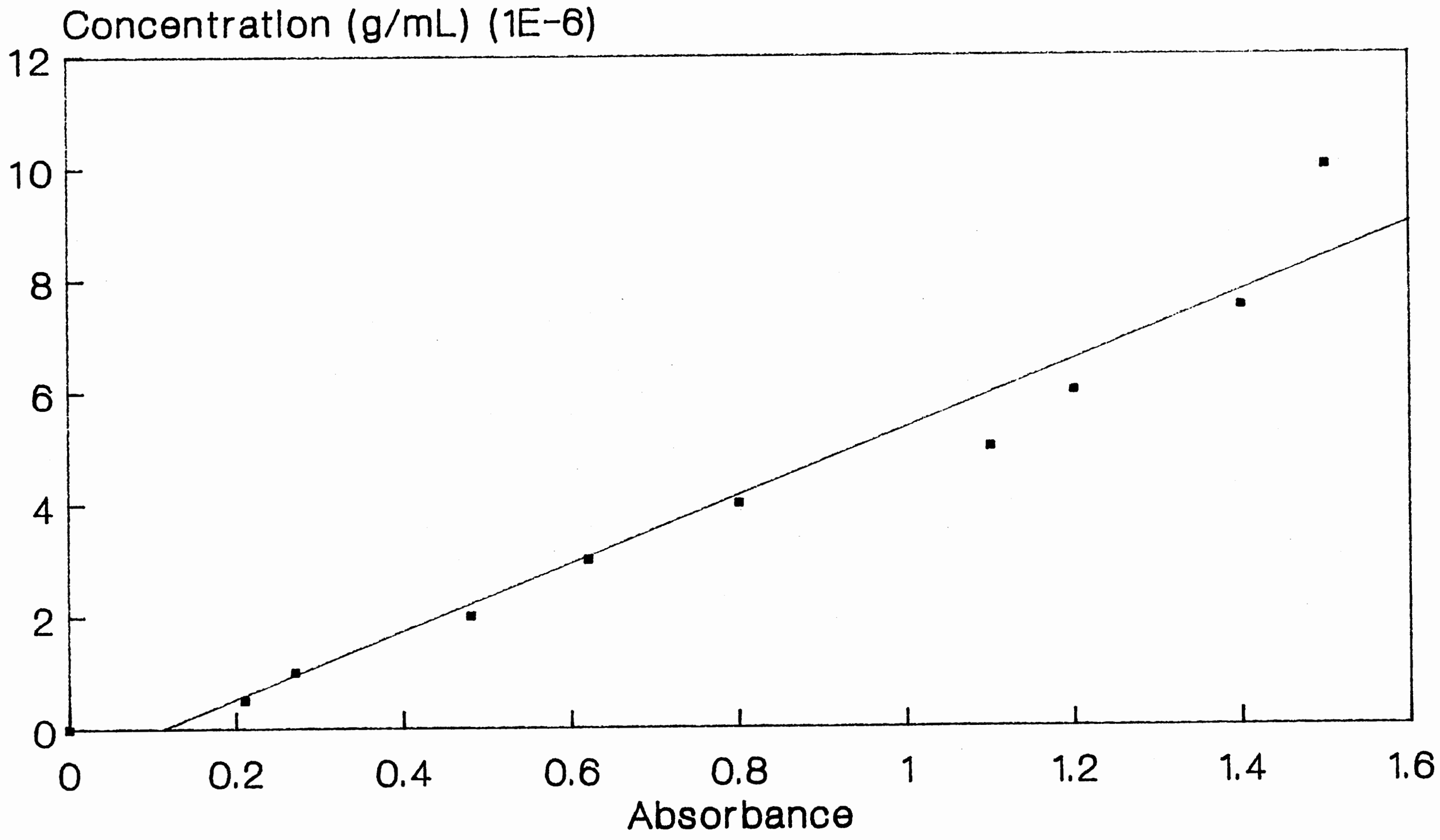

Figure 2. Spec-20 calibration at 590 nm.

Runs were made in this concentration range at 591 nm and 590 nm wavelengths to obtain the corresponding absorbance values. The data obtained are shown in Tables XXVI and XXVII, in Appendix B.

Batch Reaction

Calculations

An important condition for the assumption of pseudo-first order for this reaction is that $C_{B0} >> C_{A0}$. As shown in the following calculations, the initial conditions for this reaction were chosen to meet this requirement.

Mol.Wt of NaOH = 40 g/mole

Mol.Wt of crystal violet dye = 408 g/mole

As reported by Hudgins and Cayrol (19) the dye concentrations needed for this experiment are of the order of 10^{-5} M. Based on this, 0.03 g/L solution of dye was made, which gave a dye concentration of 7.353×10^{-5} M.

For the purpose of this experiment, 0.02 M and 0.04 M NaOH solutions were used with 7.353×10^{-5} M dye solution. C_{A0} and C_{B0} were obtained as follows:

$0.01 \text{ L} \times 7.353 \times 10^{-5} \text{ M dye} = 7.353 \times 10^{-7}$ moles

$0.30 \text{ L} \times 0.02 \text{ M NaOH} = 6.0 \times 10^{-3}$ moles

total volume = 0.31 L

$C_{A0} = 7.353 \times 10^{-7} \text{ moles}/0.31 = 2.372 \times 10^{-6}$ M

$C_{B0} = 6.0 \times 10^{-3} \text{ moles}/0.31 = 0.0194$ M

The molar ratio C_{B0}: C_{A0} becomes 8179:1. This is sufficiently large for the assumption of pseudo-first order to be reasonable.

In the same way, 0.01 L of 7.353 x 10^{-5} M dye solution was mixed with 0.35 L of 0.02 M NaOH to obtain C_{A0} = 2.043 x 10^{-6} M and C_{B0} = 0.0194 M which gives a ratio of 9496:1.

For the 0.04 M NaOH solution, 0.01 L of 7.353 x 10^{-5} M dye solution was mixed with 0.30 L of 0.04 M NaOH to obtain C_{A0} = 2.372 x 10^{-6} M and C_{B0} = 0.0387 M which is a ratio of 16315:1. Again, 0.01 L of 7.353 x 10^{-5} M dye solution mixed with 0.35 L of 0.04 M NaOH gives C_{A0} = 2.043 x 10^{-6} M and C_{B0} = 0.0389 M which is a ratio of 19041:1. These four sets of concentration were used for the kinetics study.

Procedure

For clarity, the batch runs were classified as A,B,C,D, and E to identify the purposes for which they were made, but the procedure was the same and was as follows:

1. 0.03 g of dye, 0.8 g and 1.6 g of dry NaOH are measured with a chemical balance and diluted to make 7.353 x 10^{-5} dye solution, 0.02 M NaOH and 0.04 M NaOH solutions respectively.

2. After a 15 min warm up, the Spec-20 was set to 0% transmittance by completely blocking the light path and with a blank (deionized water) it was set to 100% transmittance.

3. The appropriate volume of NaOH solution was poured into a beaker and set to stir on a magnetic stirrer to ensure complete mixing. A thermometer was also inserted in the solution to measure the temperature.

4. With a pipette, 10 ml of the dye solution was transferred to a test tube.

5. The 10 ml dye solution was poured into the stirring NaOH solution and a stop watch started simultaneously.

6. At given times, samples of the reacting solution were taken from the beaker with a pipette, transferred to a Spec-20 cell (7/16" i.d test-tube), and inserted in the Spec-20 sample compartment for an absorbance reading.

This sampling process took about 20 seconds and the suggested effect on the obtained data is discussed in the section on reproducibility. The detailed procedure and precautions taken for each class of runs are discussed in the next section.

Reaction Results

Batch A Runs

The NaOH used for this experiment had been in use for a long time (2 yrs). To ensure that it was dry, the NaOH (powder) was put in an oven at a temperature of about 100°C for a period of time and then transferred to a dessicator.

Runs A.1 and A.2 were made with 0.02 M NaOH. The observed absorbance readings were converted to concentration with the calibration curve and the data are given in Tables XXVIII and XXIX in Appendix B. The plots are shown in Figures 3 and 4 respectively. A linear regression (least square fit) was done with a HP-41C calculator to find the correlation and slope.

Runs A.3 and A.4 were made with 0.04 M NaOH. Since the concentration of NaOH in these runs were doubled, the rate constants, by stoichiometry, were expected to be twice the values in A.1 and A.2. Therefore, runs A.3 and A.4 were made to prove the validity of that

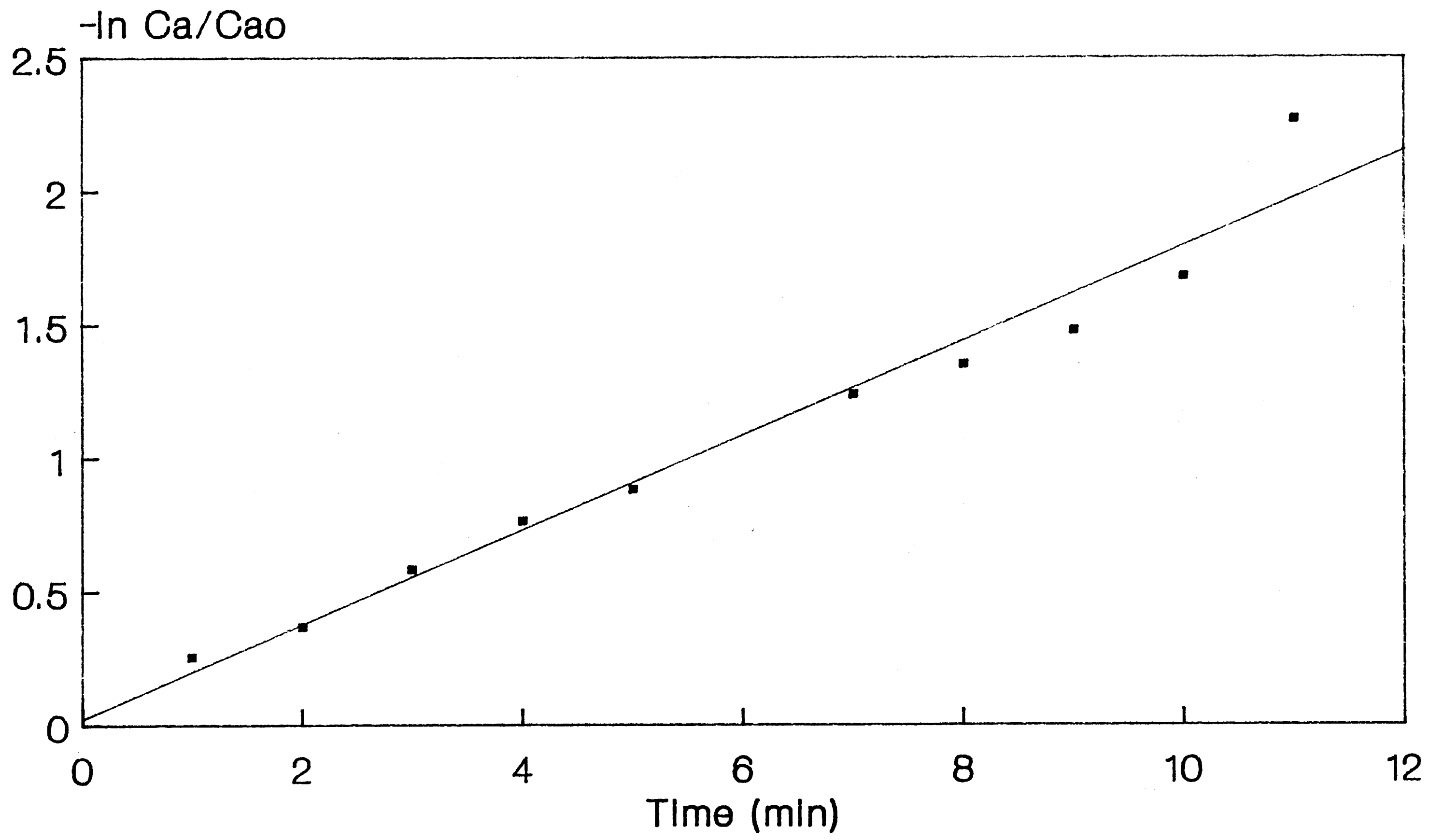

Figure 3. Plot of Run A.1 data

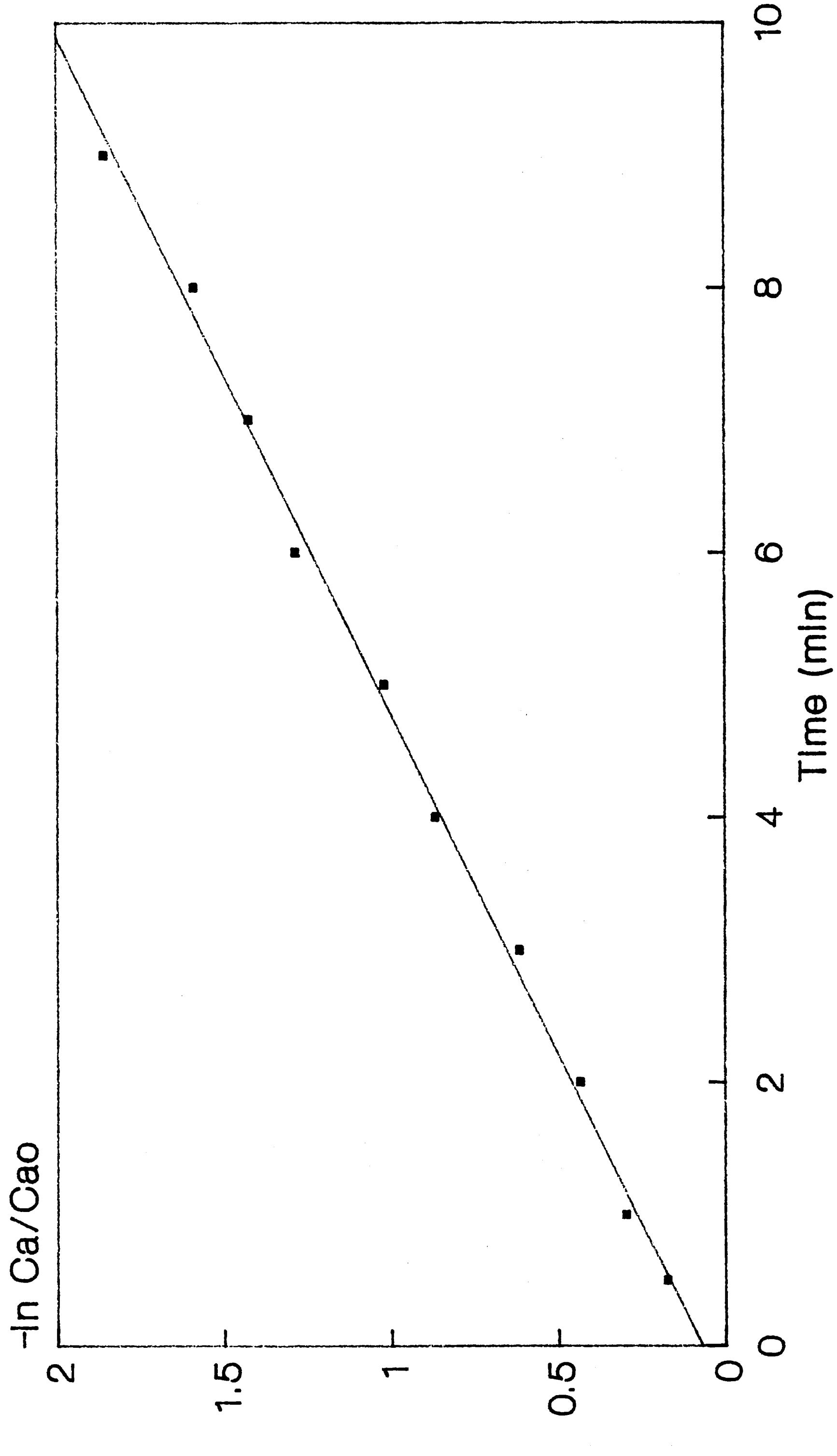

Figure 4. Plot of Run A.2 data

concept. The data are given in Tables XXX and XXXI in Appendix B and the plots are shown in Figures 5 and 6.

Runs A.5 to A.10 were made to obtain replicates of the earlier runs. Runs A.5 and A.8 were made with freshly prepared dye solution in the same order of concentration as runs A.1 to A.4. The data obtained for these runs are given in Tables XXXII to XXXV in Appendix B. Runs A.9 and A.10 are replicates of A.3 and A.1. They were made with the same dye solution as A.1 to A.4, and the data are given in Tables XXXVI and XXXVII in Appendix B. The temperatures of the solutions for each of these runs were recorded.

The absorbance readings obtained (raw data), were converted to concentration using the calibration curve and then divided by the molecular weight of dye to get the values in moles/litre. Some approximations were made in this process. In order to eliminate the possibility of errors arising from these conversions and approximations, the raw absorbance data were used directly for linear regression. To do this a value was needed for an initial absorbance (Ao) i.e. the absorbance at time, t=0, when there was no reaction. The Ao value was determined as follows:

(a) 300 ml of deionized water (2 ppm, 80 microhm/cm) was mixed with 10 ml of 7.353×10^{-5} M dye solution and stirred at room temperature (24^{o}C). A sample of this solution was put into the Spec-20 to find Ao=0.23.

(b) The same procedure was used for 350 ml of deionized water and 10 ml of 7.353×10^{-5} M dye solution (26^{o}C) to find Ao = 0.21.

These Ao values were used with the absorbance values in runs A.1 to A.10 to obtain ln Ao/A. Ao=0.23 was used in the runs that have 10 ml of

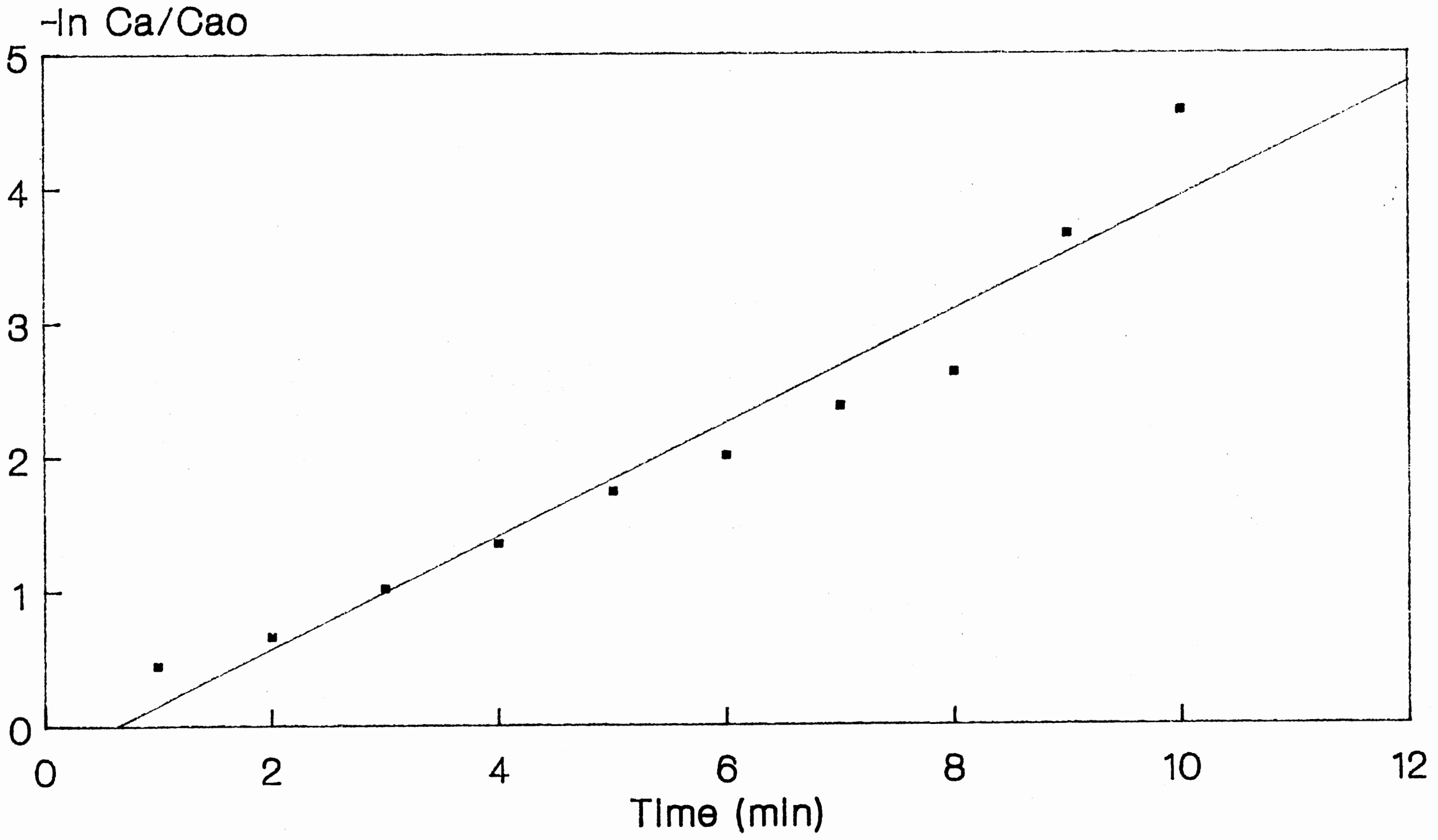

Figure 5. Plot of Run A.3 data

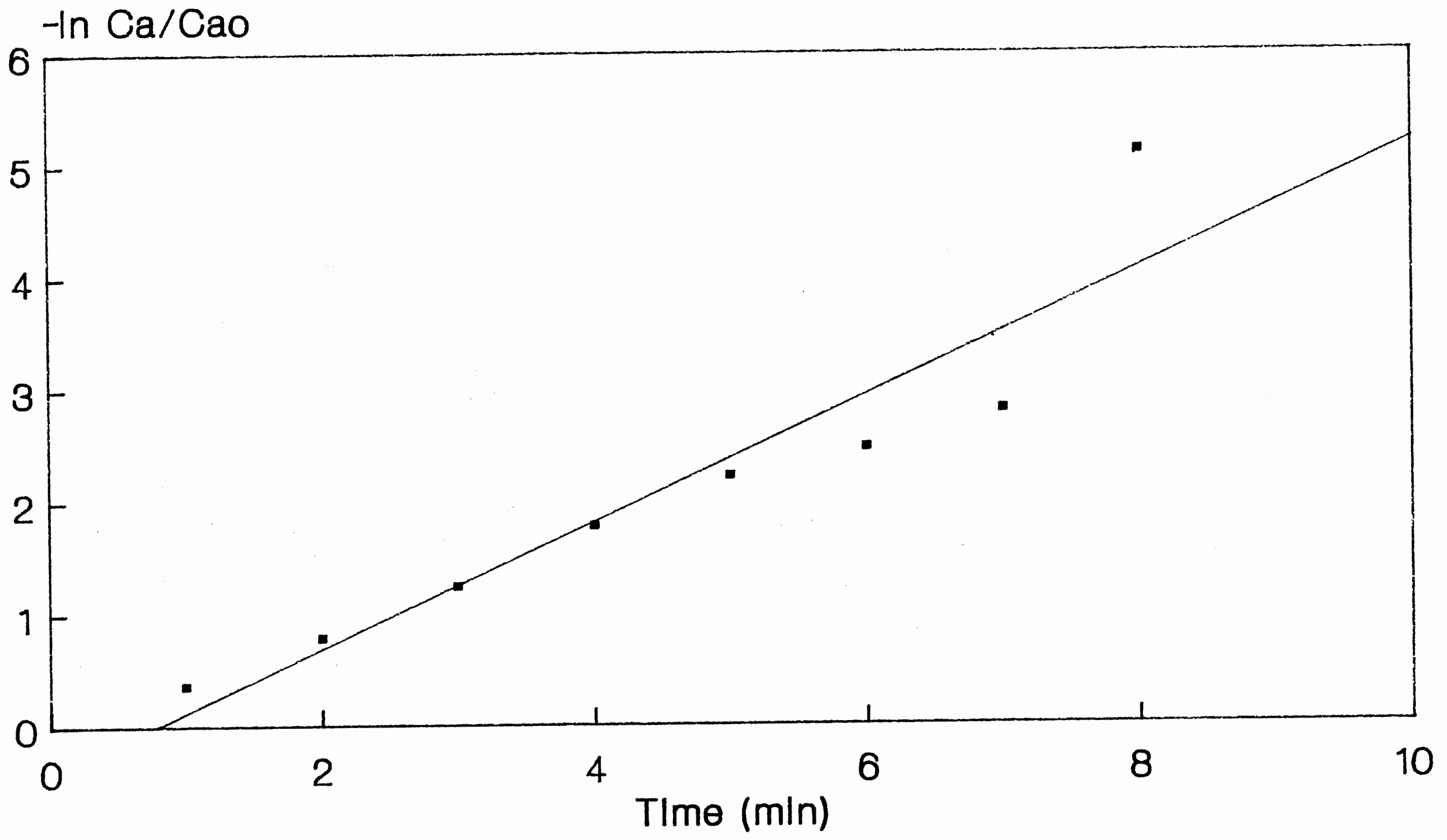

Figure 6. Plot of run A.4 data

dye solution reacting with 300 ml of NaOH and Ao=0.21 was used in the runs that have 10 ml of dye solution reacting with 350 ml of NaOH.

Plots of ln Ao/A vs. time for runs A.1 to A.4 are shown in Figures 7 to 10. The corresponding tables and regression parameters are given in Tables XXXVIII to XLI in Appendix B. Also Tables XLII to XLVII in Appendix B give absorbance regressions for runs A.5 to A.10. Plots were not made for these data.

Batch Runs B and C

In batch A-runs the same sample cell was used for each sample and the readings were taken by visual inspection of the Spec-20.

In batch B and C runs, a digital multimeter was connected to the accessory analog output of the Spec-20 to obtain a digital display of the readings. This was intended to eliminate any errors due to visual effects. In addition, different sample cells (made of the same material) were used for each sample and the pipette was cleaned between samples. These measures were designed to eliminate the possibility of contamination of a sample by a previous one.

The digital output was calibrated from 0% to 100% transmittance and the obtained readings were converted to absorbance by the relation;

$$A = -\log_{10} T$$

Ao values were determined with a digital multimeter using the procedure described in batch A runs section. The regressed absorbance values for runs B.1, B.2, C.1 and C.2 are given in Tables XLVIII to LI in Appendix B.

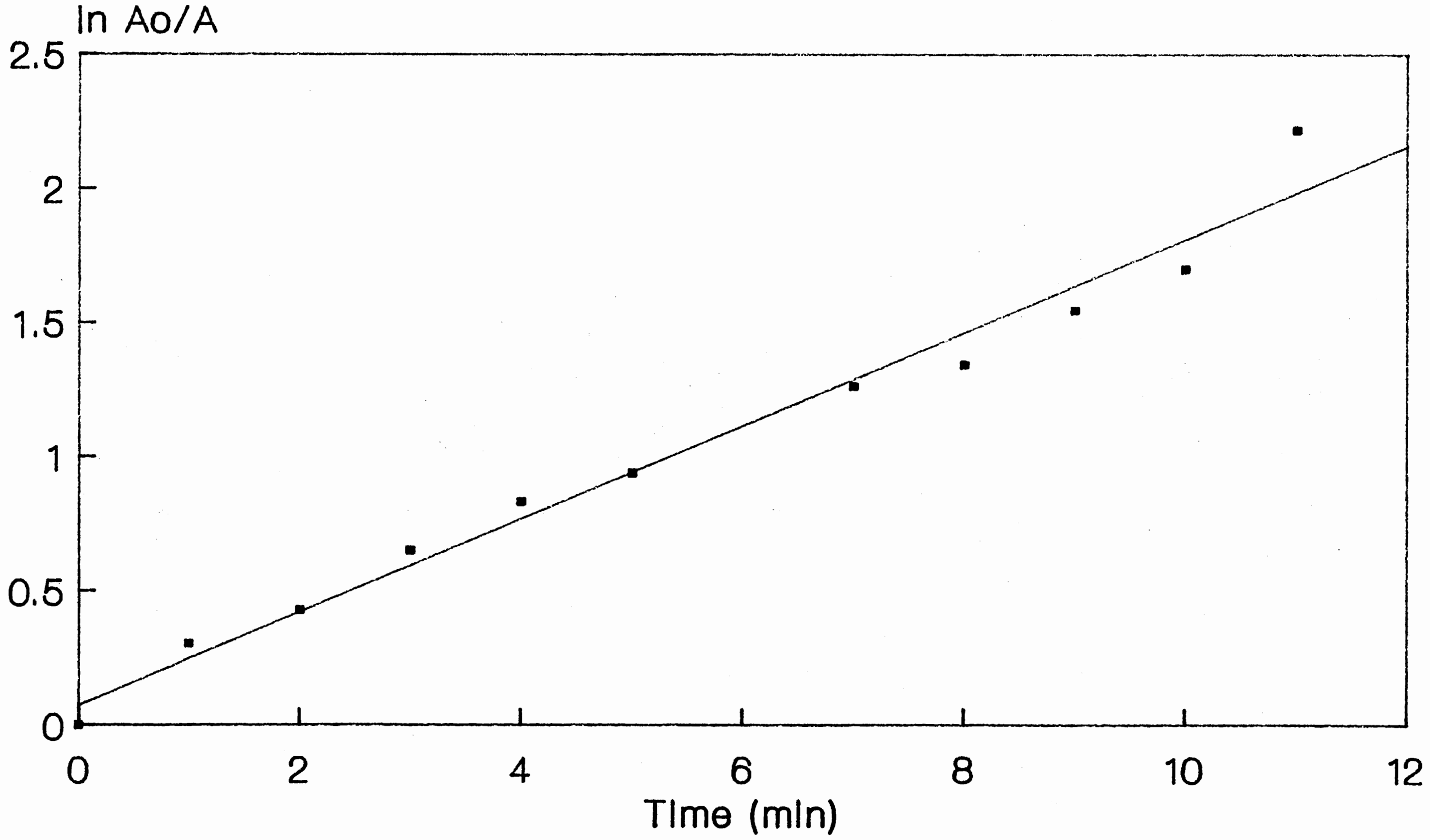

Figure 7. ln Ao/A vs. time for run A.1

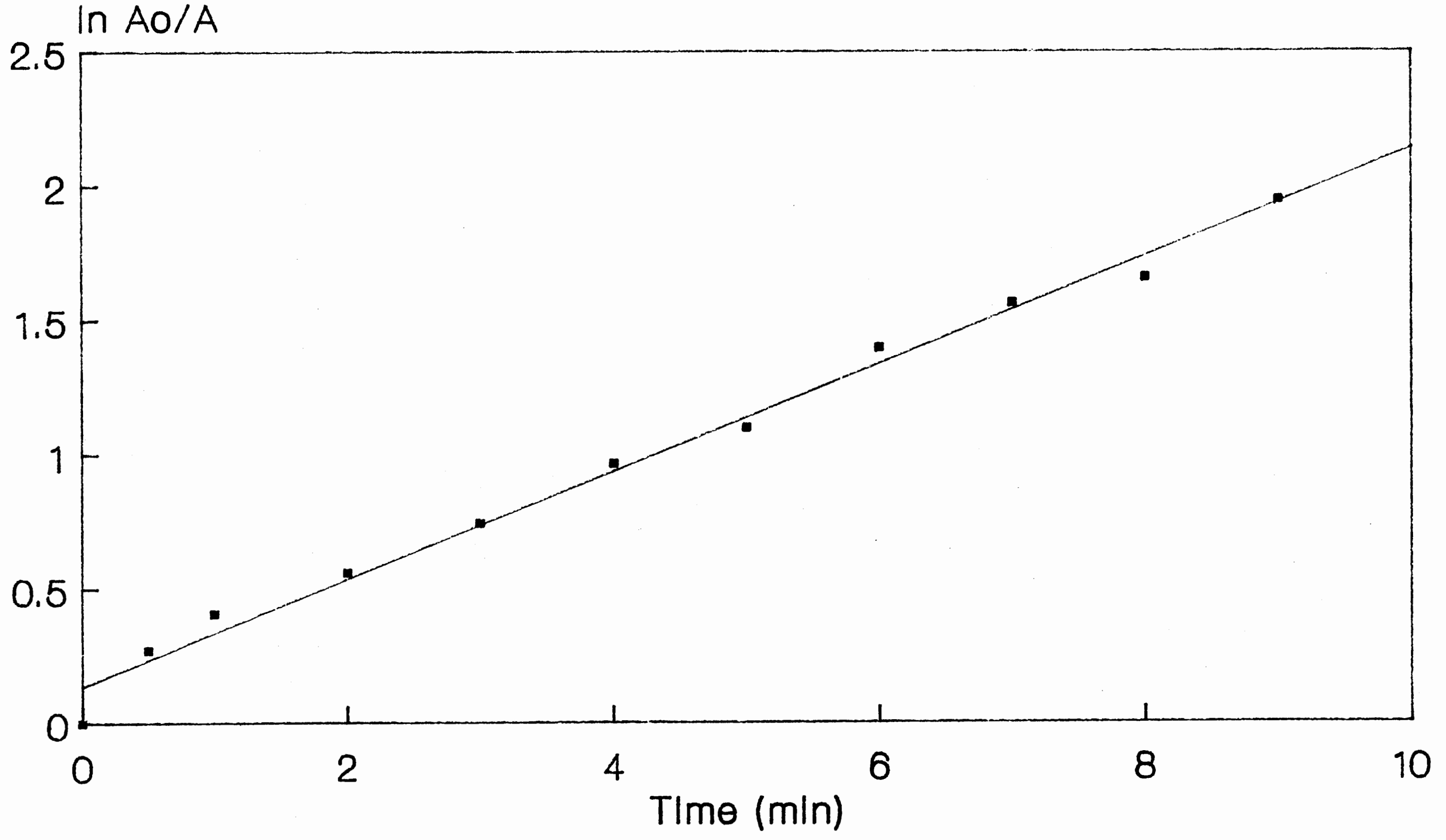

Figure 8. ln Ao/A vs. time for run A.2

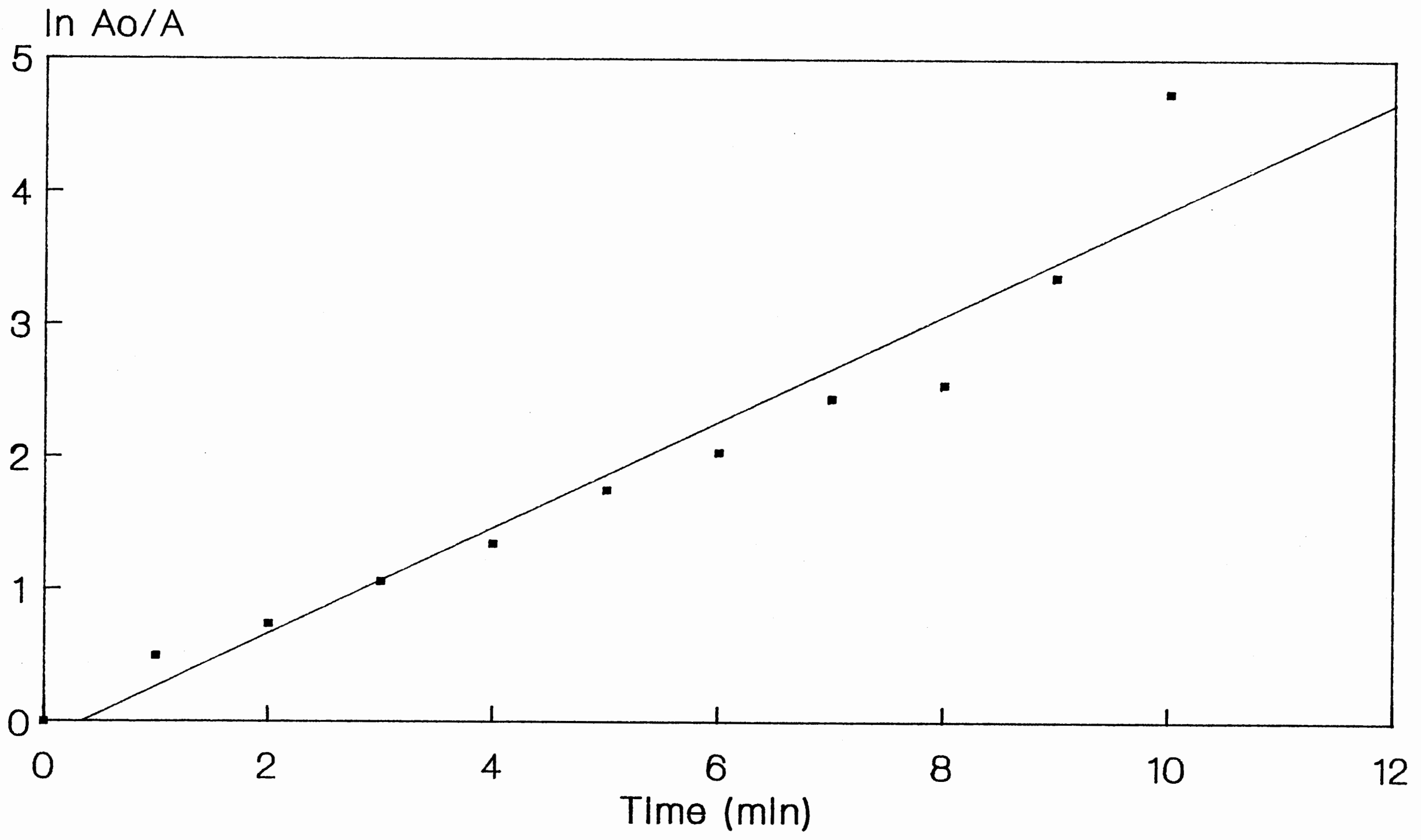

Figure 9. ln Ao/A vs. time for run A.3

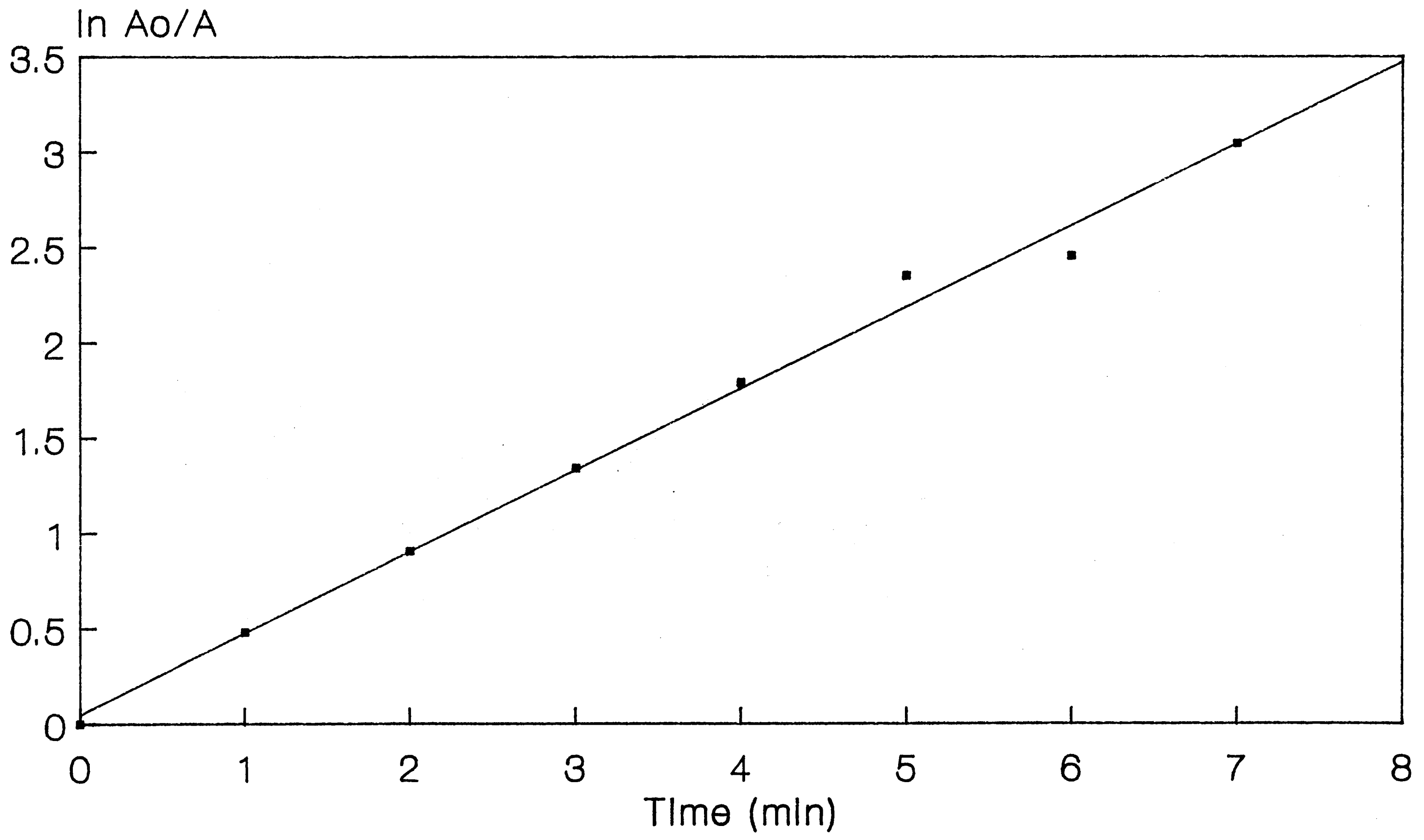

Figure 10. ln Ao/A vs. time for run A.4

Batch D-Runs

The D-runs were made at different temperatures to study the effect of temperature on the reaction rate constant.

All D-runs were made with 10 ml of dye solution and 300 ml of 0.02 M NaOH in a beaker. The NaOH solution was cooled in ice, or heated, to the desired temperature after initial stirring. The dye solution was added at the desired temperature and the resultant solution stirred continuously as the samples were taken. Since this was not in a constant temperature bath, it was not possible to perform the experiment under completely isothermal conditions. However, initial and final temperatures for each run were recorded. The average of these two temperatures was used in the final determination of the rate constant expression described in the section on determination of rate constant.

Tables for batch runs D.1 to D.5 are given in Tables LII to LVI in Appendix B.

To determine the Ao values used for these runs, 10 ml of dye solution was diluted in 300 ml of deionized water (conductivity 0.8 microhm/cm). At temperature lower than room temperature, solution was cooled with ice and at higher temperatures it was heated. The digital multimeter was used to read the absolute transmittance which was converted to absorbance by the relationship given in the section on batch runs B and C. A table of these Ao values is given in Table LVII in Appendix B.

In the reactions of the dye solution with NaOH, there was a remarkable increase in the rate of disappearance of color of dye as the temperature of the reaction was increased. This suggests an increase in reaction rate at higher temperatures.

On the other hand, in determining the initial absorbance (Ao) with deionized water at the designated temperatures, there was no identifiable trend. Since; (a) the solution was continuously stirred to avoid concentration gradients (b) the temperature was not lowered below the water saturation point or freezing point, (c) the temperature was not raised above the boiling point; a significant change in density was not expected. Again it was assumed that there was no reaction between the dye solution and the deionized water. Therefore the Ao value was expected to be the same at all temperatures. Based on this reasoning, the average of the temperatures (21^oC) was taken to be the room temperature and also an average of the Ao readings was taken to give Ao = 0.2506. This Ao value was used for all the regressions in D-runs.

Batch E-Runs

The E-runs were made to investigate a probable effect of dissolved CO_2 on the concentration of NaOH at atmospheric pressure. The following procedure was used:

1. 0.8 g NaOH was dissolved in 1L of deionized water i.e 0.02 M NaOH and kept open to air for at least 3 hrs.

2. 1L of water was heated up to 100^oC and allowed to boil for about 2 mins. It was expected that most of the dissolved gasses would evaporate through this process.

3. The boiled water was allowed to cool to room temperature and 0.8 g NaOH was dissolved in it.

4. 10 ml of dye solution was reacted with 300 ml of 0.02 M NaOH obtained with ordinary deionized water and then repeated for boiled deionized water.

Runs E-1 and E-2 were made accordingly and the data are given in Tables LVIII and LIX in Appendix B. It was observed that the difference between the results of the two runs is only 0.1% which, for practical purposes is not significant.

Reproducibility

Runs A.5 to A.10 were made to check the reproducibility of the batch experiment. The high correlations obtained in the linear regressions is an indication of overall reproducibility of the experimental data.

For the regressions based on concentration, the deviations ranged from 0.6 to 6%. This was considered to be within the analytical precision range. The deviations may be attributed to (a) approximations made in converting to concentrations from absorbance, (b) difference in room temperature on various days of the runs, (c) possible variations in measurement of dye and NaOH for each day of the runs, (d) possible contamination of one sample by a previous one in the course of the run due to use of one cell, (e) variation in the time used in pipetting and inserting samples in the Spec-20, since the reaction still goes on during this time.

However, regressions based on the raw absorbance data were found to have smaller deviations. Comparison made between runs shows good agreement with an average of 4% difference. This indicates that the experimental data are fairly reproducible.

Determination of Rate Constant

For the reaction,

$$-dC_A/dt = KC_AC_B$$

if $C_{Bo} >> C_{Ao}$, then C_B remains approximately constant at all times:

Let

$$KC_{Bo} = K'$$

then

$$-dC_A/dt = K'C_A$$

The summaries of the K-values obtained for runs A.1 to A.10 using regressions based on concentration and absorbance are given in Tables I and II respectively.

Since the results of the experiments indicate a temperature dependency for the reaction, an Arrhenius plot was made for all the runs.

The Arrhenius equation is given by,

$$K = K_o[\exp(- E/RT)]$$

or

$$\ln K = \ln K_o - E/RT$$

By plotting ln K vs. 1/T from the experimental results, the parameters, K_o and E can be determined:

$$\text{slope} = - E/R$$

$$\text{intercept} = \ln K_o$$

A summary of 1/T and ln K for all the runs is given in Table III. The plot of ln K vs. 1/T is shown in Figure 11. Reported data from a group at Rensselaer Polytechnic Institute on the same reaction was also plotted in Figure 11.

TABLE I

SUMMARY OF K VALUES FOR CONCENTRATIONS

Date: 12-2-87

Temp(^{o}C)	Run #	K'(min^{-1})	C_{Bo}(M)	K=K'/C_{Bo}(1/mol min)
24	A.1	0.1580	0.0194	8.1443
24	A.2	0.1954	0.0194	10.0722
24	A.3	0.3686	0.0387	9.5245
24	A.4	0.4185	0.0389	10.7584
24	A.5	0.1424	0.0194	7.3402
26	A.6	0.1966	0.0194	10.1340
26	A.7	0.2467	0.0387	6.3747
26	A.8	0.2876	0.0389	7.3933
26	A.9	0.2308	0.0387	5.9638
24	A.10	0.1566	0.0194	8.0722

TABLE II

SUMMARY OF K VALUES FOR ABSORBANCE

Date: 12-2-87

Temp(^{o}C)	Run #	K'(min^{-1})	C_{Bo}(M)	K=K'/C_{Bo}(1/mol min)
24	A.1	0.1701	0.0194	8.7680
24	A.2	0.1928	0.0194	9.9381
24	A.3	0.4098	0.0387	10.5891
24	A.4	0.4218	0.0389	10.8432
24	A.5	0.1386	0.0194	7.1443
26	A.6	0.1853	0.0194	9.5515
26	A.7	0.2491	0.0387	6.4367
26	A.8	0.2937	0.0389	7.5501
26	A.9	0.2263	0.0387	5.8475
24	A.10	0.1516	0.0194	7.8144

TABLE III

ARRHENIUS PLOT DATA FOR ALL RUNS

10 ml of 7.353×10^{-5} M dye solution Date: 1-17-88
300 ml of 0.02 M NaOH

Run #	Temp(^{o}C)	K(l/mol min)	1/T(K^{-1})	ln K
A.1	24	8.7680	3.367×10^{-3}	2.171
A.2	24	10.0722	3.367×10^{-3}	2.310
A.3	24	9.5245	3.367×10^{-3}	2.254
A.4	24	10.7584	3.367×10^{-3}	2.376
A.5	24	7.3402	3.367×10^{-3}	1.993
A.6	26	10.1340	3.344×10^{-3}	2.316
A.7	26	6.3747	3.344×10^{-3}	1.852
A.8	26	7.5501	3.344×10^{-3}	2.022
A.9	26	5.8475	3.344×10^{-3}	1.766
A.10	24	7.8144	3.367×10^{-3}	2.056
B.1	24	6.8144	3.367×10^{-3}	1.919
B.2	24	6.9845	3.367×10^{-3}	1.944
C.1	24	8.6434	3.367×10^{-3}	2.157
C.2	24	11.2416	3.367×10^{-3}	2.420
D.1	13	2.6237	3.50×10^{-3}	0.9646
D.2	38	44.8557	3.22×10^{-3}	3.8035
D.3	17	3.6649	3.45×10^{-3}	1.2988
D.4	30	11.1598	3.30×10^{-3}	2.4123
D.5	22	5.5773	3.39×10^{-3}	1.7187
E.1	25	9.1392	3.356×10^{-3}	2.2126
E.2	25	9.1289	3.356×10^{-3}	2.2114

TABLE IV

SUMMARY OF EFFECT OF TEMPERATURE ON RATE CONSTANT

C_{Bo} = 0.0194 M Date: 12-16-87

Run #	Av. Temp. (^{o}C)	K' (min^{-1})	K($l/mol\ min^{-1}$)	1/T (K^{-1})	ln K
D.1	13	0.0509	2.6237	$3.50x10^{-3}$	0.9646
*D.2	38	0.0711	44.8557	$3.22x10^{-3}$	3.8035
D.3	17	0.1082	3.6649	$3.45x10^{-3}$	1.2988
D.4	30	0.2165	11.1598	$3.30x10^{-3}$	2.4123
D.5	22	0.8702	5.5773	$3.39x10^{-3}$	1.7187

Correlation coeff. = 0.9989, Intercept = 26.3228, Slope = 7250.4955
*Point not used in linear regression

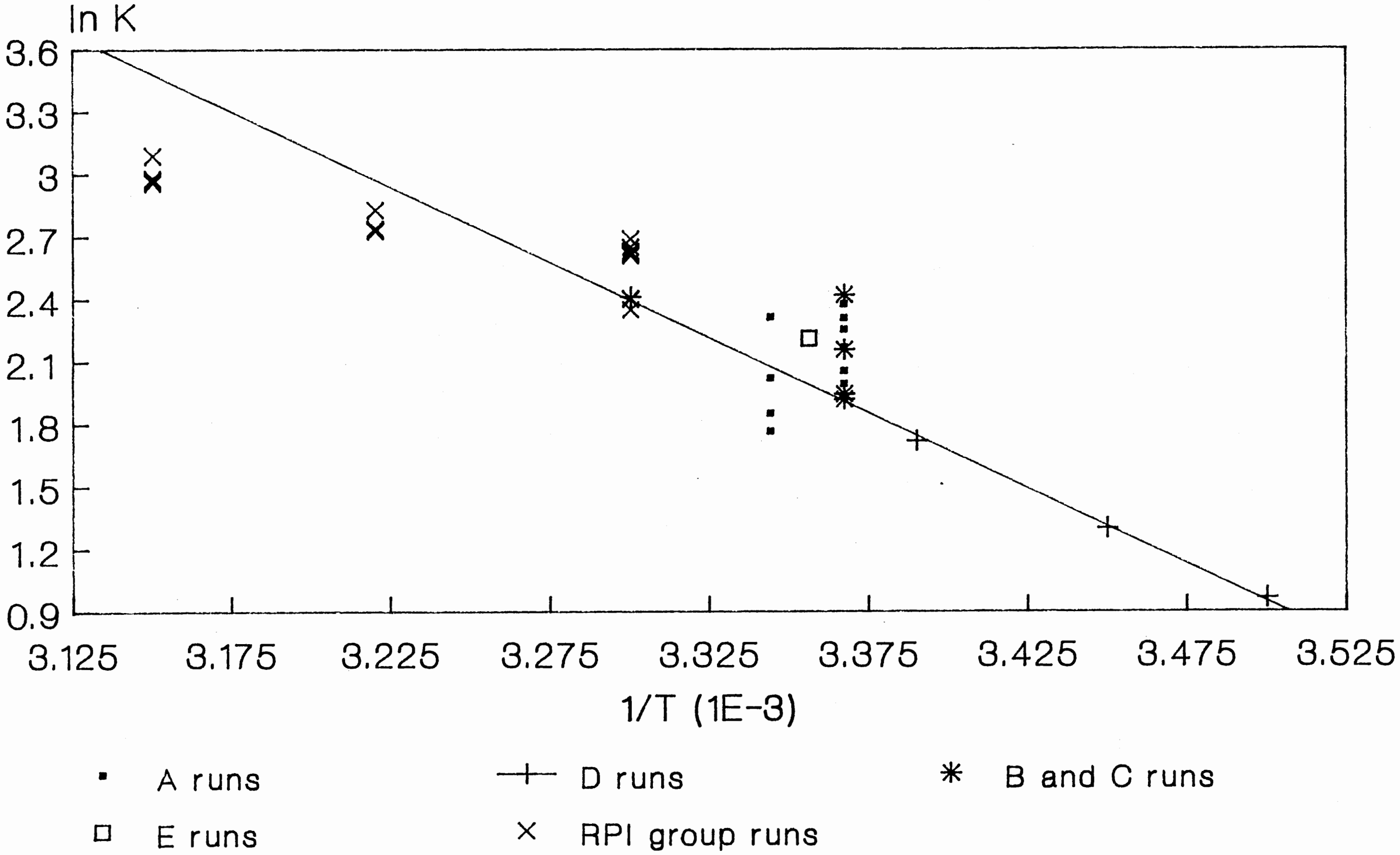

Figure 11. Arrhenius plot for batch runs

The plot shows that the values obtained from the D-runs give the best approximation of the temperature dependency of the rate constant. Based on this observation, a linear regression was made on the D-run data, the summary of which is given in Table IV.

From this regression,

$R^2 = 0.9989$

$a = 26.3228$ (intercept)

$b = -7250.4955$ (slope)

From,

$$\ln K = \ln K_0 - E/RT$$

using the above parameters,

$$\ln K = 26.3228 - 7250.496/T$$

or

$$K = 2.703 \times 10^{11} \exp(-7250.50/T)$$

This is the predicted equation for the temperature dependency of the rate constant of this experiment. It was used in calculation of K for further investigations in this work.

For R = 1.987 cal/mol.K, the activation energy (E) for this reaction is calculated to be 14406.7 cal.

At a room temperature of 25°C, the rate constant

$K = 7.33$ L/mol min and the pseudo first order rate constant $K' = 0.293$ min^{-1}.

Asfour (20) obtained $K' = 0.2$ min^{-1} while Hudgins and Cayrol (19) obtained $K' = 0.54$ min^{-1}. The group at Rensselaer Polytechnic Institute obtained $K' = 0.247$ min^{-1}. The results of the RPI group are given in Table V.

TABLE V

RESULTS FROM R.P.I GROUP

Run B.1		Run B.2		Run B.3		Run B.4	
T=30^{o} C		T=20^{o} C		T=38^{o} C		T=45^{o} C	
1/T=3.3x10^{-3}		1/T=3.3x10^{-3}		1/T=3.22x10^{-3}		1/T=3.15x10^{-3}	
K	ln K	K	ln K	K	ln K	K	ln K
13.6378	2.61	10.4872	2.35	15.5336	2.74	21.9781	3.09
13.8755	2.630	11.0206	2.40	15.2578	2.73	19.5867	2.98
13.8629	2.629	11.0691	2.404	16.8822	2.83	19.4946	2.97
13.7010	2.62	14.0780	2.65	15.4389	2.737	19.2834	2.96

CHAPTER IV

EXPERIMENTAL FLOW SYSTEM AND PROCEDURE

Description of Experimental Apparatus

A schematic diagram of the proposed experimental set-up for this work is given in Figure 12. The apparatus comprises the following:

1. Reservoirs: Two 50-L polyethylene tanks labelled TK1 and TK2 for dye and sodium hydroxide solutions, respectively.

2. Pumps: Two 1/3 hp, model D-11, centrifugal pumps, manufactured by Eastern Pump Industries, labelled P1 and P2 for dye and sodium hydroxide, respectively.

3. Rotameters: Two rotameters and a flow indicator labelled R1, R2 and R3 were used in this experiment. R1 is the dye flow meter, Fischer & Porter model # 10A6131N, with a stainless steel float and a maximum flow rate of 1250 ml/min. R2 is the sodium hydroxide flow meter, Fischer & Porter model # 10A6132N, with a stainless steel float and a maximum flow rate of 1340 ml/min. R3 is a flow indicator made by Matheson Gas Products with maximum flow of 1300 ml/min and a stainless steel float.

4. Reactor: The reactor was made of 50 ft of 1/4 in i.d Teflon tubing, wound on a 4 in diameter PVC pipe 3 ft in length. The tubings were purchased from Cole-Parmer Instrument Company. For further investigation in this work, the reactor configuration and orientations were changed. 50 ft of 1/4 in i.d Teflon tubing was used with 2 in

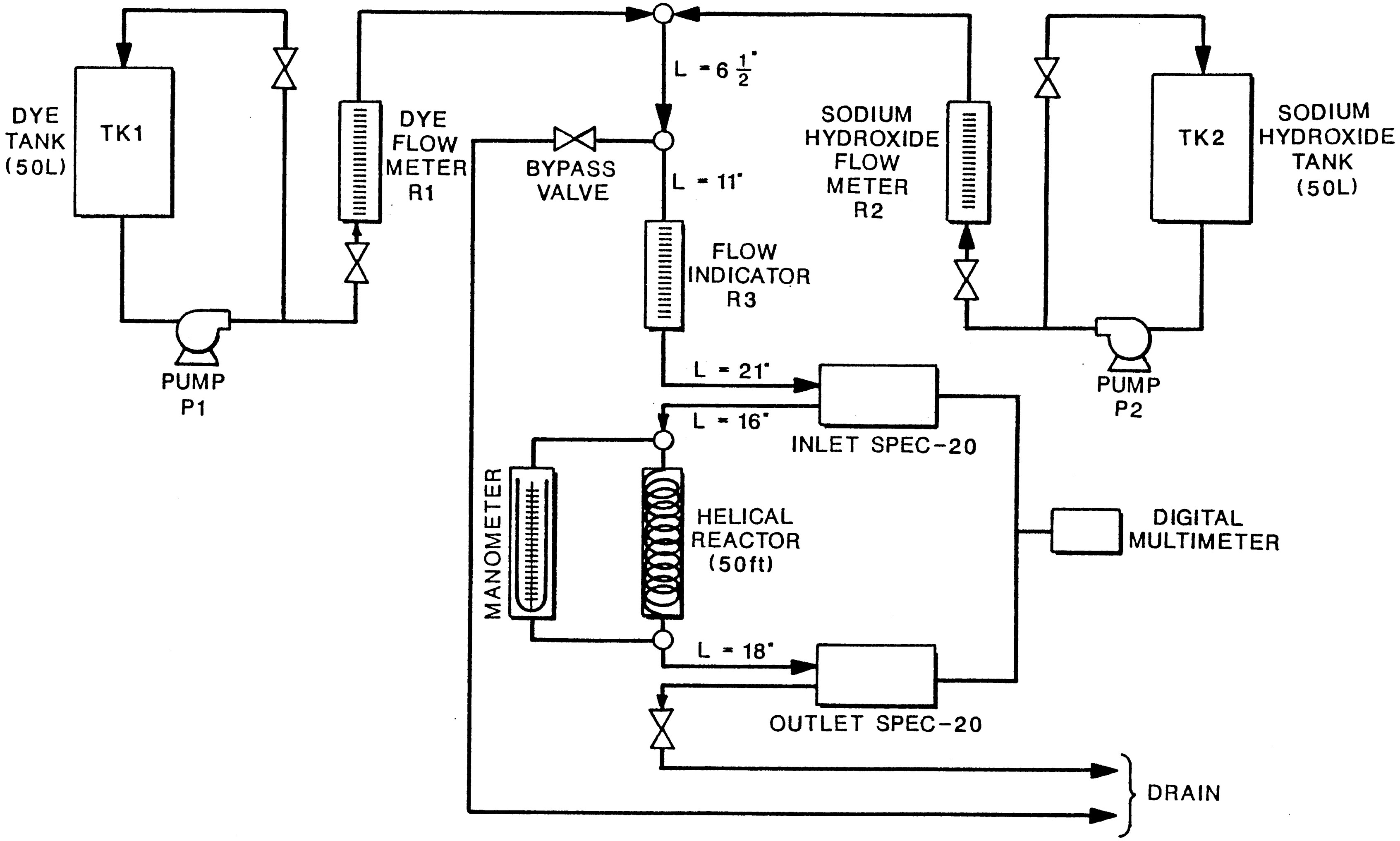

Figure 12. Schematic diagram of experimental set-up.

diameter PVC pipes; 50 ft of 1/8 in i.d polyethylene tubing, also purchased from Cole-Parmer were used with both 4 in and 2 in PVC pipes. The reactor could be kept in a vertical or horizontal position.

5. Manometer: A 50 in differential manometer was used to measure pressure drop across the reactor.

6. Spectrophotometers: Two Spectronic 20's, made by Bausch & Lomb and Milton Roy, were used to take the inlet and outlet sample readings.

7. Valves: Needle valves were used to adjust the flows.

The two pumps P1 and P2 were used mainly to pump the reactants into the system through rotameters R1 and R2 during experimentation and also to recycle the reactants back to the tanks to insure perfect mixing before the start of the experiment. The reactant streams mix in a tee-joint, go through the flow indicator R3 and flow into the inlet Spec-20 for an inlet sample reading before going into the reactor. The tubular reactor was in the form of a helical coil wound on PVC support and the flow exiting the reactor went through another spectrometer for outlet sampling. The manometer was connected from the inlet and outlet points of the reactor and read the pressure drop in the reactor.

In this experimental set-up, some design features were different from that used by previous investigators.

Hudgins and Cayrol (19) and Asfour (20) used reservoirs and constant head tanks. In this work, reservoirs and pumps were being used. This is mainly due to convenience since headroom was limited in the room where the experiments were conducted.

The flow-through Spec-20 cell introduced by Asfour was modified to minimize dead volume by elongating the innertube to leave a little clearance at the bottom of the curvette. In addition, this enables the fluid in curvette to be well mixed at all times.

The question arose that this elongation which cut through the light path of the Spec-20, might result in lower transmittance values. To resolve this, the Spec-20 was calibrated with the modified flow cell.

The reaction mechanism for this experiment is a pseudo first order reaction. For this to be valid, the initial concentration of sodium hydroxide in the reactor (C_{Bo}) has to be in large excess of that of the dye (C_{Ao}). Previous investigators tried to achieve this by keeping a constant NaOH: dye flow rate ratio and varying both flow rates. Asfour used a ratio of 9:1. In this proposed set-up, two new features were used; a flow indicator before the reactor that showed the exact amount of solution going into the reactor and a bypass valve, before the flow indicator, controlling the amount that went into the reactor. The idea was to keep a 1:1 flow-rate ratio and concentration ratio of NaOH and dye through R1 and R2. Hence, C_{Bo} and C_{Ao} were kept constant through the experiment. With the aid of the bypass-valve, the residence time in the reactor can be varied while still keeping C_{Bo} and C_{Ao} constant.

Previous investigators have reported the use of tygon tubing for the reactor. The obvious change in color of the tubing to a deep purple is an indication that the dye reacts with the tube. This may affect the results of the experiment. In this work teflon and polyethylene tubings were used which have higher resistance to dye absorption on the tubing wall.

In previous work, part of the solution going to the reactor was taken for sampling. In this work the entire flow passes through the

Spec-20's and the length of tubing between the Spec-20's and the reactor inlet and outlet was kept very short (about 1 ft).

In place of mixers in the reservoir tanks, recycle lines were provided for the tanks and the reactants were recycled with the pumps for a long period of time before the start of the experiment to insure good mixing. This design is more reliable and requires less total energy.

It was intended that all these modifications in design would lead to experimental results that are more representative and have less error than that of previous investigators.

Sizing and Design Specifications

Since this equipment was also being designed for use in an undergraduate unit operations laboratory, one of the primary goals was to size it to run long enough for a lab session. Therefore, continuous operation time was chosen to be 150 mins (2 1/2 hrs).

Two 50-L reservoirs were available for the reactants storage. Hence the design parameters were determined based on this capacity.

Some inherent assumptions were made in determining the design specifications and operating conditions.

1) The reaction mechanism was assumed to be pseudo-first order with $C_{Bo} >> C_{Ao}$.

2) The dye and NaOH solutions were assumed to be dilute enough to have the same physical properties as water. Therefore, the viscosity of the solutions were assumed to be equal to that of water (1 centipoise or 6.7197×10^{-4} lbm/ft-s). Density of solutions were assumed to be equal to that of water (62.34 lbm/ft^3).

3) The solutions were assumed to be at constant density within the reactor which makes it possible to relate the holding time or mean residence time (τ) to the volume (V) and volumetric flow rate (v_o).

$$\tau = V/v_o$$

4) The temperature was assumed to be uniform throughout the reactor.

The reactor was sized for both 1/4 in i.d tubing and 1/8 in i.d tubing. It was intended that this investigation be done in the laminar flow regime, therefore for the 1/4 in i.d tubing, a Reynolds number range of 197 - 2000 was chosen. This corresponds to a flow rate range of about 58 - 600 ml/min.

Design calculations based on 1/4 in i.d tube:

(a). Dye concentration:

From,

$$X_A = \frac{C_{Ao} - C_A}{C_{Ao}} = 1 - \frac{C_A}{C_{Ao}} = 1 - \frac{[A]}{[A]_o}$$

and using different conversion values, a possible range of dye concentrations were determined. From the calibration curve, Ao was fixed at 1.6 to give a maximum initial molar concentration of dye in the system as 13×10^{-6} M. A value of 12×10^{-6} M was chosen as the optimum concentration in the reactor. Based on this, the concentration of dye required in the tank was determined as follows:

$$\frac{[C_A]_{tank} \times Q_{dye}}{Q_{dye} + Q_{NaOH}} \leq 12 \times 10^{-6}$$

For a design maximum flow rate of 600 ml/min, Q_{dye} = dye flow rate = 300 ml/min, Q_{NaOH} = NaOH flow rate = 300 ml/min on a 1:1 basis.

$$\frac{C_{A_{tank}} \times 300}{300 + 300} = 12 \times 10^{-6}$$

$$\Rightarrow C_{A_{tank}} = 2.4 \times 10^{-5} \text{ M}$$

Molecular weight of dye = 408 g/mole

Mass of dye required to make up a 2.4×10^{-5} M dye solution in a 50 L tank is given by,

$$2.4 \times 10^{-5} \frac{\text{moles}}{\text{L}} \times 50\text{L} \times 408\frac{\text{g}}{\text{mole}}$$

$$= 0.4896 \text{ grams}$$

or 0.49g in 50 liters

(b). NaOH Concentration:

For a simple first order reaction

$$A + B = \text{Products} \qquad (1)$$

the rate equation is given by,

$$-r_A = K\, C_A C_B \qquad (2)$$

Assuming pseudo-first order,

$$-r_A = K\, C_A C_{Bo} \qquad (3)$$

$$K\, C_{Bo} = K' \qquad (4)$$

The first order expression can be expressed in integral form as,

$$K' = -\ln(1-X_A) \qquad (5)$$

Assume $X_A = 0.9$

$$K' = -\ln(1-.9) = 2.3026$$

At Reynolds number 1970, $\tau = 0.834$ min. and at 197, $\tau = 8.34$ min. units

From the batch expression, at room temperature, ($T = 25^{o}$C) K = 7.33 L/mol min

$K'_{@8.34} = 0.2761 \text{ min}^{-1}$

$K'_{@0.834} = 2.7609 \text{ min}^{-1}$

From equation (4),

$K' = 7.33\, C_{Bo} = 0.2761 \text{ min}^{-1}$

$\Rightarrow C_{Bo} = 0.0377 = 0.04 \text{ M}$

This is referred to as the mixing cup concentration of NaOH and it is expected to be approximately constant throughout the reactor.

The concentration of NaOH in tank was determined as follows,

$$\frac{C_{B_{tank}} \times Q_{NaOH}}{Q_{NaOH} + Q_{dye}} = C_{Bo}$$

$$\frac{C_{B_{tank}} \times 300}{600} = 0.04$$

$\Rightarrow C_{B_{tank}} = 0.08 \text{ M}$

Molecular weight of NaOH = 40 g/mole

Thus the mass of NaOH required to make a 0.08 M solution in a 50-L tank is given by,

$$0.08\text{M} \times 50\text{L} \times \frac{40\text{g}}{\text{mole}} = 160 \text{ grams}$$

The C_{Ao}: C_{Bo} ratio is found to be 1:3333 which is sufficient for the pseudo-first order assumption to hold.

Calculations for 1/8 in i.d tubes were made with the same procedure.

Calibration of Spec-20's and Rotameters for Continuous Flow

As mentioned in description of experimental apparatus the flow cell design for the spectrometers was modified to eliminate dead volume and enhance proper mixing of the fluid in the cell curvette. As a result of this, the Spec-20's were recalibrated for continuous flow operation.

The dye solution was found to be light sensitive which affects the Spec-20 reading. The cell compartment was covered with a black material and paper foil to restrict the interference by light in the laboratory. Vibrations can also interfere with the reading. Connections were made from the analog output of the Spec-20's to a digital multimeter. With the aid of a switch, the inlet and outlet Spec-20 readings were made.

Calibration curves using the modified flow cell for the Spec-20's are shown in Figures 13 and 14.

The rotameters were also calibrated with the stainless steel float balls sitting on the graduation mark. The curves for the rotameter calibrations are shown in Figures 15, 16 and 17.

Preparation of Solutions

As per design specifications, 2.4×10^{-5} M dye solution was made up in the dye tank and 0.08 M sodium hydroxide solution was made up in the NaOH tank.

To make the dye solution, 0.49 g of crystal violet dye was dissolved in 50 L of deionized water. The deionization was done using Barnstead Ultra-pure Cartridges. The conductivity of the water was measured with a conductivity meter. Water with conductivity in the range of 0.8 - 750 microhm/cm had been used at different times in the course of this work. There is no direct relationship between the performance of the experiment and the conductivity of water.

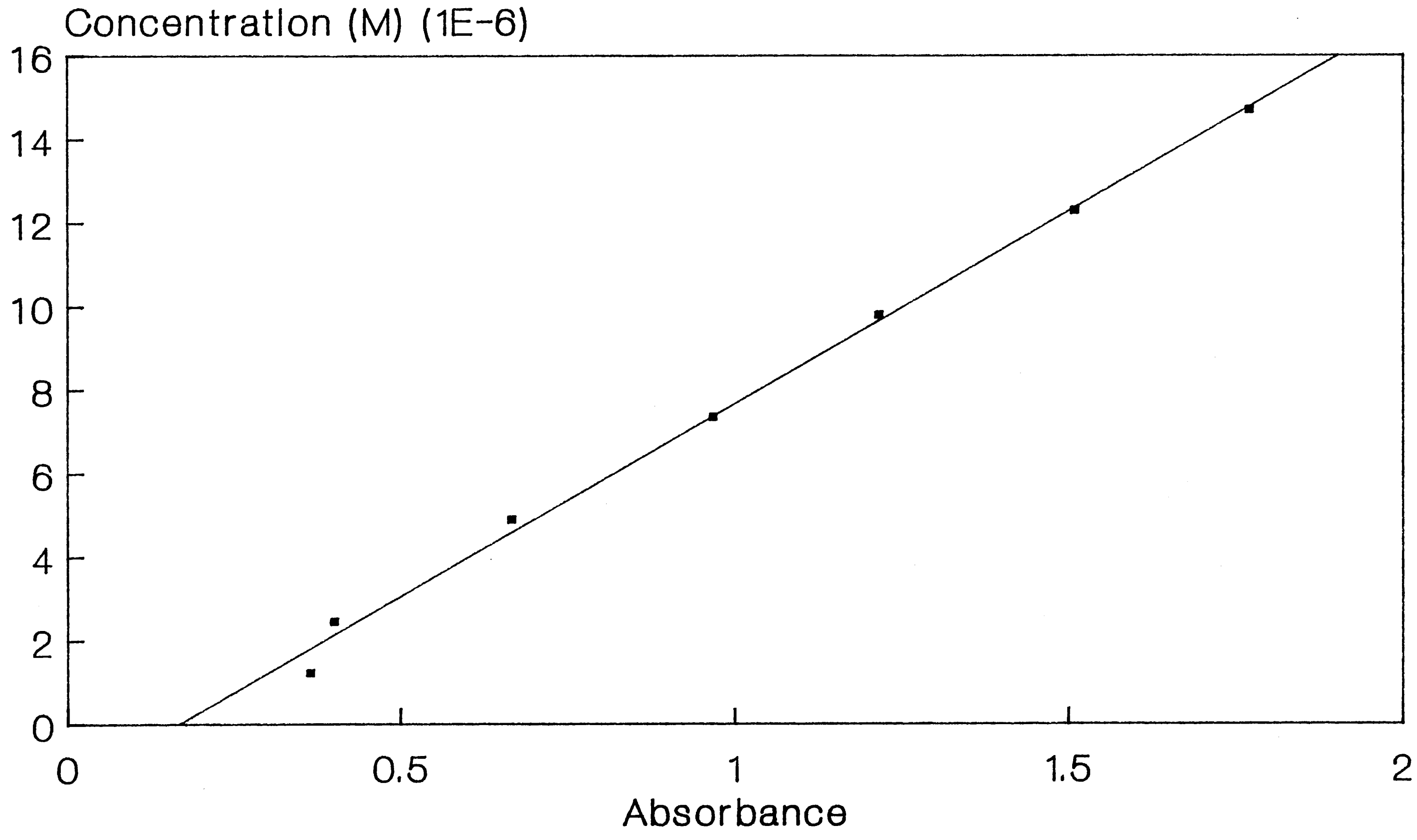

Figure 13. Inlet Spec-20 calibration

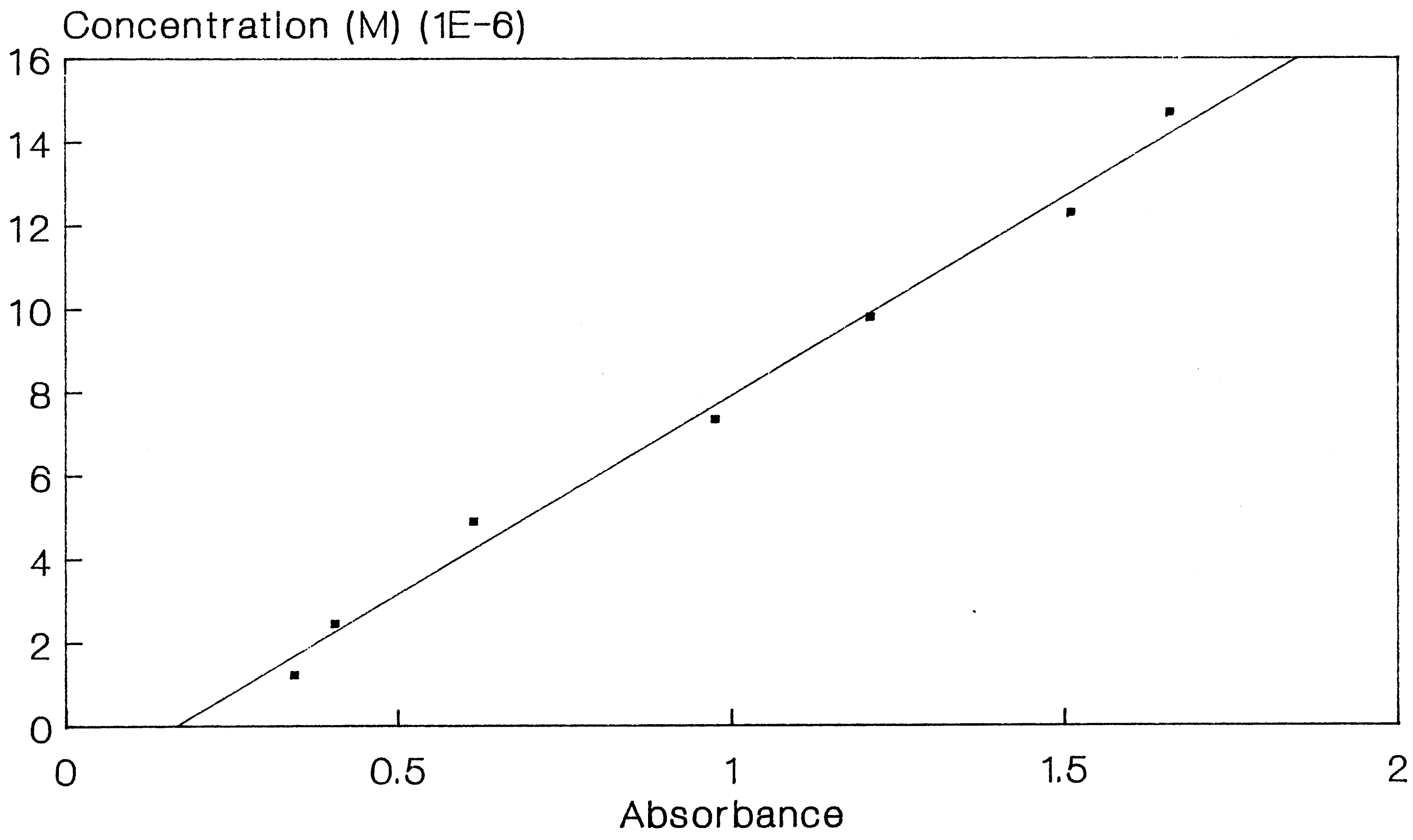

Figure 14. Outlet Spec-20 calibration

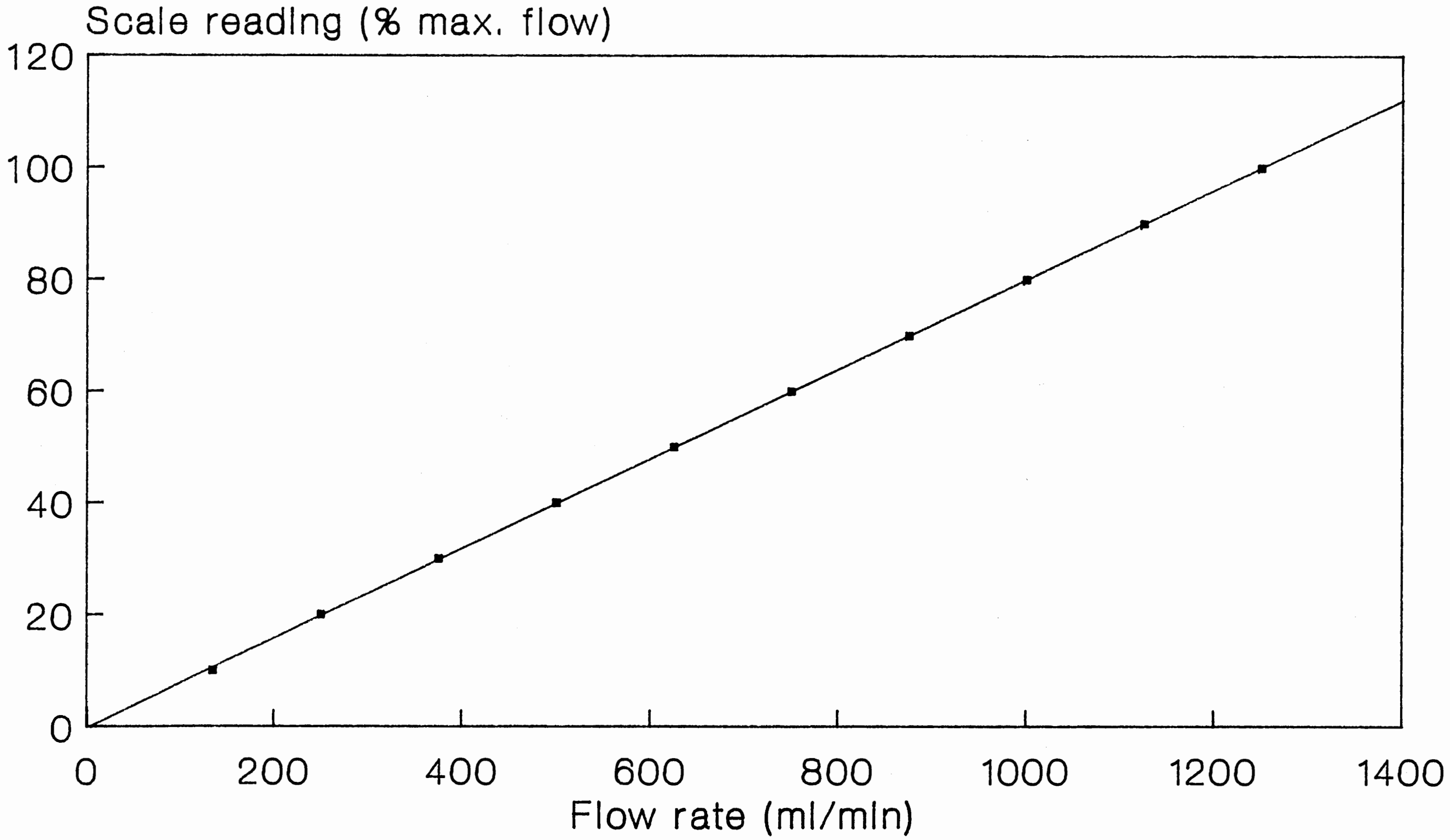

Figure 15. Calibration curve for R1

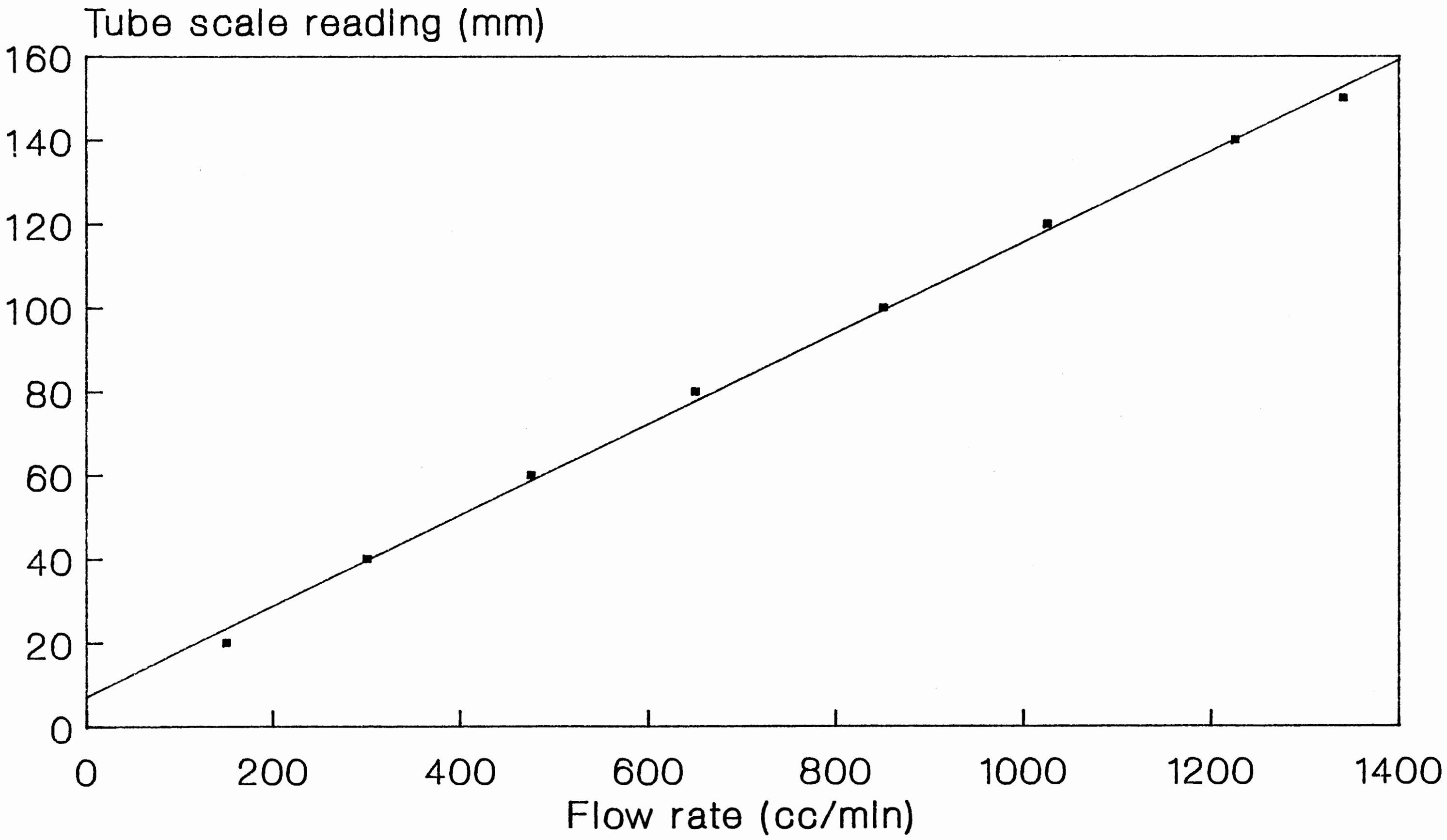

Figure 16. Calibration curve for R2

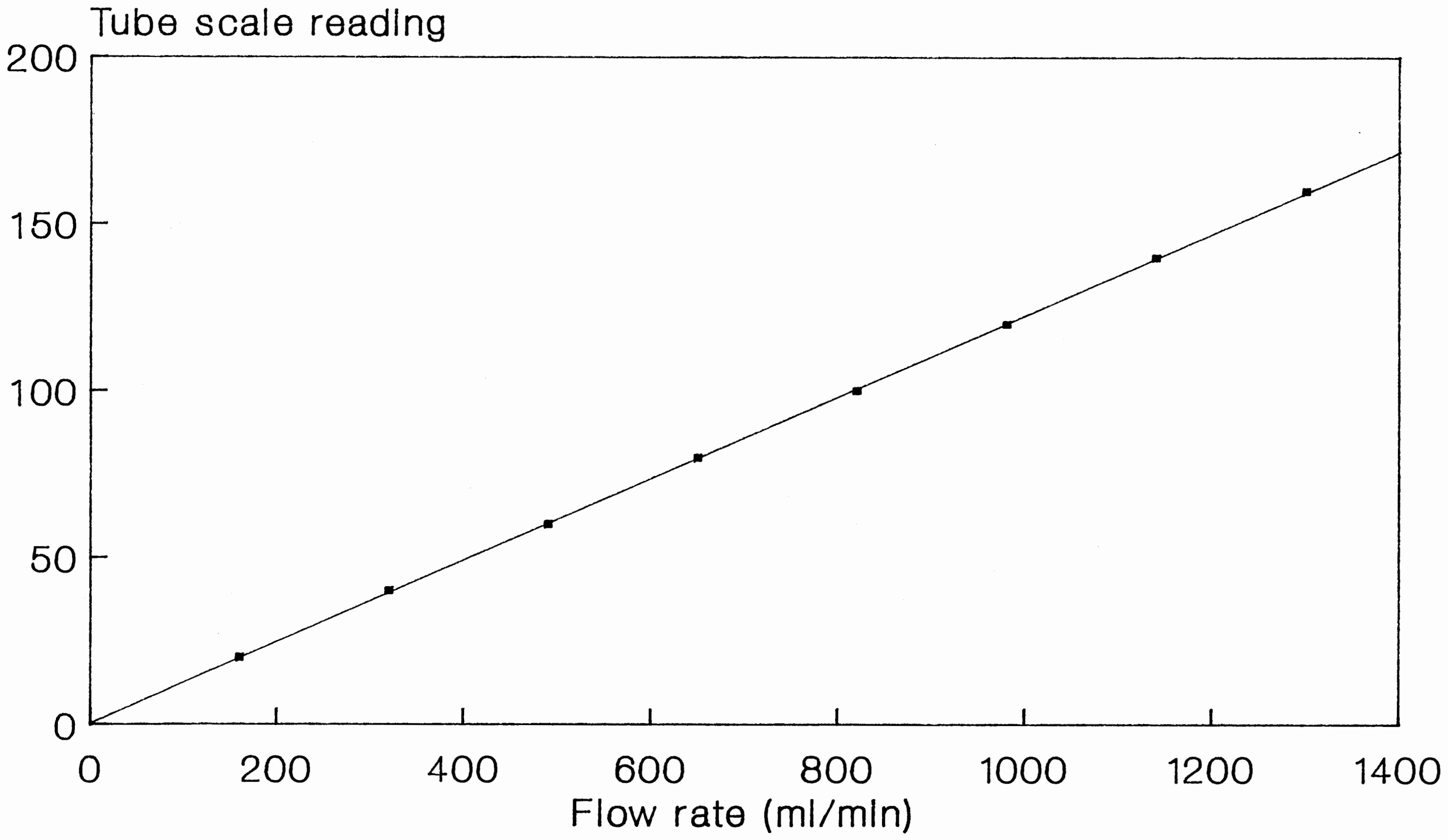

Figure 17. Calibration curve for R3

For the NaOH solutions, 160 g of NaOH was dissolved in 50 L of deionized water. The NaOH used was of the powder form and the measurements were done on the OHAUS balance.

The solutions were separately recycled for at least 30 mins before the start of the experimental runs.

Sampling Technique

The design time for this experiment is about 2 1/2 hrs and the capacity of each reservoir is 50 L. To meet the time demand, the rotameters for dye and NaOH were required to put 300 ml/min of reactants into the system at all times. The recycle lines were shut during runs. When the bypass valve was opened to obtain runs at lower residence time, the pressure drop across the reactor was observed to fall and in order to meet the pressure drop demands, the rotameter readings in the dye and NaOH lines went up. The rotameters were set back to give 300 ml/min each time the residence time in the reactor was altered. For the dye rotameter, the scale reading corresponding to 300 ml/min is 24 and for the NaOH rotameter, it is 40. Bubbles are often noticed after flow rate changes using the bypass valve. These were allowed to leave the system before any readings were taken.

As a rule of thumb, it is expected that steady state is reached when 3 times the reactor volume amount of reactants go through the reactor. However, at high flow rates, steady-state is found to be attained quicker than at low flow rates. As are indicated in the experimental results, steady-state time for this work ranged from 5 mins at high flow rates to 25 mins at low flow rates. This was the time that elapsed, after flow changes were made, before inlet and outlet readings were taken. To monitor the approach to steady state in the reactor, inlet and outlet readings were taken at smaller time intervals and as steady state was approached, the readings were found to be consistent.

Experimental Procedures

The following procedure was employed in running the experiment.

(1) Dissolve 160 g NaOH in 50 L of deionized water.

(2) Dissolve 0.49 g crystal violet-dye in 50 L of deionized water.

(3) With the inlet valves to the reactor system closed and the recycle valves open; start pumps and recycle the reactants in the tanks for about 15-30 mins to ensure good mixing.

(4) Record room temperature, dye solution temperature and NaOH solution temperature.

(5) Warm up Spec-20's (about 15 mins) and zero the Spec-20's with NaOH solution flowing through the system.

(6) Open valve to let in dye solution to the reactor system and close the two recycle lines.

(7) Set R1, R2 and R3 to read the desired values using the bypass valve and the control knobs in R1 and R2. Any bubbles at this point should be allowed to leave the system before proceeding.

(8) Set stop watch and take inlet and outlet transmittance readings at desired time intervals. This enables one to monitor the change in C_A with time and see the development of steady-state.

(9) At steady-state, record inlet and outlet transmittance and pressure drop (inches of Hg).

(10) Change flow rate into reactor by opening the bypass valve and repeat 7-9.

The transmittance values obtained were converted to absorbance with the relation,

$$A = -\log_{10} T$$

For experiment,

$$X_A = \frac{Ao - A}{Ao} = \frac{C_{Ao} - C_A}{C_{Ao}}$$

For plug flow,

$$X_A = 1 - e^{-K'\tau}$$

where

$$K' = KC_{Bo}$$

and K was determined at the average temperature.

For Laminar flow,

$$X_A = 1 - \left(\frac{N_R}{2}\right)^2 E\left(\frac{N_R}{2}\right) + \left(\frac{N_R}{2} - 1\right) \exp^{(-N_R/2)}$$

where

$N_R = K'\tau$ and E(x) is the exponential integral available in mathematical handbooks (34). Reynolds number, $N_{Re} = \rho vD/\mu$ and $\tau = V/v_o$.

CHAPTER V

EXPERIMENTAL RESULTS FOR CONTINUOUS FLOW SYSTEM

The results from the final design of the continuous flow system will be presented in this chapter. A total of 14 runs were made at this stage and the runs were labeled to reflect the difference in purpose for which they were made. A general description is given for each class of runs including the objective for the runs.

Reproducibility of the results and error analysis are also discussed in this chapter after the data from the experiment.

The runs were made with 50 ft lengths of 1/4 in. i.d coil and 1/8 in. i.d coil wrapped around 4 in. diameter and 2 in. diameter P.V.C pipes (3 ft long). The final design included the use of flow cells with elongated inner tube to eliminate dead volume and enhance proper mixing of the reacting solution in the cell curvature. A diagram of this flow cell design is shown in Figure 18. The spectrophotometers were recalibrated for continuous flow based on this design. Figures 13 through 17 show the calibration curves for the spectrophotometers and the flow meters used in this experiment.

Since constant temperature baths were not used, the operating temperature for this experiment varied in accordance with the ambient temperature at time and day of run. However, all runs were made in a temperature range from 20°C to 27°C. The Reynolds number range for

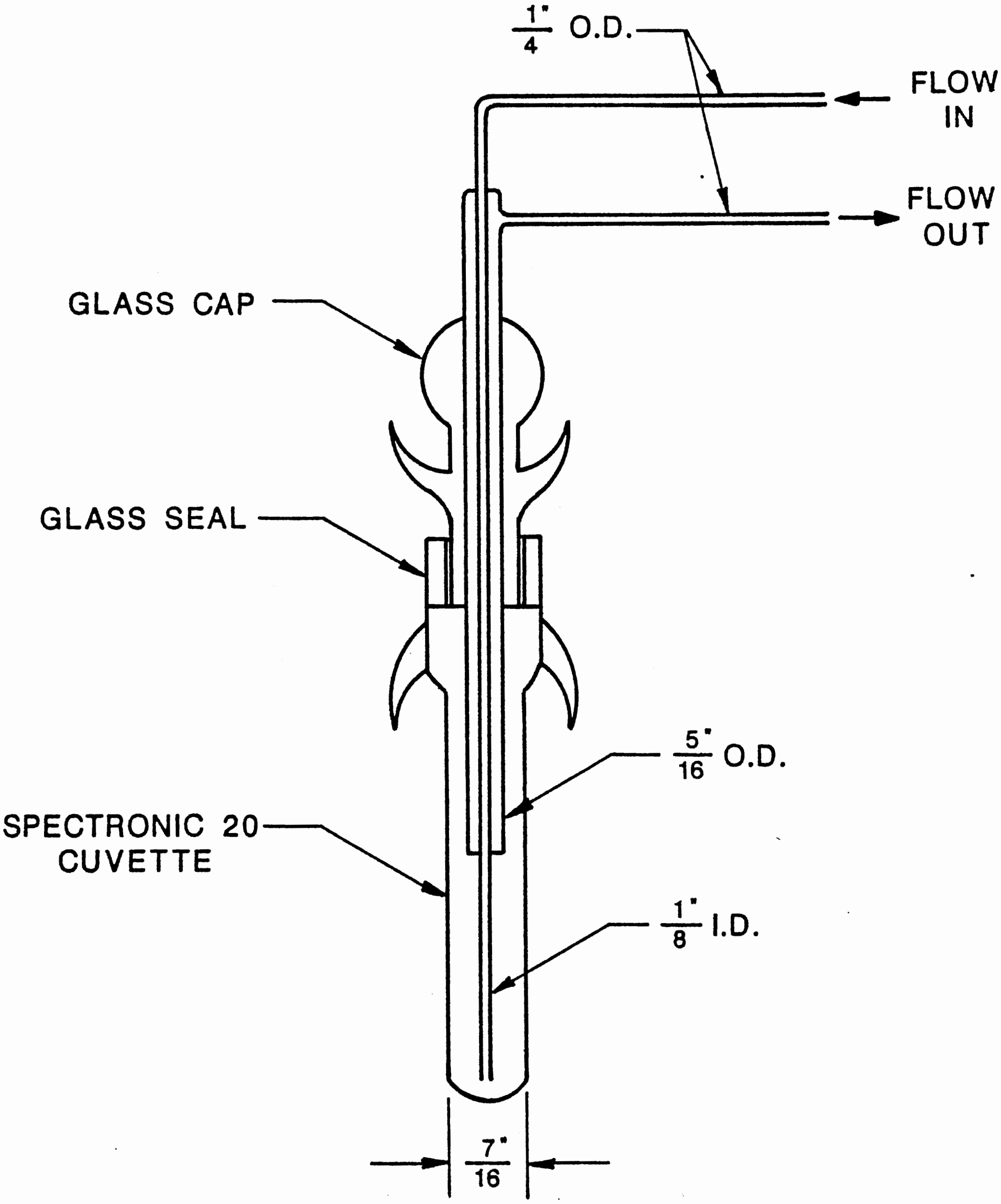

Figure 18. Flow-through cuvette for the Spectronic-20.

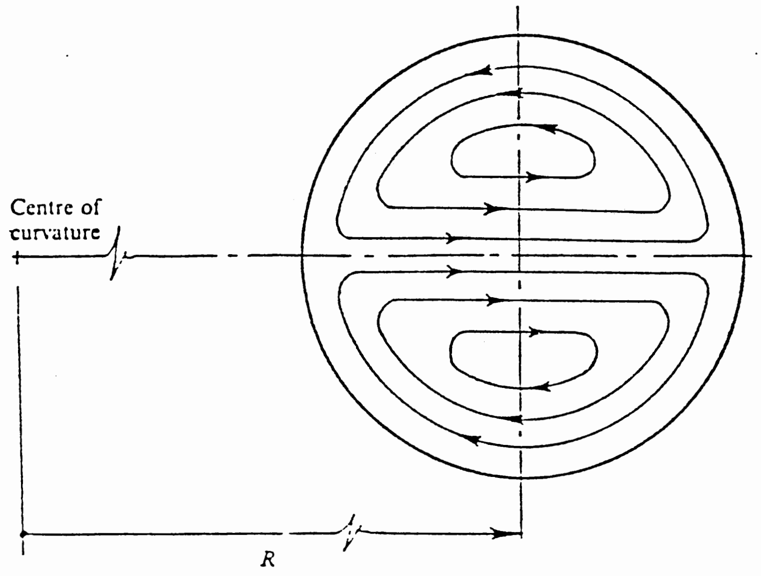

Figure 18a. Secondary Flow Pattern in Coiled Tubing.

which the runs were made are 331-2004 for 1/4 in. i.d coil and 166-1002 for 1/8 in. i.d coil. The Spec-20's were set to 100% transmittance with sodium hydroxide solution running through the system and the allowed steady-state time ranged from 5 mins for the highest flow rate to 30 min for the lowest flow rate. This was the time that elapsed before the inlet and outlet readings were recorded. However, at smaller time intervals, for each flow rate, inlet and outlet readings of the unsteady state concentrations were taken to monitor the development of steady state.

Z - Runs

The Z-runs were made with 50 ft length of 1/4 in. i.d coil wrapped on 4 in. i.d pipes placed in a horizontal position. Four runs namely Z-6, Z-7, Z-10 and Z-11 were made in this category.

In Z-6 and Z-7, the reacting solutions were run from the lowest flow rate to the highest flow rate while in Z-10 and Z-11, the reverse was the case. This was designed to obtain a stable method of operation. It was found that operating from highest to lowest flow rate was better in order to expel the air bubbles trapped in the system when it was not operating.

The results for the Z-runs are given in Tables VI to IX with the corresponding calculated conversion values for plug flow tubular reactor and laminar flow tubular reactor.

Plots showing comparison of conversions from experiment, plug flow and laminar flow reactors are shown in Figures 19 to 22.

The conversions (X_A) were obtained thus:

$$\text{Expt:} \quad X_A = \frac{A_{in} - A_{out}}{A_{in}}$$

TABLE VI

RUN Z-6

Room Temperature = 25^{o} C
NaOH Temperature = 26^{o} C
Dye Temperature = 20^{o} C

Date: 3-13-88
Conductivity = 750 micro ohm/cm

Q(ml/min)	T_{in}	T_{out}	A_{in}	A_{out}	ΔP(psi)	τ(min)	X_A EXPT	X_A PFTR	X_A LFTR
600	0.016	0.120	1.796	0.921	1.52	0.802	0.487	0.225	0.202
500	0.016	0.125	1.796	0.903	1.33	0.960	0.497	0.263	0.230
400	0.016	0.157	1.796	0.804	1.03	1.206	0.552	0.319	0.288
300	0.017	0.214	1.770	0.670	0.79	1.604	0.621	0.400	0.343
200	0.019	0.303	1.721	0.519	0.59	2.394	0.698	0.533	0.475
100	0.023	0.481	1.638	0.318	0.47	4.857	0.806	0.787	0.710

TABLE VII

RUN Z-7

Room Temperature = 25° C
NaOH Temperature = 26° C
Dye Temperature = 20° C

Date: 3-13-88
Conductivity = 750 micro ohm/cm

Q(ml/min)	T_{in}	T_{out}	A_{in}	A_{out}	ΔP(psi)	τ(min)	X_A EXPT	X_A PFTR	X_A LFTR
600	0.017	0.100	1.770	1.000	1.47	0.802	0.4350	0.225	0.202
500	0.015	0.112	1.824	0.951	1.28	0.96	0.479	0.263	0.230
400	0.015	0.146	1.824	0.836	1.03	1.206	0.542	0.319	0.288
300	0.016	0.206	1.796	0.686	0.79	1.604	0.618	0.400	0.343
200	0.019	0.310	1.721	0.509	0.59	2.394	0.704	0.533	0.475
100	0.023	0.478	1.638	0.321	0.47	4.857	0.804	0.787	0.710

TABLE VIII

RUN Z-10

Room Temperature = 25^{o} C
NaOH Temperature = 25^{o} C
Dye Temperature = 25^{o} C

Date: 3-21-88
Conductivity = 9 micro ohm/cm

Q(ml/min)	T_{in}	T_{out}	A_{in}	A_{out}	ΔP(psi)	τ(min)	X_A EXPT	X_A PFTR	X_A LFTR
600	0.063	0.222	1.201	0.654	1.52	0.802	0.455	0.210	0.190
500	0.071	0.274	1.149	0.562	1.33	0.96	0.511	0.245	0.217
400	0.071	0.317	1.149	0.499	1.03	1.206	0.566	0.298	0.272
300	0.080	0.433	1.097	0.364	0.79	1.604	0.668	0.375	0.328
200	0.091	0.611	1.041	0.214	0.59	2.394	0.794	0.504	0.432
100	0.123	0.824	0.910	0.084	0.49	4.857	0.908	0.759	0.670

TABLE IX

RUN Z-11

Room Temperature = 26° C
NaOH Temperature = 26° C
Dye Temperature = 26° C

Date: 4-7-88
Conductivity = 780 micro ohm/cm

Q(ml/min)	T_{in}	T_{out}	A_{in}	A_{out}	ΔP(psi)	τ(min)	X_A EXPT	X_A PFTR	X_A LFTR
600	0.096	0.231	1.0177	0.6364	1.52	0.802	0.3747	0.242	0.215
500	0.102	0.268	0.9914	0.5719	1.33	0.960	0.4232	0.282	0.259
400	0.106	0.314	0.9747	0.5031	1.03	1.206	0.4839	0.340	0.304
300	0.094	0.360	1.0269	0.4437	0.79	1.604	0.5679	0.425	0.382
200	0.115	0.522	0.9393	0.2823	0.49	2.394	0.6994	0.562	0.492
100	0.158	0.752	0.8013	0.1238	0.39	4.857	0.8455	0.813	0.712

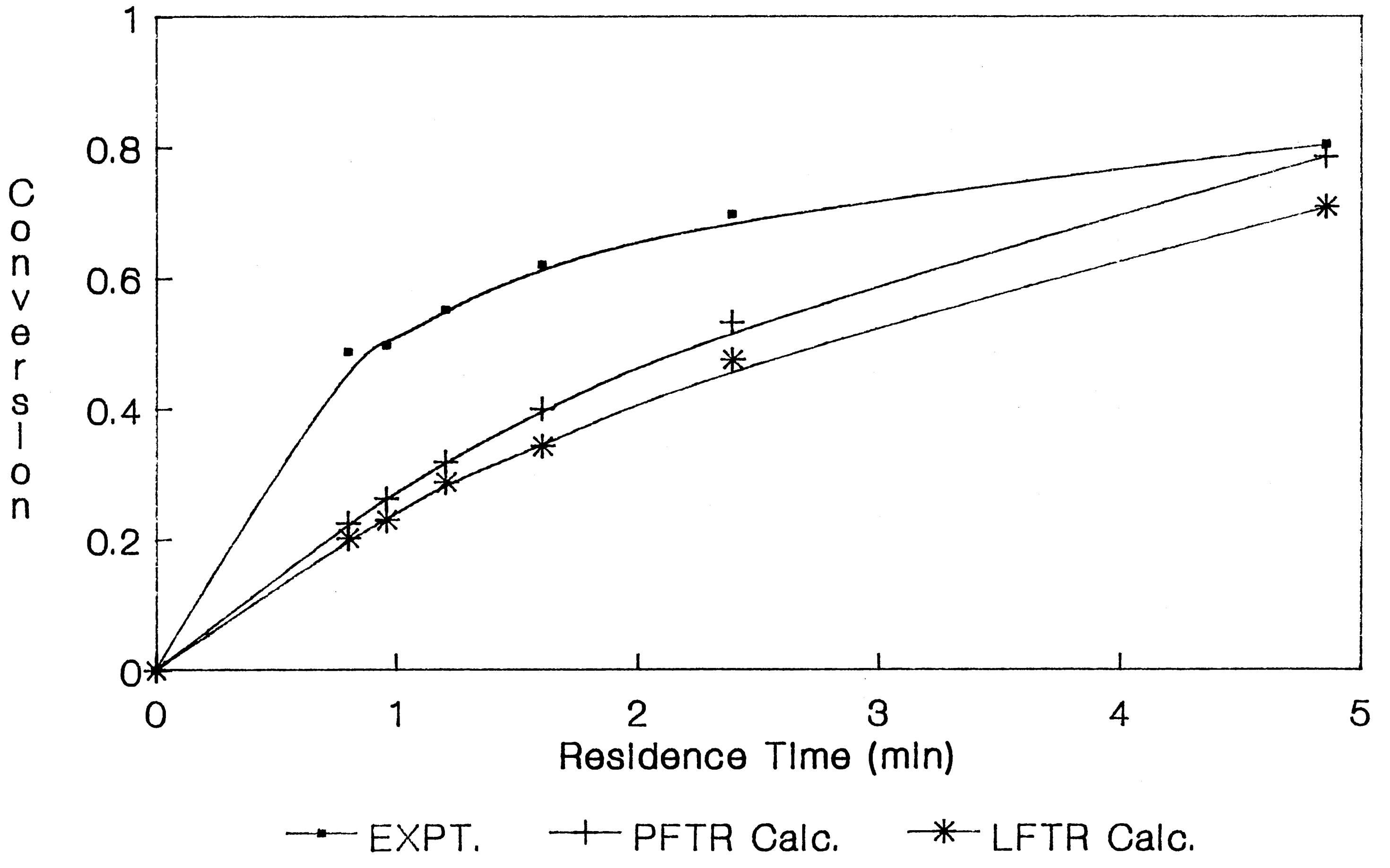

Figure 19. Reactor Models for Run Z-6.

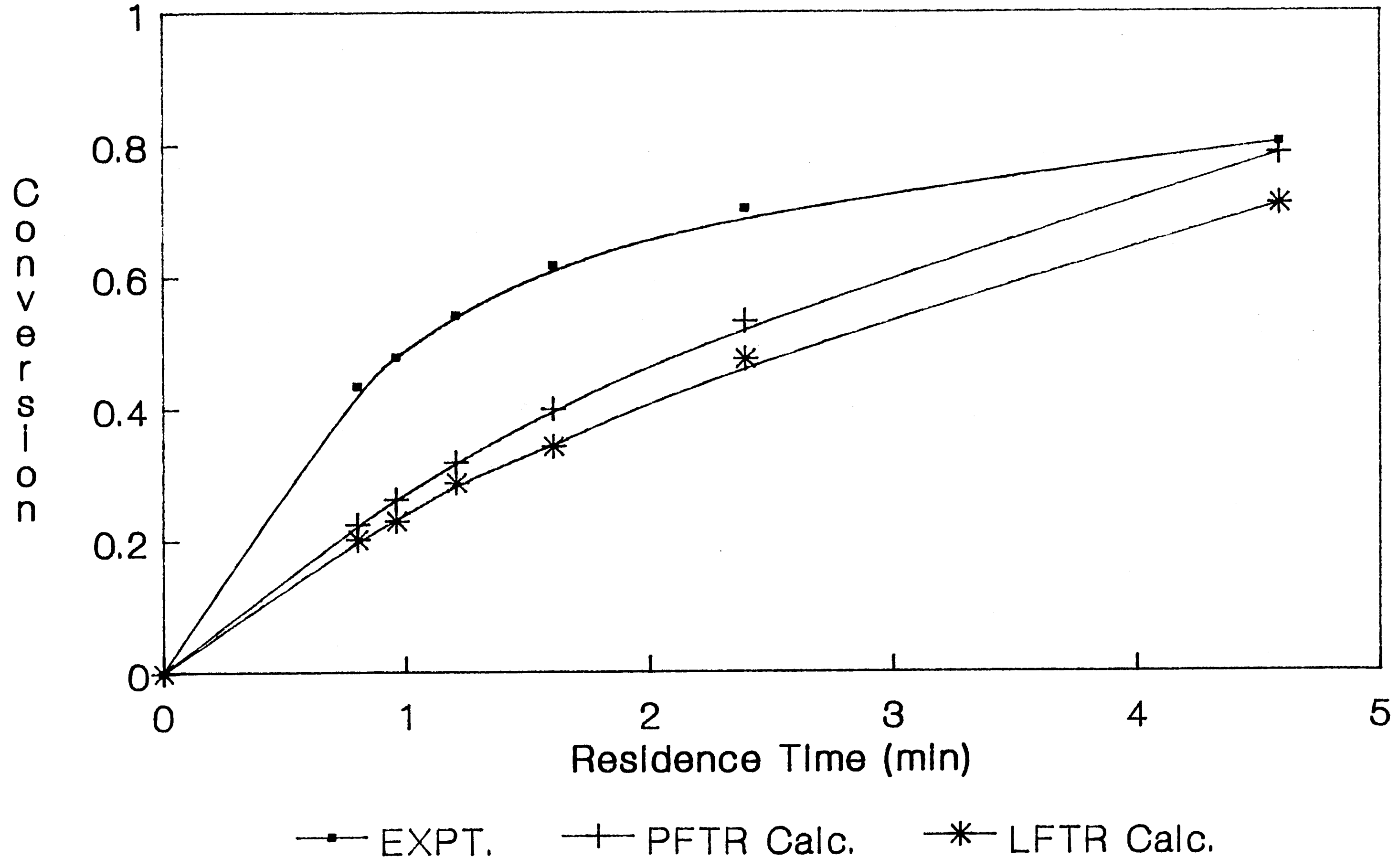

Figure 20. Reactor Models for Run Z-7.

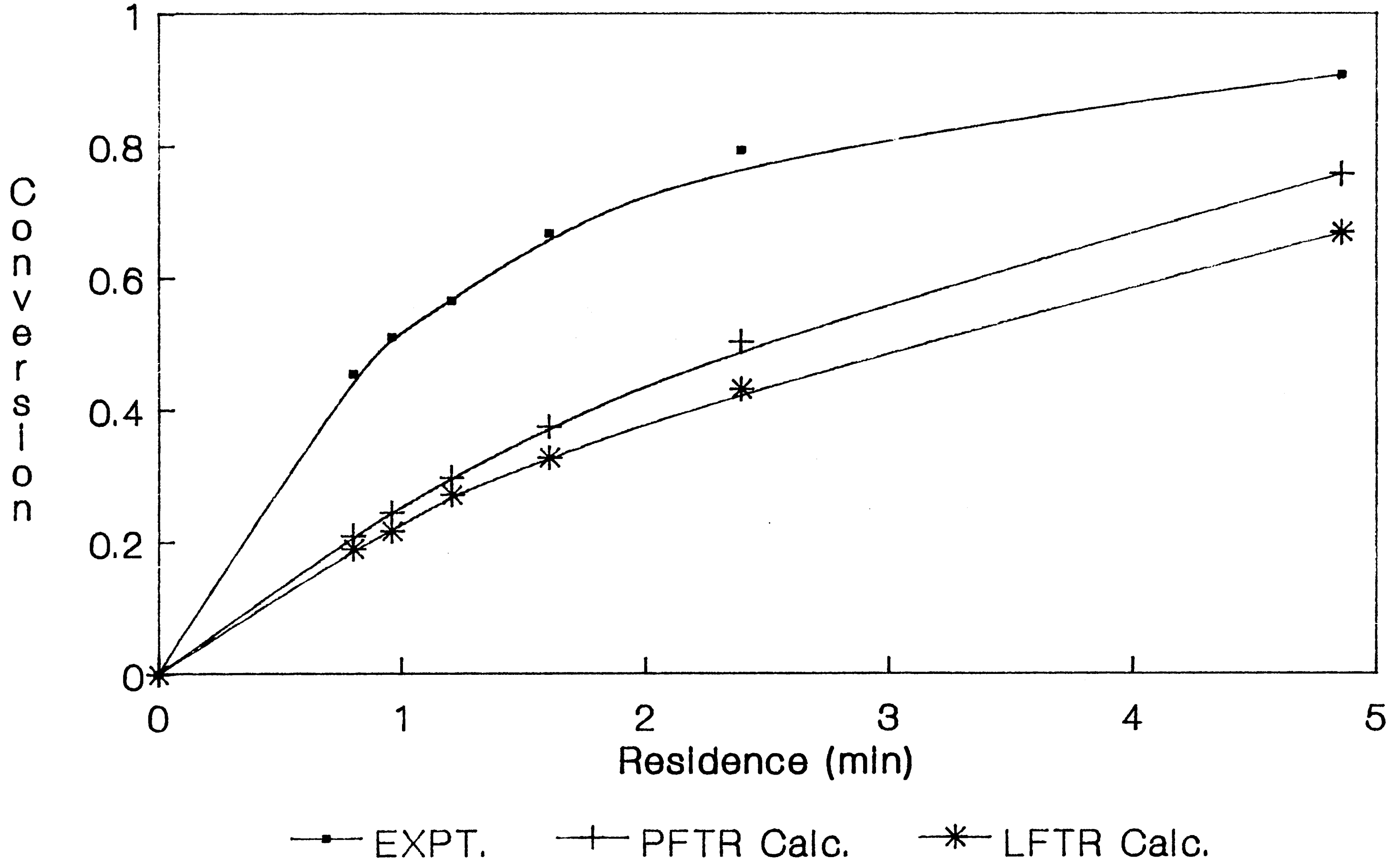

Figure 21. Reactor Models for Run Z-10

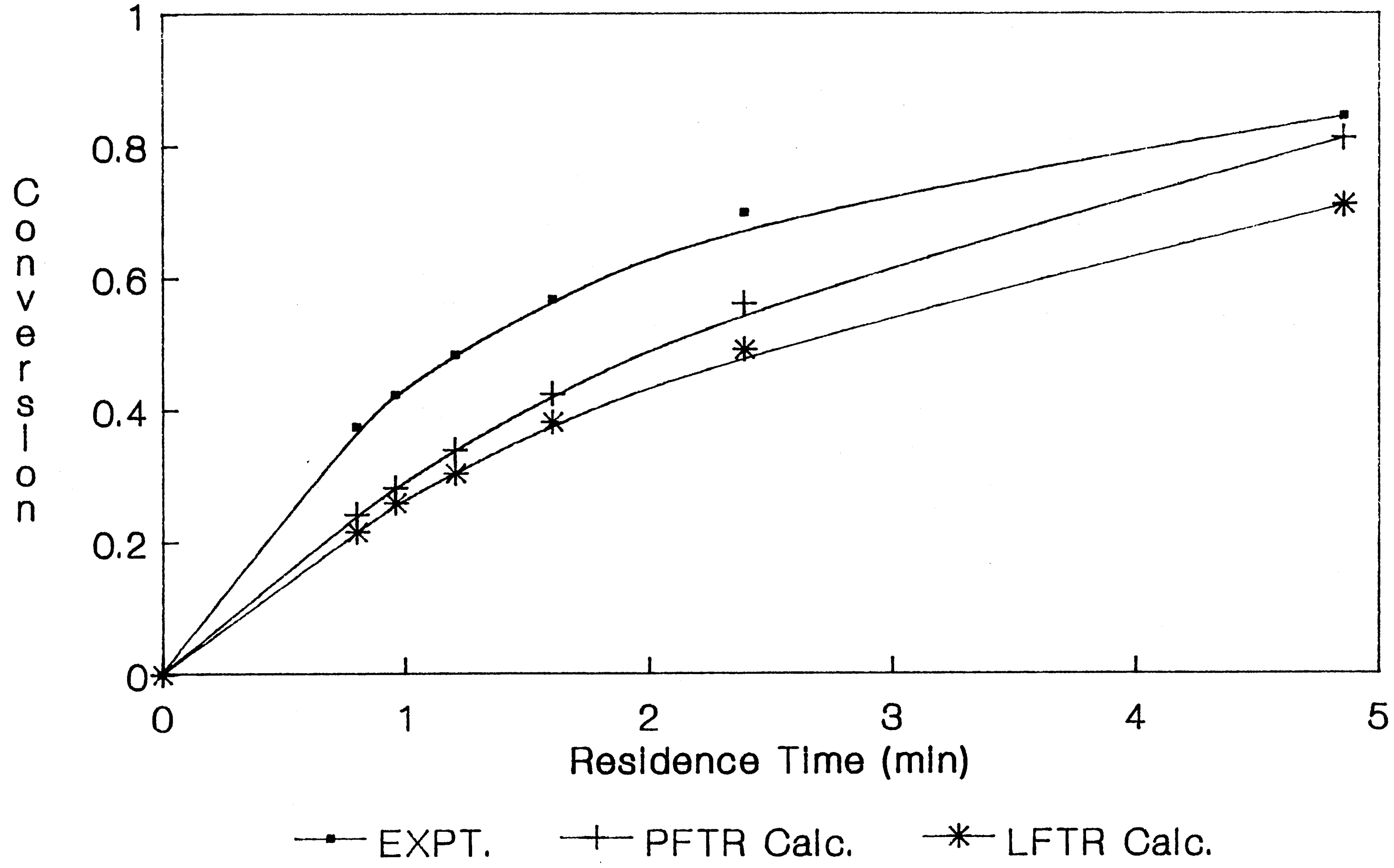

Figure 22. Reactor Models for Run Z-11

PFTR: $X_A = 1 - e^{-K'\tau}$

where, K' = the reaction rate constant was obtained from the Arrhenius equation in Chapter III at the operating temperature

LFTR: $X_A = 1 - \left(\frac{N_R}{2}\right)^2 E\left(\frac{N_R}{2}\right) + \left(\frac{N_R}{2} - 1\right) \exp\left(-N_R/2\right)$

where, $N_R = K'\tau$, $\tau = V/v_0$

The function E(y) is defined by:

$$E(y) = \int_y^\infty \frac{\exp(-\phi)}{\phi} d\phi$$

and tabulated in standard tables (34) as $-E_i(-X)$.

From the plots, it was observed that the calculated values for the plug flow and laminar flow reactors were lower than the conversions obtained from experiment. But, according to literature, the plug flow reactors are supposed to have the highest possible conversions.

Since, the rate constant (K) is the only experimental value introduced into these calculations, from batch kinetics study, a parametric study was done to see the effect of error in the K values on the plug flow conversions.

This study showed that the rate constant obtained from batch experiment has to be more than 60% in error for the plug flow conversion to be more than experiment. This was considered unlikely, hence the decision to obtain experimentally, the conversions for laminar flow and possibly plug flow to see how they compare with experiments.

Secondly, a study of the reaction mechanism was made to explore the validity of the pseudo-first order assumption for this reaction. The result of this study is shown in a later section on analysis of data.

Run Z-10 was made with water of the lowest conductivity and the suggested effect is given in the section on reproducibility.

Run Z-11, though at a conductivity much higher than Z-10, was made as a duplicate to Z-10.

S-Runs

The S-runs were made with a 50 ft length of 1/4 in. i.d straight tube reactor at the same residence times as the Z-runs. This was designed to obtain, experimentally, the conversions for a laminar flow tubular reactor and compare with the calculated values in the Z-run tables. The X_A values for these runs are given in Tables X and XI. These values were found to be much higher than the calculated values and fit more to the helical coil results as expected.

Figure 23 shows the straight tube laminar flow experimental conversions in comparison with helical coil conversions.

These results gave support to the suspicions of the invalidity of the assumed reaction mechanism.

Q-Runs

The Q-runs were made with 50 ft length of 1/4 in. i.d coils around a 4 in. i.d pipe. They were made to investigate the effect of body forces or reactor orientation on the performance of the reactor. In these runs, the reactor was kept in a vertical upright position, as opposed to the horizontal position in the other runs.

Run Q-1 was for an upflow of reacting solutions while Q-2 was for a downflow of solutions. The results are given in Tables XII and XIII,

TABLE X

RUN S-1

Room Temperature = 25^{o} C
NaOH Temperature = 25^{o} C
Dye Temperature = 24^{o} C

Date: 3-30-88
Conductivity = 800 micro ohm/cm

Q(ml/min)	T_{in}	T_{out}	A_{in}	A_{out}	ΔP(psi)	τ(min)	EXPT(X_A)	S.S(min)
600	0.290	0.490	0.5376	0.3098	1.13	0.802	0.4237	10
500	0.292	0.518	0.5346	0.2857	0.96	0.96	0.4657	15
400	0.316	0.600	0.5003	0.2218	0.79	1.206	0.5568	18
300	0.320	0.660	0.4949	0.1805	0.64	1.604	0.6353	20
200	0.336	0.764	0.4737	0.1169	0.49	2.394	0.7532	25
100	0.382	0.905	0.4179	0.0434	0.37	4.857	0.8963	30

TABLE XI

RUN S-2

Room Temperature = 24^{o} C
NaOH Temperature = 25^{o} C
Dye Temperature = 23^{o} C

Date: 4-13-88
Conductivity = 700 micro ohm/cm

Q(ml/min)	T_{in}	T_{out}	A_{in}	A_{out}	ΔP(psi)	τ(min)	EXPT(X_A)	S.S(min)
600	0.096	0.259	1.0177	0.5867	1.03	0.802	0.4235	10
500	0.096	0.281	1.0177	0.5513	0.93	0.960	0.4583	15
400	0.091	0.308	1.0410	0.5114	0.74	1.206	0.5087	18
300	0.111	0.397	0.9547	0.4012	0.49	1.604	0.5797	20
200	0.119	0.565	0.9245	0.2480	0.39	2.394	0.7318	25
100	0.168	0.815	0.7747	0.0888	0.29	4.857	0.8853	30

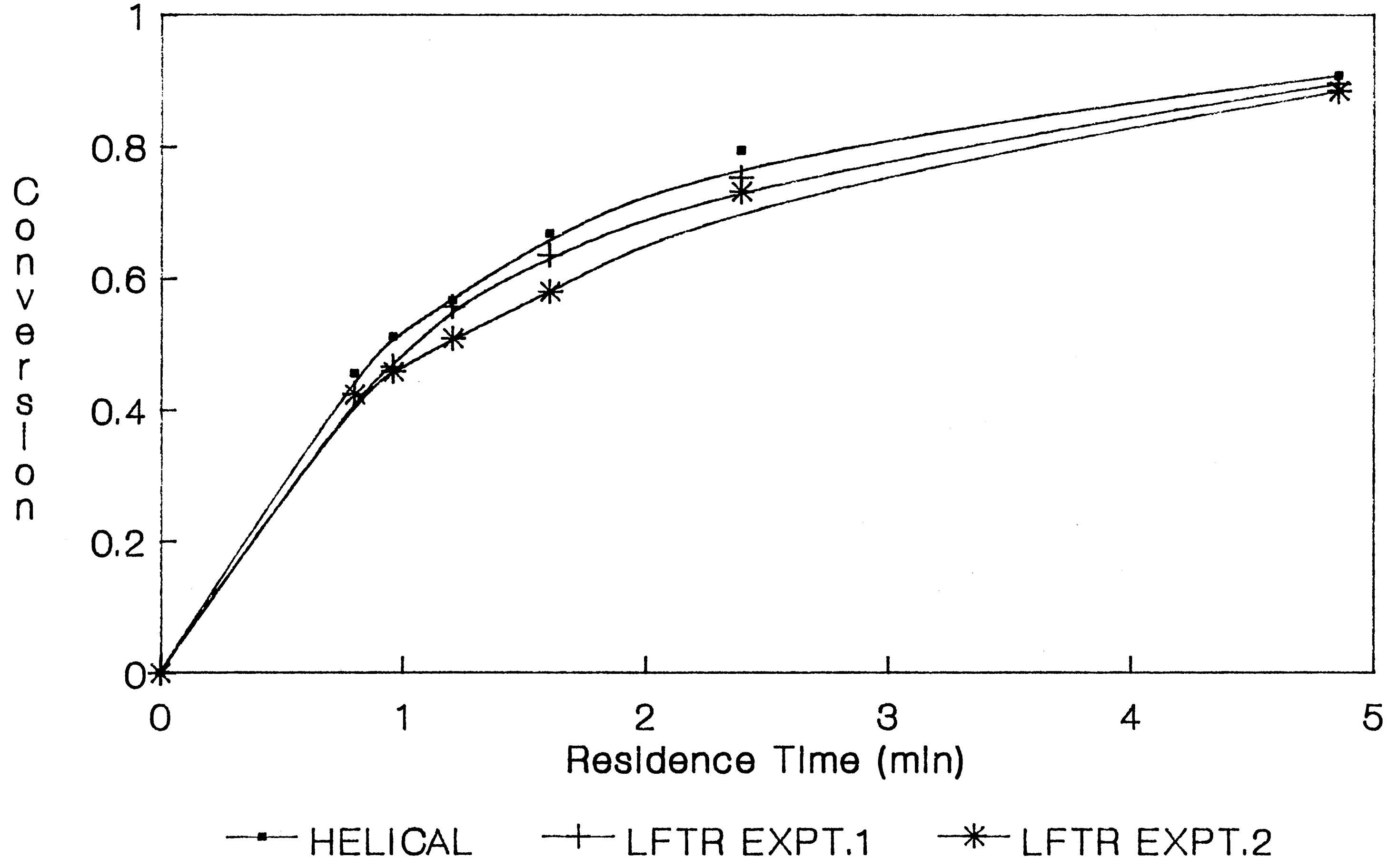

Figure 23. HELICAL vs. LFTR EXPT.

TABLE XII

RUN Q-1

Room Temperature = 27^o C
NaOH Temperature = 27^o C
Dye Temperature = 23^o C

Date: 4-5-88
Conductivity = 850 micro ohm/cm

Q(ml/min)	T_{in}	T_{out}	A_{in}	A_{out}	ΔP(psi)	τ(min)	EXPT(X_A)	S.S(min)
600	0.098	0.236	1.0088	0.6271	1.65	0.802	0.3784	10
500	0.118	0.306	0.9281	0.5143	1.33	0.960	0.4459	12
400	0.138	0.391	0.8601	0.4078	1.08	1.206	0.5259	15
300	0.165	0.525	0.7825	0.2798	0.79	1.604	0.6424	18
200	0.114	0.522	0.9431	0.2823	0.59	2.394	0.7006	20
100	0.168	0.793	0.7747	0.1007	0.39	4.857	0.8700	23

TABLE XIII

RUN Q-2

Room Temperature = 26° C
NaOH Temperature = 27° C
Dye Temperature = 26° C

Date: 4-6-88
Conductivity = 800 micro ohm/cm

Q(ml/min)	T_{in}	T_{out}	A_{in}	A_{out}	ΔP(psi)	τ(min)	EXPT(X_A)	S.S(min)
600	0.098	0.253	1.0088	0.5969	1.60	0.802	0.4083	10
500	0.101	0.290	0.9957	0.5376	1.03	0.960	0.4601	12
400	0.105	0.376	0.9788	0.4248	0.88	1.206	0.5660	15
300	0.116	0.383	0.9355	0.4168	0.79	1.604	0.5545	18
200	0.143	0.628	0.8447	0.2020	0.49	2.394	0.7608	20
100	0.193	0.791	0.7144	0.1018	0.37	4.857	0.8575	23

respectively, and a plot showing the comparison of the results to those of horizontal position is given in Figure 24.

P-Runs

The P-runs were carried out with 50 ft length of 1/4 in. i.d teflon tubes wrapped around 2 in. i.d pipe. The essence of these runs was to study the effect of change of radius of curvature on the reactor performance for the same residence time.

Two runs were made in this class and the results are given in Tables XIV and XV. A plot showing the effect of change in radius of curvature is shown in Figure 25.

K-Runs

The K-runs were made with 1/8 in. i.d teflon tubes wrapped around 4 in. i.d pipes. This was intended to investigate the effect of change of the reactor diameter and number of coils on the reactor performance. The suggested effects are discussed in the next chapter.

Two runs were made and the results are given in Tables XVI and XVII. A plot showing the effect of change in coil diameter is given in Figure 26.

J-Runs

In these runs, it was intended to study the effect of change in radius of curvature for 1/8 in. i.d coiled tubes. The tubes were wrapped around a 2 in. i.d pipe. The same length of tubes was used as in previously discussed results.

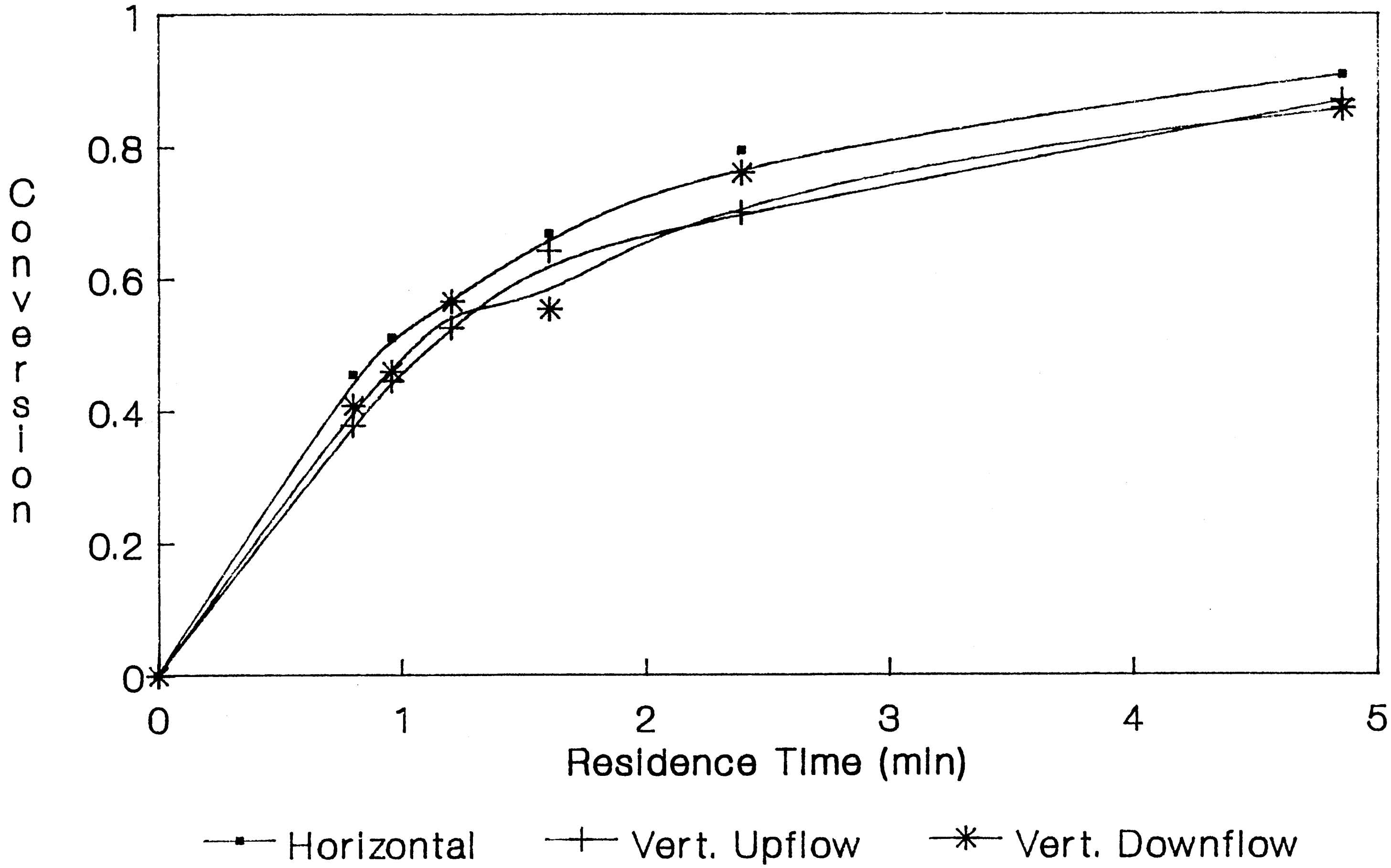

Figure 24. Effect of reactor position

TABLE XIV

RUN P-1

Room Temperature = 24^{o} C
NaOH Temperature = 23^{o} C
Dye Temperature = 23^{o} C

Date: 4-13-88
Conductivity = 730 micro ohm/cm

Q(ml/min)	T_{in}	T_{out}	A_{in}	A_{out}	ΔP(psi)	τ(min)	EXPT(X_A)	S.S(min)
600	0.081	0.216	1.0915	0.6655	2.41	0.802	0.3903	10
500	0.088	0.239	1.0555	0.6216	1.92	0.960	0.4111	15
400	0.093	0.255	1.0315	0.5935	1.52	0.4247	1.206	18
300	0.095	0.304	1.0223	0.5171	0.93	1.604	0.4941	20
200	0.106	0.457	0.9747	0.3401	0.54	2.394	0.6511	25
100	0.161	0.689	0.7932	0.1618	0.34	4.857	0.7960	30

TABLE XV

RUN P-2

Room Temperature = 24° C
NaOH Temperature = 26° C
Dye Temperature = 21° C

Date: 4-13-88
Conductivity = 730 micro ohm/cm

Q(ml/min)	T_{in}	T_{out}	A_{in}	A_{out}	ΔP(psi)	τ(min)	EXPT(X_A)	S.S(min)
600	0.090	0.231	1.0458	0.6364	2.50	0.802	0.3915	10
500	0.084	0.233	1.0757	0.6326	1.96	0.960	0.4119	15
400	0.092	0.259	1.0362	0.5867	1.50	1.206	0.4338	18
300	0.102	0.314	0.9914	0.5031	1.03	1.604	0.4926	20
200	0.121	0.456	0.9172	0.3410	0.59	2.394	0.6282	25
100	0.170	0.646	0.7696	0.1898	0.34	4.857	0.7534	30

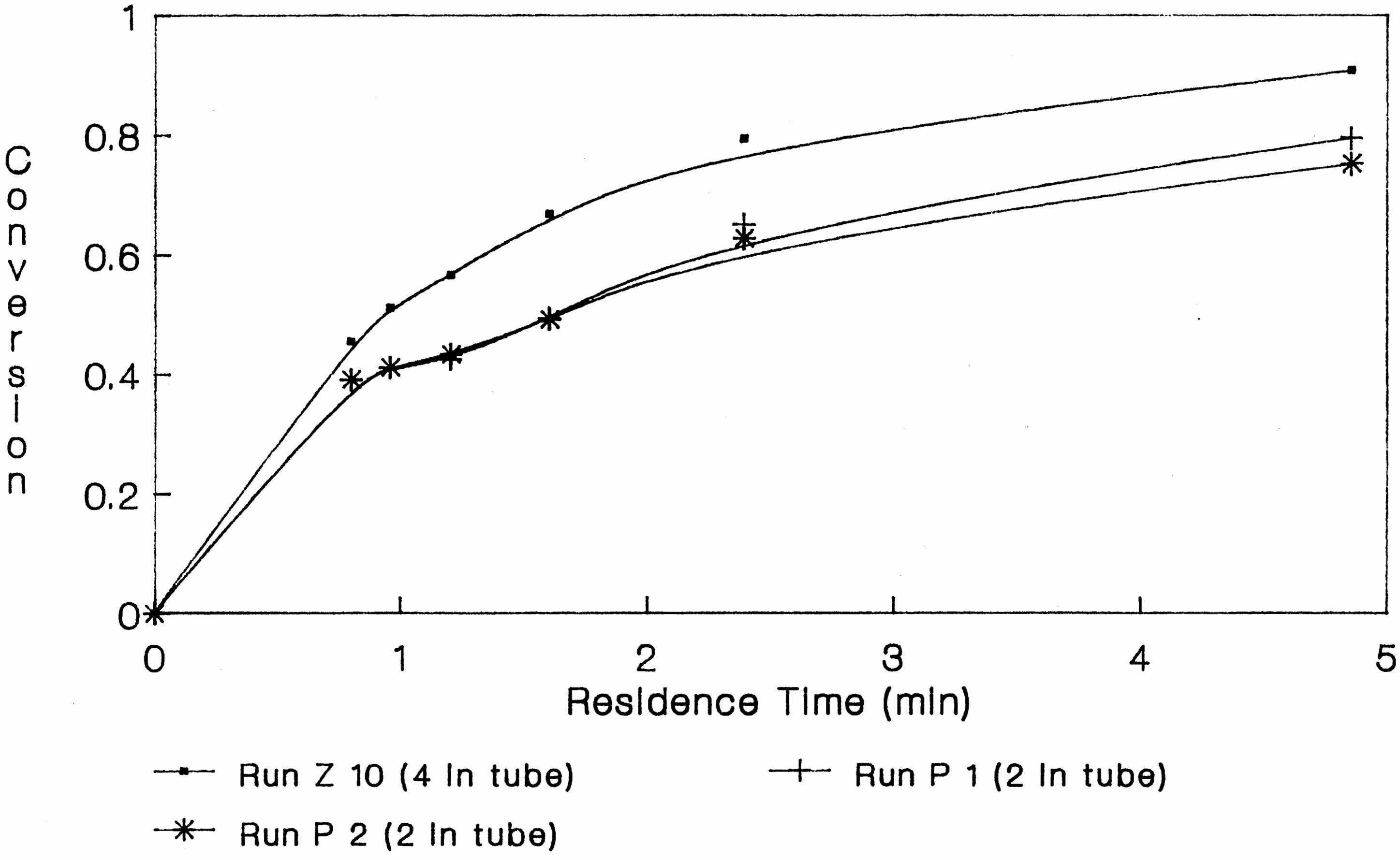

Figure 25. Effect of radius of curvature in 1/4 in. i.d

TABLE XVI

RUN K-1

Room Temperature = 25° C
NaOH Temperature = 27° C
Dye Temperature = 27° C

Date: 4-14-88
Conductivity = 650 micro ohm/cm

Q(ml/min)	T_{in}	T_{out}	A_{in}	A_{out}	ΔP(psi)	τ(min)	EXPT(X_A)	S.S(min)
150	0.158	0.383	0.8013	0.4168	3.49	0.802	0.4799	10
126	0.172	0.475	0.7645	0.3233	2.16	0.960	0.5771	15
100	0.198	0.561	0.7033	0.2510	1.77	1.206	0.6431	18
75	0.228	0.635	0.6421	0.1972	1.47	1.604	0.6928	20
50	0.452	0.883	0.3449	0.0540	0.59	2.394	0.8433	25
25	0.424	0.888	0.3726	0.0516	0.49	4.857	0.8616	30

TABLE XVII

RUN K-2

Room Temperature = 25^{o} C
NaOH Temperature = 26^{o} C
Dye Temperature = 22^{o} C

Date: 4-15-88
Conductivity 600 micro ohm/cm

Q(ml/min)	T_{in}	T_{out}	A_{in}	A_{out}	ΔP(psi)	τ(min)	EXPT(X_A)	S.S(min)
150	0.130	0.295	0.8861	0.5302	3.34	0.802	0.4016	10
126	0.148	0.390	0.8297	0.4089	2.36	0.960	0.5072	15
100	0.150	0.470	0.8239	0.3279	1.92	1.206	0.6020	18
75	0.198	0.594	0.7033	0.2262	1.23	1.604	0.6784	20
50	0.282	0.790	0.5498	0.1024	0.79	2.394	0.8138	25
25	0.465	0.899	0.3325	0.0462	0.59	4.857	0.8610	30

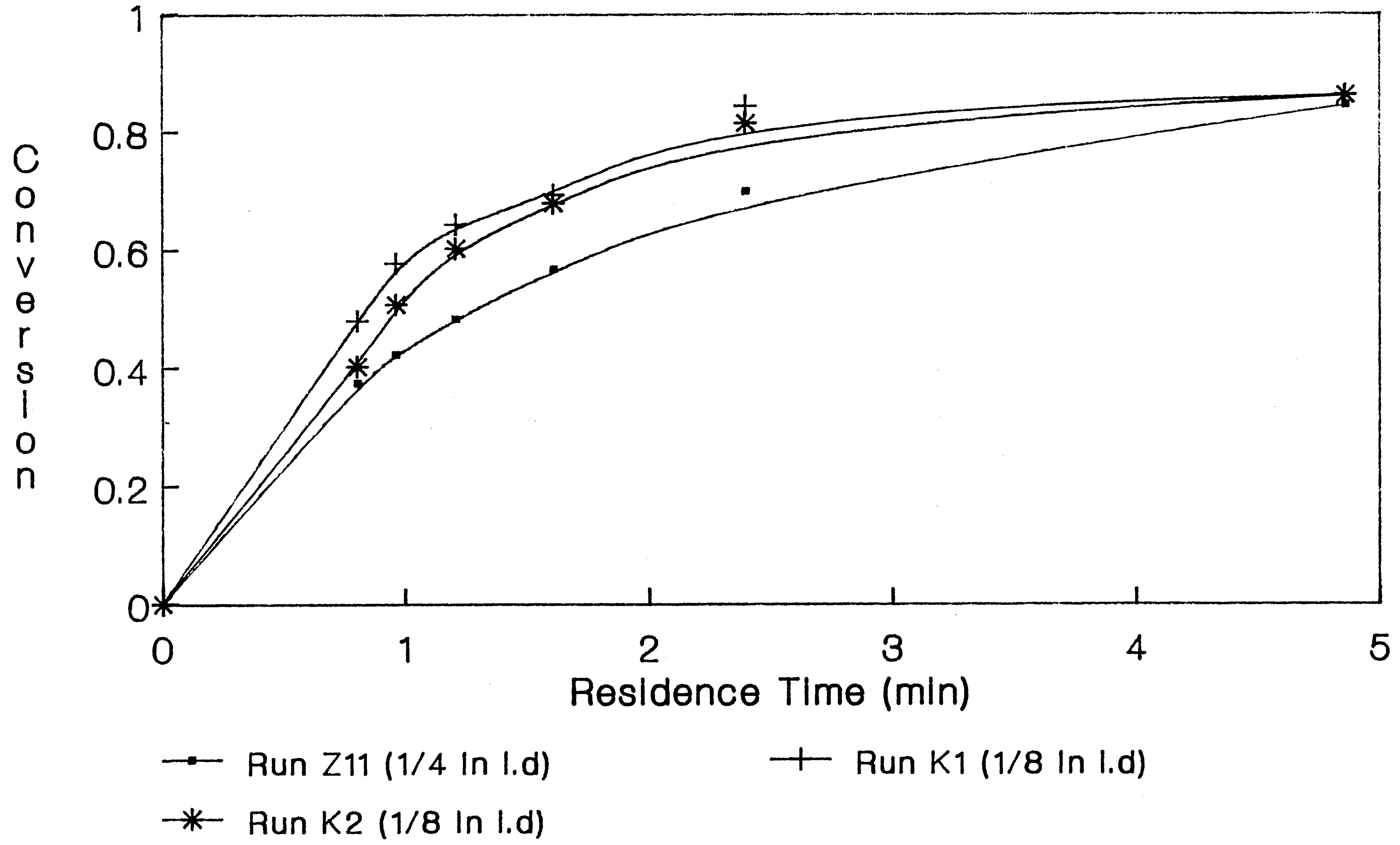

Figure 26. Effect of coil diameter

Two runs were made and the results are given in Tables XVIII and XIX.

Reproducibility

The performance of the system and the precision of the equipment used for data collection in this study were of high importance to the results obtained. In this section, the various factors that might lead to irreproducibility are discussed and the overall reproducibility of the data is given.

(1) When setting the Spec-20's to 100% transmittance before the start of each run, it was observed that the inlet Spec-20 was off the 100% mark by 17%. At 100% mark, it read 83%.

As a result of this, a performance check was made on the Spec-20's by running ordinary water and dye solution through the system. The readings from the two were found to be in good agreement within 2%.

Again, the readings were found to be more stable at night than daytime. This was due to the noise level in the laboratory environment during the day but it was not perceived as a major source of error.

(2) The flow indicator and NaOH rotameter were dismantled on a few occasions to clean the tubes due to deposition of sodium hydroxide in the tubes. This is an indication of possible NaOH deposits on the reactor walls too. But the extent of the uncertainty arising from this factor is not known.

(3) As given in the tables, some of the runs were made at different temperatures. The temperature of the reacting solution in each run was assumed to be constant but without a constant temperature

TABLE XVIII

RUN J-1

Room Temperature = 23° C
NaOH Temperature = 25° C
Dye Temperature = 22° C

Date: 4-16-88
Conductivity = 580 micro ohm/cm

Q(ml/min)	T_{in}	T_{out}	A_{in}	A_{out}	ΔP(psi)	τ(min)	EXPT(X_A)	S.S(min)
150	0.178	0.357	0.7496	0.4473	4.12	0.802	0.4032	10
126	0.183	0.421	0.7375	0.3757	2.75	0.960	0.4906	15
100	0.194	0.481	0.7122	0.3179	2.16	1.206	0.5537	18
75	0.219	0.596	0.6596	0.2248	1.55	1.604	0.6592	20
50	0.253	0.745	0.5969	0.1278	0.69	2.394	0.7858	25
25	0.402	0.880	0.3958	0.0555	0.44	4.857	0.8597	30

TABLE XIX

RUN J-2

Room Temperature = 24° C
NaOH Temperature = 23° C
Dye Temperature = 22° C

Date: 4-16-88
Conductivity = 700 micro ohm/cm

Q(ml/min)	T_{in}	T_{out}	A_{in}	A_{out}	ΔP(psi)	τ(min)	EXPT(X_A)	S.S(min)
150	0.147	0.297	0.8327	0.5272	4.86	0.802	0.3668	10
126	0.158	0.369	0.8013	0.4330	3.14	0.960	0.4597	15
100	0.165	0.419	0.7825	0.3778	2.45	1.206	0.5172	18
75	0.194	0.538	0.7122	0.2692	1.77	1.604	0.6220	20
50	0.254	0.736	0.5952	0.1331	1.03	2.394	0.7763	25
25	0.387	0.387	0.874	0.4123	0.64	4.857	0.8581	30

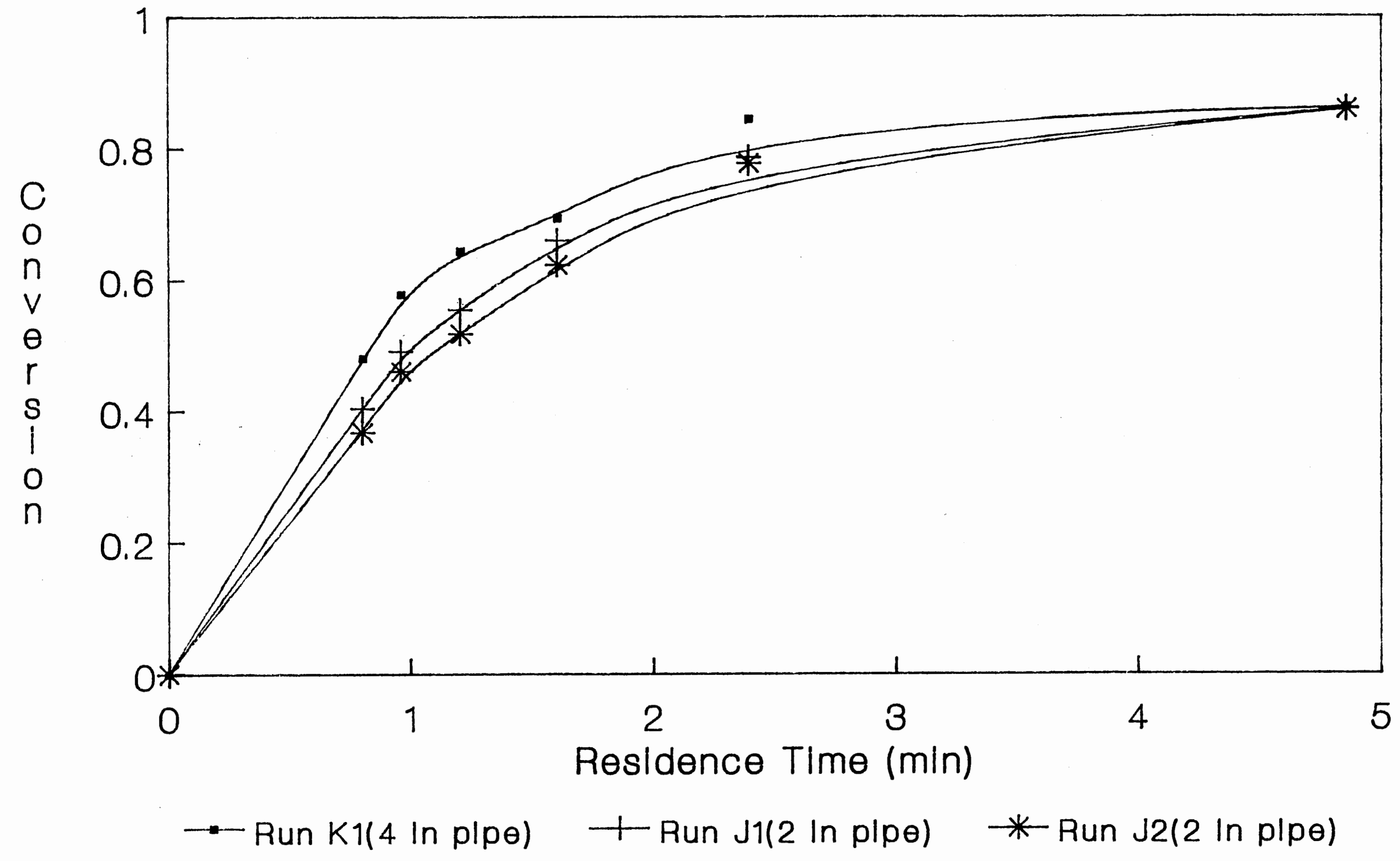

Figure 27. Effect of Radius of Curvature in 1/8 in. i.d

bath, this is hard to substantiate. So the actual temperature profile is not known and the degree of uncertainty from this error is not known.

(4) As also indicated in the tables, the conductivity of the water used was not the same for every run. Since it is not known what impurities the reaction is sensitive to, the uncertainty arising from this factor is not known.

As a result of these factors, each experimental run was duplicated to check the reproducibility.

For 1/4 in. i.d coil on 4 in. i.d pipe in the horizontal position, Run Z-7 was made as a duplicate of Z-6 and the results show an average deviation of 2.8%. In the vertical position, runs Q-1 and Q-2, though in different flow directions, show an average deviation of 6.7%. For the straight tube laminar flow results, run S-2 is a duplicate of S-1 and the results show an average of 3.8% difference.

For 1/4 in. coil on 2 in. diameter pipe, P-2 is a duplicate of P-1 and the average difference in results is 2%. For the K-runs, the average difference in conversion is 6.7% and that for J-runs is 4.5%.

Overall, given limits of experimental error, the results for this study were found to be reproducible.

Analysis of Data

As shown in Figures 19-22, the calculated conversion for plug flow and laminar flow models were less than the experimental conversion. Since the results of the experiment were found to be consistent, an investigation was made on the variables used in the calculations.

The hypothesized mechanism for this reaction is given by

$$- r_A = - \frac{dC_A}{dt} = KC_AC_B$$

for $C_{Bo} >> C_{Ao}$

$$- r_A = - \frac{dC_A}{dt} = K'C_A$$

where

$$K' = KC_{Bo}$$

This implies a pseudo-first order assumption.

The conversion for plug flow first order reaction is given by:

$$X_A = 1 - e^{-k'\tau}$$

where

$$\tau = V/v_o$$

The volume, V, is a function of tube geometry and constant. Therefore the parameters that might introduce error into the calculation are, K, C_{Bo}, v_o and of course a wrong assumption in reaction mechanism.

The investigation was carried out as follows.

An analysis of the complete rate equations was made using the differential method of data analysis given by Levenspiel (17).

From the mechanism

$$- r_A = - \frac{dC_A}{dt} = K\ f(c) \tag{1}$$

For, nth order reaction,

$$- r_A = - \frac{dC_A}{dt} = K'C_A^n \tag{2}$$

integrating,

$$\ln(- r_A) = \ln K' + n \ln C_A \tag{3}$$

With the aid of a computer software package, a plot of the concentrations (absorbance) and residence time data obtained from the experiment was made for each run. The best curve fitting equations for these data was found to be a polynomial of the form,

$$y = a_0 + a_1 x + a_2 x^2 + \ldots + a_n x^n \ldots \qquad (4)$$

For a third order polynomial

$$y = a + bx + cx^2 + dx^3 \qquad (5)$$

$$C_A = a + b\tau + C\tau^2 + d\tau^3 \qquad (6)$$

Differentiation of equation 6 gives

$$\frac{dC_A}{d} = b + 2C\tau + 3d\tau^2 \qquad (7)$$

but,

$$-\frac{dC_A}{d\tau} = -r_A$$

The slope of the curves at each residence time value was determined and a plot of $\ln(-r_A)$ vs $\ln C_A$ was made for each run.

As represented by equation 3, the slope of this plot, n, gives the order of the reaction and the intercept, lnK', gives the rate constant.

The rate constants obtained from this analysis were used to calculate the conversions for plug flow and laminar flow models, if the order of the reaction is close to first order as assumed.

A comparison of the rate constants was made with those obtained from the batch experiment, given by $\ln K = 26.3228 - 7250.496/T$.

Some of the data obtained from these analysis are given in Tables XX-XXII.

TABLE XX

ANALYSIS OF DATA FOR RUN Z-10

τ(min)	C_A(A)	$-r_A$	$\ln(-r_A)$	$\ln C_A$
0.0	1.201	-	-	-
0.0	1.149	-	-	-
0.0	1.149	-	-	-
0.0	1.097	-	-	-
0.0	1.041	-	-	-
0.0	0.910	-	-	-
0.802	0.654	0.4431	-0.8140	-0.425
0.960	0.562	0.4033	-0.9081	-0.576
1.206	0.499	0.3454	-1.0631	-0.695
1.604	0.364	0.2618	-1.3400	-1.011
2.394	0.214	0.1335	-2.0140	-1.542
4.857	0.084	0.0528	-2.9415	-2.477

TABLE XXI

REACTOR COMPARISON USING ANALYZED DATA

τ(min)	X_A EXPT	X_A PFTR	X_A LFTR
0.802	0.455	0.440	0.392
0.960	0.511	0.501	0.429
1.206	0.566	0.582	0.502
1.604	0.668	0.689	0.612
2.394	0.794	0.823	0.748
4.857	0.908	0.970	0.864

TABLE XXII

SUMMARY OF DATA ANALYSIS

Run #	T(^oC)	Order(n)	K'(KC_{Bo})$^{min-1}$	K(L/mol min)	ln K	$\frac{1}{T}$ x10^3(T in K)
Z-10	25	1.061	0.7236	18.09	2.895	3.356
Z-11	25.5	1.010	0.5062	12.66	2.538	3.350
S-1	24.5	1.024	0.6419	16.05	2.776	3.361
S-2	24	0.835	0.4884	12.21	2.502	3.367
Q-1	25	0.653	0.4424	11.06	2.403	3.356
Q-2	26.5	0.795	0.5127	12.82	2.551	3.339
P-1	23	0.948	0.4165	10.41	2.343	3.378
P-2	23.5	1.306	0.5059	12.65	2.537	3.373

To check the actual concentration of sodium hydroxide in the tank, the same procedure used in making up solutions for the runs was used to make up a sodium hydroxide solution in the tank. By calculation, this solution is 0.08 M NaOH solution.

After recycling, six 25 ml-volumes of the solution were withdrawn from the tank with a pippette and titrated with 1 N hydrochloric acid (HCl) solution, using phenolphthalene indicator.

The average concentration of NaOH in the tank from the six titrations was 0.079 M. This indicates that the sodium hydroxide solution was accurate as prepared for the runs and eliminates the chance of C_{Bo} being in error as used in the conversion calculations for plug flow and laminar flow tubular reactors.

The fluctuation, if any, in the sodium hydroxide and dye rotameter readings was such that there was a flow of 300 ± 20 ml/min from each tank. This gives a maximum flow of 600 ± 40 ml/min in the reactor. An analysis on the conversion using the residence times obtained with this flow limits gave a deviation of ± 5.6% from the conversion obtained in the experiment.

This was considered to be within limits of experimental error. Thus v_o was not a major source of error which implies that the residence time (τ) values used in the calculations were accurate.

Since C_{Bo} and v_o were apparently accurate, a detailed analysis was made on the rate constants.

Table XX gives the analysis for Run Z-10. A plot of $\ln(-r_A)$ vs $\ln C_A$ was made to obtain the order of reaction for this run as 1.06 and the pseudo first order rate constant K' as 0.724 min^{-1}. Using this K' value and $C_{Bo} = 0.04$, the conversions for plug flow and laminar flow reactors were calculated to see how they compare with the experiment. The results of these calculations are given in Table XXI and the plot is shown in Figure 28. The trend seen in this plot agrees with that of Asfour (20). The helical flow model falls between the plug and laminar model. At low residence time, it crosses the plug flow model. This can be attributed to experimental error at these points.

Similar analysis was made on the results of every run that properly fits the polynomial equation. A summary of these analysis is given in Table XXII.

A linear regression was made on the ln K vs 1/T values in Table XXII to obtain,

Correlation, $R^2 = 0.29$
Intercept, $a = 16.6658$
Slope, $b = -4195.74$

from the Arrhenius equation,

$$\ln K = \ln K_0 - E/RT$$

Using the above parameters, a new equation for the temperature dependency of the rate constant was found to be,

$$\ln K = 16.6658 - 4195.74/T$$

For R = 1.987 cal, the activation energy was calculated as 8.3 K cal.

The correlation coefficient, R^2, for this regression was found to be low as given above. This can be attributed to the very small temperature difference in the runs as seen in the table.

However, the activation energy indicates a low temperature dependency for this reaction.

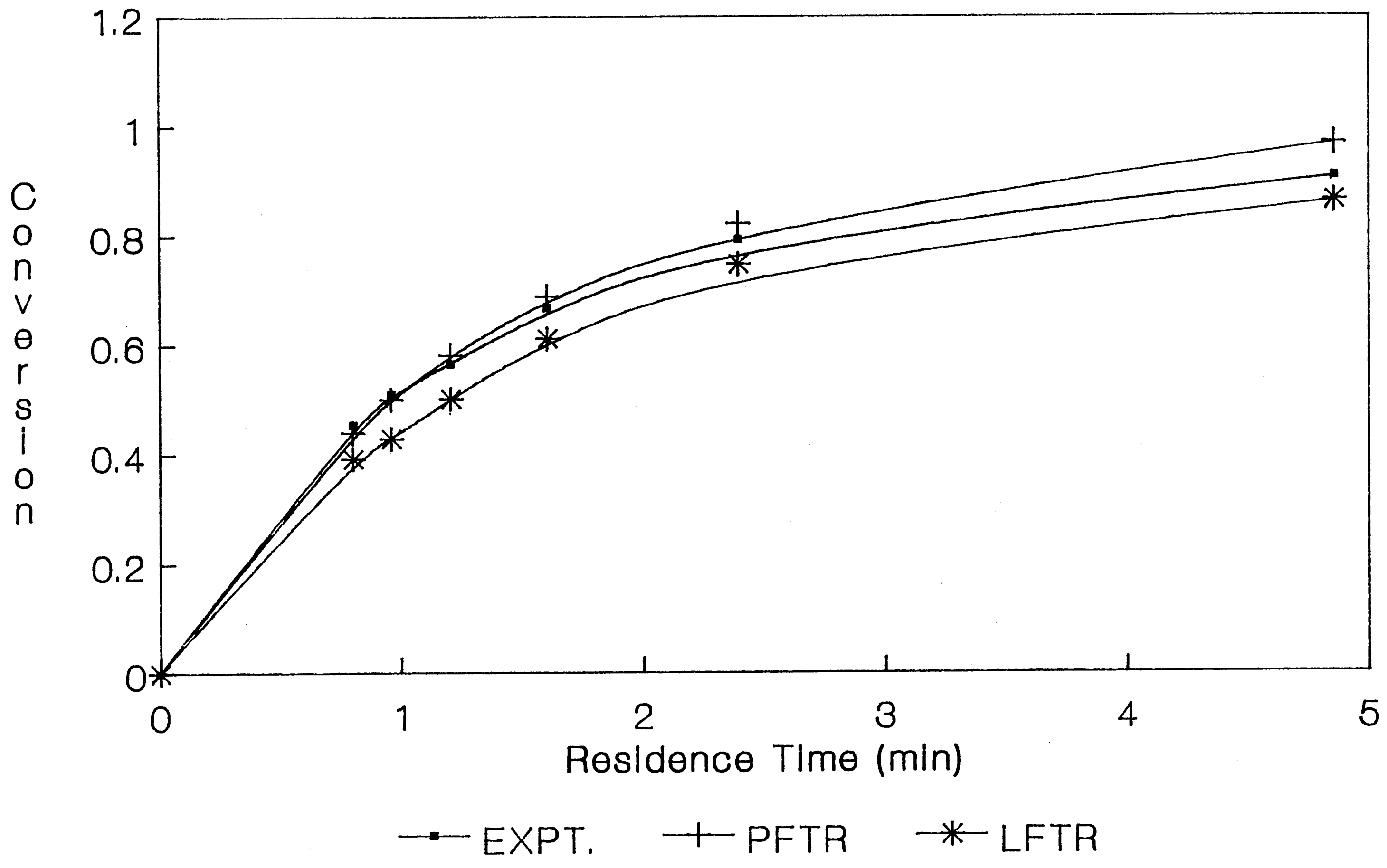

Figure 28. Comparison with analyzed data

From the above equation, at room temperature of 25^{o}C,

K = 13.3 L/mol min

CHAPTER VI

DISCUSSION

Helically-coiled tubular reactors have been shown to have the advantages of higher heat transfer coefficients, less space and reduced axial dispersion (6,9) over straight tubes. At high Reynolds number, it has been proven (6) that straight tubes have higher pressure drop than helical tubes at low Reynolds numbers and as such higher power consumption.

The aim of this work was to propose an alternative to the use of straight tube reactors at high Reynolds number and as such high pressure drop, with practically no cost in the product conversion. It was intended to achieve this by taking advantage of the secondary mixing effects in helically-coiled tubular reactors.

In this chapter, the effects of various changes of variables made in this work will be discussed. Some proposed models in the literature will be used to make inferences from the experimental results.

Effect of Change in Reactor Orientation

The effects of reactor orientation are seen in the plots comparing Z and Q runs. The results show that the conversions for the reactor in the horizontal positions are slightly higher than that in the vertical positions especially at high flow rates. This is in agreement with the results of Sakra, et al. (7) who did a similar study based on dispersion number (D/uL). In any case, the slight difference may be due to the difference in velocity profile as observed by Mori and Nakayama (13). On the other hand, the pressure drop in both cases is within ± 5%.

The difference in conversion is considered to be within limits of experimental uncertainty and as such is negligible.

The results of run Q-1 and Q-2 show that upflow or downflow of the reacting solutions in a vertical reactor, have no appreciable effect on the reactor performance.

Koutsky and Adler (6) also ran tests in both horizontal and vertical positions to prove the negligible effect of body forces.

Effects of Change in Reactor Geometry

Changes were made in the radius of curvature and the coil diameter of the reactor.

As shown in Figure 25, the results of P and Z runs indicate a considerable decrease in conversion with a decrease in radius of curvature for a 1/4 in. i.d coil. Also Figure 27 and the results of the K and J runs show the same trend of effect for the 1/8 in. i.d coil. This implies a decrease in the effects of secondary mixing.

As reported by Srinivasan, et al. (9), Berg and Bonilla gave a correlation for the inside heat transfer coefficient in helices for this Reynolds number range as,

$$h_i = \frac{C_p \mu}{di} [0.0000229 + 0.000636 (di/D)] (N_{Re})^{1.29}$$

where,

di = tube diameter

D = coil diameter

The diameters used in this work were put into this equation and the calculated values showed that, for the same coil diameter, D, a decrease in radius of curvature, which means decrease in di, led to a decrease in h_i by about the same magnitude. This agrees with the results of the experiment as decreased heat transfer coefficient implies a decrease in the effects of secondary mixing.

The results of the P-runs also show a higher pressure drop across the reactor, than the Z-runs, due to a decrease in radius of curvature.

This can be explained with the correlations given in the literature for the hydrodynamics in helical coils. As reported in Srinivasan, et al. (9), Dean characterized the dynamic similarity of fluid flow through a helix as

$$N_{Dn} = N_{Re}(di/D)^{0.5}$$

where,

di = tube diameter

D = coil diameter

N_{Dn} = Dean number.

Based on this correlation, White (12) gave the following relationship for the pressure head loss in a coil:

$$\Delta H = C(8/N_{Re})\ [4l/di)(u^2/g)]$$

where

$$\frac{1}{C} = \left\{1 - [1 - (11.6/N_{Dn})^{0.45}]^{1/0.45}\right\}$$

for $11.6 < N_{Dn} < 2000$.

Calculations were made using the experimental data for P and Z runs and the results show that ΔH is higher with a reduction in di, which conforms with the results of the experiment.

In this work, changes were also made in the coil diameter. As shown in Figure 26, the results from the Z and K runs indicate an increase in conversion with a decrease in coil diameter.

The Berg and Bonilla equation given above for inside heat transfer coefficient, were also used to evaluate this trend.

For the same di and N_{Re}, a decrease in coil diameter, D, implies an increase in the ratio (di/D) which results in an increase in heat transfer coefficient. This infers that there is an increase in secondary mixing effects which is demonstrated by the higher conversions observed in the experiment.

Again, the White correlation given above can be used to explain the higher pressure drop obtained with a decrease in coil diameter.

For the same Reynolds number, N_{Re}, a decrease in coil diameter will result in an increase in mean velocity, u, and an increase in Dean number, D_n. From the correlation, it is evident that this will amount to an increase in the pressure head loss, ΔH.

No attempt was made to study the effect of pitch change on the reactor performance. This may be a subject for further investigation.

Pressure Drop

The pressure drop in all the runs made were found to be within ± 5% deviation.

The pressure drop data for run Z-10, in 50 ft reactor length, 1/4 in. coil diameter, were used in comparing the experimental results with the pressure drop obtained by calculation from published correlations.

For straight tube in laminar flow, McCabe and Smith (30) correlation was used

$$\Delta P_s = \frac{32\ L\ \bar{v}\mu}{g_c D^2}$$

Koutsky and Adler (6) gave the pressure drop in helical tubes as,

$$\Delta P_c = \frac{c_1 f_1 \rho L\ \bar{v}^{\ 2}}{2\ D\ g_c}$$

where

f_1 = $64/N_{Re}$

c_1 = ratio of friction factor of helical tube to friction factor of straight tube, in laminar flow.

The c_1 value was obtained from their plot for ellipticity (a/b) = 1.

The results from these calculations are given in Table XXIII.

From the results, it is seen that pressure drop obtained from the experiment agrees fairly with the predicted values from Koutsky and Adler (6) correlation.

Again, it is found that the experimental values are higher (up to 65%) than the predicted values from McCabe and Smith correlation. These observations are found to be true for other runs.

To obtain the same pressure drop as experiment, from a straight tube under turbulent flow conditions, the Reynolds number required for each experimental pressure drop is obtained with the correlation in McCabe and Smith (30).

$$\Delta P = \frac{2fL\ \rho\bar{v}^{\ 2}}{g_c D}$$

TABLE XXIII

PRESSURE DROP COMPARISON

Q(ml/min)	N_{Re}	$\bar{v}$(ft/s)	EXPT. ΔP(Psi)	Straight Tube Calculated ΔP_s(Psi)	Coiled Tube Calculated ΔP_c(Psi)
600	2004	1.039	1.52	0.56	1.56
500	1671	0.866	1.33	0.46	1.16
400	1336	0.692	1.03	0.37	0.85
300	1003	0.520	0.79	0.28	0.61
200	672	0.348	0.59	0.19	0.37
100	331	0.172	0.49	0.09	0.18

where,

f = friction factor, obtained from Moody plots for smooth tubes in viscous flow.

The Reynolds number required is used to determine the velocity, length of reactor, residence time and pressure drop required to obtain the same conversion as the experiment, in a reactor of the same diameter under turbulent flow conditions.

With N_{Re} known,

$$\bar{v}_{required} = N_{Re}\ \mu/\rho\ D$$

For plug flow,

$$X_A = 1 - e^{-K'\tau}$$

With $X_A = X_A$ (EXPT.) and K' = pseudo rate constant obtained in analysis of data (K' = 0.7236 min^{-1} for run Z-10),

$$\tau \text{ required} = \frac{\ln(1-X_A)}{-K'}$$

$$L_{(required)} = \bar{v}\tau$$

Hence,

$$\Delta P = \frac{2fL\rho\bar{v}^2}{g_c D} \quad .$$

The results obtained from these calculations for run Z-10 are given in Table XXIV.

From these results, it is found that the pressure drop required to obtain the same conversion as a helically-coiled tubular reactor under laminar flow, in a straight tube under turbulent flow is about 61%

TABLE XXIV

DETERMINATION OF REQUIRED PRESSURE DROP

EXPT. ΔP(Psi)	EXPT. X_A	Required N_{Re}	Required τ(min)	Required $\bar{v}$(ft/s)	Required L(ft)	f	Required ΔP(Psi)
1.52	0.455	5400	0.84	2.80	141	0.003	4.28
1.33	0.511	4500	0.99	2.33	138	0.0037	3.59
1.03	0.566	3500	1.15	1.81	125	0.005	2.65
0.79	0.668	2400	1.52	1.24	113	0.0078	1.76

$K' = 0.7236$, $D = 0.0208$ ft.

TABLE XXV

SUMMARY OF EXPERIMENTAL CONDITIONS

Run #	Room Temp (^{o}C)	Water conductivity (microhm/cm)	Reactor Position	Flow Pattern	Geometry
Z-6	25	750	Horizontal	Low to High Flow Rates	1/4 in. tube i.d, 4 in. pipe
Z-7	25	750	"	"	" "
Z-10	25	9	"	High to Low Flow Rates	" "
Z-11	26	780	"	"	" "
S-1	25	800	Straight Tube	"	" "
S-2	24	700	Straight Tube	"	" "
Q-1	27	850	Vertical	Upward Flow	" "
Q-2	26	800	Vertical	Downward Flow	" "
P-1	24	730	Horizontal	High to Low Flow Rates	1/4 in. tube i.d, 2 in. pipe
P-2	24	730	"	"	" "
K-1	25	650	"	"	1/8 in. tube i.d, 4 in. pipe
K-2	25	600	"	"	" "
J-1	23	580	"	"	1/8 in. tube i.d, 2 in. pipe
J-2	24	700	"	"	" "

higher than the coiled tube and the reactor length required is more than twice that of a helical reactor.

Put in economic terms, this implies that the cost of energy is higher by, probably, the same order of magnitude.

Effect of Temperature Change

The effect of temperature is seen in the rate constant. From the analysis of data, an Arrhenius equation of the form

$$\ln K = 16.6658 - 4195.74/T$$

was obtained.

For a reaction at $25^{\circ}C$, this equation gives a pseudo-first order rate constant of 0.53 min^{-1}. If the temperature increases by one degree celsius (i.e. $26^{\circ}C$), the rate constant becomes 0.56 min^{-1}. If the temperature decreases by one degree (i.e $24^{\circ}C$), the rate constant becomes 0.51 min^{-1}. This gives an average deviation of ± 4.5%.

Therefore the temperature dependency of the reaction is very low.

CHAPTER VII

CONCLUSIONS

A continuous flow helically-coiled tubular reactor had been successfully designed for use in undergraduate laboratory. The change of color of the reaction mixture between the reactor inlet and outlet was due to conversion along the reactor. This visual effect is expected to give the students a feel of what is going on inside the reactor. The fact that very long tubes can be put into a small area for experimental work is very convenient for laboratory space.

The following conclusions can be drawn from the experimental results:

(1) The hydrolysis reaction between sodium hydroxide and crystal violet dye is of low temperature dependency.

(2) The reactor orientation or direction of flow of reacting solutions has a negligible effect on the conversion.

(3) The conversion in the reactor decreases with a decrease in radius of curvature while the pressure drop increases.

(4) The conversion and the pressure drop in the reactor increase with a decrease in coil diameter.

(5) In the laminar regime, the pressure drop in a helical reactor is higher than that in an equivalent length of straight tube reactor.

(6) The reaction is actually of a fractional order but the assumption of a pseudo-first order is valid for all practical purposes.

(7) For the same conversion and reactor diameter, the pressure drop in a helical reactor under laminar flow is less than that in a straight tube (plug flow) reactor under turbulent conditions.

This means that power consumption will be less for the helical flow reactor and the cost of energy is lower. Therefore, it is more economical to use helical flow reactors and operate in laminar flow instead of using plug flow reactors in turbulent regime, if conversion or product yield is the ultimate goal.

Recommendations

(1) Constant temperature bath should be used for the reactor and the reactants.

(2) Studies should be done in the turbulent regime for both the helically-coiled reactor and the straight tube. This will require two bigger pumps of equal capacity installed in the system and will give a good prediction of the plug flow model.

(3) A mixer may be needed for the reactants to ensure that a solution of uniform concentration goes into the reactor.

(4) For the purpose of the undergraduate laboratory work, it is suggested to use the same materials, of known weight, in all dye and sodium hydroxide measurements. This will reduce the chances of error due to measurements.

(5) In the event that the stainless steel balls in the flow meter tubes get stuck, it is suggested to dismantle the flow meter and clean the tubes with a dilute acid solution. This solution should not be run through the reactor system for fear of interference with the reaction results.

(6) Though it is not certain what impurities affect the reaction, it is recommended to use water with low conductivity (2-100 micro ohm/cm).

(7) The experimental system should be flushed with water after each run. This will reduce the deposition of sodium hydroxide in the tube walls.

(8) The sodium hydroxide solution used in this study was first prepared in a beaker and then poured into the sodium hydroxide tank. It is hereby recommended to filter that solution before pouring it into the tank. This will eliminate the cloudiness of the solution in the tank due to presence of undissolved sodium hydroxide and also help to reduce the deposition of sodium hydroxide in the system.

(9) One titration should be made on the sodium hydroxide solution in the tank. This will ensure that the procedure used in making up the solution is accurate.

(10) A different reaction should be investigated to see the general applicability of the pseudo-first order assumption.

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APPENDIX A

PHYSICAL CONSTANTS AND CALCULATIONS

Viscosity $\mu_{water} = 6.7197\text{x}10^4$ lbm/ft-s

Density = ρ_{water} = 62.34 lmb/ft^3

Reactor length = 50 ft

Pitch = h/R_c

where,

h = distance between two adjacent loops of the helix

R_c = radius of curvature (the radial distance from the axis of tube to the center of the cross section of the helix)

1. For 1/4 in. i.d tube on 4 in. pipe

D = 1/4 in. = 0.0208 ft

$$\text{Area} = \frac{\pi D^2}{4} = 3.40 \times 10^{-4} \text{ ft}^2$$

Volume = 0.017 ft^3

R_c = pipe inner radius + pipe thickness + tube outer radius

= 2 in. + 1/4 in. + (5/16 in./2)

= 2.4063 in.

h = 6.16 in. = 0.375 in.

$$\text{Pitch} = \frac{0.375}{2.4062} = 0.156$$

Number of turns = 40

2. For 1/4 in. tube on 2 in pipe

R_c = 1 in. + 3/16 in. + (5/16 in./2)

= 1.344 in.

h = 6/16 in. = 0.375 in.

$$\text{Pitch} = \frac{0.375}{1.344} = 0.279$$

Number of turns = 68

3. For 1/8 in. tube on 4 in. pipe

R_c = 2 in. + 1/4 in. + (3/16/2)

= 2.344 in.

h = 4/16 in. = 0.25 in.

Pitch = 0.107

Number of turns = 38

4. For 1/8 in. tube on 2 in. pipe

R_c = 1 in. + 3/16 in. + (3/16/2)

= 1.281 in.

h = 4/16 in.

Pitch = 0.195

Number of turns = 70

For 1/8 in. tube,

D = 0.0104 ft

$$\text{Area} = \frac{\pi D^2}{4} = 8.52 \times 10^{-5}\ \text{ft}^2$$

Volume = area x length = 4.26×10^{-3} ft^3

APPENDIX B

DATA FOR BATCH RUNS

TABLE XXVI

CALIBRATION CURVE DATA AT 591 NM WAVELENGTH

Date: 11-18-87

Concentration (g/mL)	Experimental Absorbance
0.5×10^{-6}	0.16
1.0×10^{-6}	0.26
2.0×10^{-6}	0.51
3.0×10^{-6}	0.68
4.0×10^{-6}	0.89
5.0×10^{-6}	0.95
6.0×10^{-6}	1.20
7.5×10^{-6}	1.38

Correlation coeff. = 0.9871, Intercept = 0.1206, Slope = 0.1747

TABLE XXVII

CALIBRATION CURVE DATA AT 590 NM WAVELENGTH

Date: 11-18-87

Concentration (g/mL)	Experimental Absorbance
0.5×10^{-6}	0.21
1.0×10^{-6}	0.27
2.0×10^{-6}	0.48
3.0×10^{-6}	0.62
4.0×10^{-6}	0.80
5.0×10^{-6}	1.1
6.0×10^{-6}	1.2
7.5×10^{-6}	1.4

Correlation coeff. = 0.9516, Intercept = 0.1937 Slope = 01497

TABLE XXVIII

DATA FOR BATCH RUN A.1

10 ml of 7.353x10^{-5} M dye
300 ml of 0.02 M NaOH

Date: 11-24-87
C_{Ao} = 2.372x10^{-6} M
C_{Bo} = 0.0194 M

Time(min)	Absorbance	Dye Conc., g/L	Dye Conc., M	- ln C_A/C_{Ao}
1	0.17	0.75x10^{-3}	1.838x10^{-6}	0.255
2	0.15	0.67x10^{-3}	1.642x10^{-6}	0.368
3	0.12	0.54x10^{-3}	1.324x10^{-6}	0.583
4	0.10	0.45x10^{-3}	1.103x10^{-6}	0.766
5	0.09	0.4x10^{-3}	9.804x10^{-7}	0.884
6	-	-	-	-
7	0.065	0.28x10^{-3}	6.863x10^{-7}	1.240
8	0.06	0.25x10^{-3}	6.128x10^{-7}	1.353
9	0.049	0.22x10^{-3}	5.392x10^{-7}	1.481
10	0.042	0.18x10^{-3}	4.412x10^{-7}	1.682
11	0.025	0.1x10^{-3}	2.451x10^{-7}	2.270

Correlation coeff. = 0.9629, Intercept = 0.0216, Slope = 0.1778

TABLE XXIX

DATA FOR BATCH RUN A.2

10 ml of 7.353x10^{-5} M dye
350 ml of 0.02 M NaOH

Date: 11-25-87
C_{Ao} = 2.043x10^{-6} M
C_{Bo} = 0.0194 M

Time(min)	Absorbance	Dye Conc., g/L	Dye Conc., M	- ln C_A/C_{Ao}
0.5	0.16	0.7x10^{-3}	1.716x10^{-6}	0.174
1	0.14	0.62x10^{-3}	1.520x10^{-6}	0.296
2	0.12	0.54x10^{-3}	1.324x10^{-6}	0.434
3	0.10	0.45x10^{-3}	1.103x10^{-6}	0.616
4	0.08	0.35x10^{-3}	8.579x10^{-7}	0.868
5	0.07	0.3x10^{-3}	7.353x10^{-7}	1.022
6	0.052	0.23x10^{-3}	5.637x10^{-7}	1.288
7	0.044	0.2x10^{-3}	4.902x10^{-7}	1.427
8	0.04	0.17x10^{-3}	4.167x10^{-7}	1.590
9	0.03	0.13x10^{-3}	3.186x10^{-7}	1.858

Correlation coeff. = 0.9970, Intercept = 0.0684, Slope = 0.1954

TABLE XXX

DATA FOR BATCH RUN A.3

10 ml of 7.353×10^{-5} M dye
300 ml of 0.04 M NaOH

Date: 11-25-87
$C_{Ao} = 2.372\times10^{-6}$ M
$C_{Bo} = 0.0387$ M

Time(min)	Absorbance	Dye Conc., g/L	Dye Conc., M	$-\ln C_A/C_{Ao}$
1	0.14	0.62×10^{-3}	1.520×10^{-6}	0.445
2	0.11	0.5×10^{-3}	1.226×10^{-6}	0.660
3	0.08	0.35×10^{-3}	8.579×10^{-7}	1.017
4	0.06	0.25×10^{-3}	6.128×10^{-7}	1.353
5	0.04	0.17×10^{-3}	4.167×10^{-7}	1.739
6	0.03	0.13×10^{-3}	3.186×10^{-7}	2.008
7	0.02	0.09×10^{-3}	2.206×10^{-7}	2.375
8	0.018	0.07×10^{-3}	1.716×10^{-7}	2.626
9	0.008	0.025×10^{-3}	6.128×10^{-8}	3.656
10	0.002	0.01×10^{-3}	2.451×10^{-8}	4.572

Correlation coeff. = 0.9420, Intercept = - 0.2714, Slope = 0.4212

TABLE XXXI

DATA FOR BATCH RUN A.4

10 ml of 7.353×10^{-5} M dye
350 ml of 0.04 M NaOH

Date: 11-25-87
$C_{Ao} = 2.043\times10^{-6}$ M
$C_{Bo} = 0.0389$ M

Time(min)	Absorbance	Dye Conc., g/L	Dye Conc., M	$-\ln C_A/C_{Ao}$
1	0.13	0.58×10^{-3}	1.4216×10^{-6}	0.363
2	0.085	0.38×10^{-3}	9.314×10^{-7}	0.785
3	0.055	0.24×10^{-3}	5.882×10^{-7}	1.245
4	0.035	0.14×10^{-3}	3.431×10^{-7}	1.784
5	0.02	0.09×10^{-3}	2.206×10^{-7}	2.226
6	0.018	0.07×10^{-3}	1.716×10^{-7}	2.477
7	0.01	0.05×10^{-3}	1.225×10^{-7}	2.814
8	0.001	0.005×10^{-3}	1.225×10^{-8}	5.117

Correlation coeff. = 0.8773, Intercept = - 0.4465, Slope = 0.5662

TABLE XXXII

DATA FOR BATCH RUN A.5

10 ml of $7.353\text{x}10^{-5}$ M dye
300 ml of 0.02 M NaOH
Temperature = 24^{o} C

Date: 11-29-87
C_{Ao} = $2.372\text{x}10^{-6}$ M
C_{Bo} = 0.0194 M

Time(min)	Absorbance	Dye Conc., M	- ln C_A/C_{Ao}
1	0.16	$1.716\text{x}10^{-6}$	0.324
2	0.135	$1.4706\text{x}10^{-6}$	0.478
3	0.115	$1.250\text{x}10^{-6}$	0.641
4	0.09	$9.804\text{x}10^{-7}$	0.884
5	0.082	$8.824\text{x}10^{-7}$	0.988
6	0.072	$7.598\text{x}10^{-7}$	1.138
7	0.065	$6.863\text{x}10^{-7}$	1.240
8	0.052	$5.637\text{x}10^{-7}$	1.437
9	0.05	$5.392\text{x}10^{-7}$	1.481
10	0.048	$4.902\text{x}10^{-7}$	1.577

Correlation coeff. = 0.9839, Intercept = 0.2356, Slope = 0.1424

TABLE XXXIII

DATA FOR BATCH RUN A.6

10 ml of $7.353\text{X}10^{-5}$ M dye
350 ml of 0.02 M NaOH
Temperature = 26^{o} C

Date: 11-29-87
C_{Ao} = $2.043\text{x}10^{-6}$ M
C_{Bo} = 0.0194 M

Time(min)	Absorbance	Dye Conc., M	- ln C_A/C_{Ao}
1	0.13	$1.4216\text{x}10^{-6}$	0.363
2	0.11	$1.226\text{x}10^{-6}$	0.511
3	0.09	$9.804\text{x}10^{-7}$	0.734
4	0.075	$8.088\text{x}10^{-7}$	0.927
5	0.06	$6.128\text{x}10^{-7}$	1.204
6	0.05	$5.392\text{x}10^{-7}$	1.332
7	0.042	$4.412\text{x}10^{-7}$	1.533
8	0.035	$3.431\text{x}10^{-7}$	1.784
9	0.03	$3.186\text{x}10^{-7}$	1.858
10	0.025	$2.451\text{x}10^{-7}$	2.121

Correlation coeff. = 0.9957, Intercept = 0.1551, Slope = 0.1966

TABLE XXXIV

DATA FOR BATCH RUN A.7

10 ml of 7.353×10^{-5} M dye
300 ml of 0.04 M NaOH
Temperature = 26° C

Date: 11-29-87
$C_{Ao} = 2.372\times10^{-6}$ M
$C_{Bo} = 0.0387$ M

Time(min)	Absorbance	Dye Conc., M	$- \ln C_A/C_{Ao}$
1	0.14	1.520×10^{-6}	0.445
2	0.11	1.226×10^{-6}	0.660
3	0.08	8.579×10^{-7}	1.017
5	0.055	5.882×10^{-7}	1.394
6	0.042	4.902×10^{-7}	1.577
7	0.042	4.412×10^{-7}	1.682
8	0.038	3.677×10^{-7}	1.864
9	0.02	2.206×10^{-7}	2.375
10	0.01	1.225×10^{-7}	2.963

Correlation coeff. = 0.9518, Intercept = 0.1550, Slope = 0.2467

TABLE XXXV

DATA FOR BATCH RUN A.8

10 ml of 7.353×10^{-5} M dye
350 ml of 0.04 M NaOH
Temperature = 26° C

Date: 11-29-87
$C_{Ao} = 2.043\times10^{-6}$ M
$C_{Bo} = 0.0389$ M

Time(min)	Absorbance	Dye Conc., M	$- \ln C_A/C_{Ao}$
1	0.12	1.324×10^{-6}	0.434
2	0.085	9.314×10^{-7}	0.785
3	0.065	6.863×10^{-7}	1.091
4	0.05	5.392×10^{-7}	1.332
5	0.032	3.431×10^{-7}	1.784
6	0.03	3.186×10^{-7}	1.858
7	0.02	2.206×10^{-7}	2.226
9	0.01	1.225×10^{-7}	2.814
10	0.009	9.804×10^{-8}	3.037

Correlation coeff. = 0.9949, Intercept = 0.2047, Slope = 0.2876

TABLE XXXVI

DATA FOR BATCH RUN A.9

10 ml of 7.353×10^{-5} M dye	Date: 11-30-87
300 ml of 0.04 M NaOH	$C_{Ao} = 2.372\times10^{-6}$ M
Temperature = 26° C	$C_{Bo} = 0.0387$ M

Time(min)	Absorbance	Dye Conc., M	$- \ln C_A/C_{Ao}$
1	0.17	1.838×10^{-6}	0.2551
2	0.13	1.4216×10^{-6}	0.5120
3	0.10	1.103×10^{-6}	0.7657
4	0.085	9.314×10^{-7}	0.9348
5	0.068	7.108×10^{-7}	1.2051
6	0.05	5.392×10^{-7}	1.4814
7	0.04	4.167×10^{-7}	1.7391
8	0.035	3.431×10^{-7}	1.933
9	0.03	3.186×10^{-7}	2.008
10	0.02	2.206×10^{-7}	2.375

Correlation coeff. = 0.9941, Intercept = 0.0517, Slope = 0.2308

TABLE XXXVII

DATA FOR BATCH RUN A.10

10 ml of 7.353×10^{-5} M dye	Date: 11-30-87
300 ml of 0.02 M NaOH	$C_{Ao} = 2.372\times10^{-6}$ M
Temperature = 24° C	$C_{Bo} = 0.0194$ M

Time(min)	Absorbance	Dye Conc., M	$- \ln C_A/C_{Ao}$
1	0.19	2.083×10^{-6}	0.1297
2	0.17	1.838×10^{-6}	0.2551
3	0.15	1.642×10^{-6}	0.3678
4	0.12	1.324×10^{-6}	0.5831
5	0.102	1.127×10^{-6}	0.7442
6	0.09	9.804×10^{-7}	0.8835
7	0.075	8.088×10^{-7}	1.0759
8	0.065	6.863×10^{-7}	1.2402
9	0.06	6.128×10^{-7}	1.3535
10	0.05	5.392×10^{-7}	1.4814

Correlation coeff. = 0.9965, Intercept = 0.0497, Slope = 0.1566

TABLE XXXVIII

DATA FOR ABSORBANCE REGRESSION ON BATCH RUN A.1

Ao = 0.23 Time(min)	Absorbance	Date: 12-1-87 ln Ao/A
0	0.23	0.0
1	0.17	0.3023
2	0.15	0.4274
3	0.12	0.6506
4	0.10	0.8329
5	0.09	0.9383
7	0.065	1.2637
8	0.06	1.3437
9	0.049	1.5463
10	0.042	1.7004
11	0.025	2.2192

Correlation coeff. = 0.9698, Intercept = 0.1019
Slope = 0.1701

TABLE XXXIX

DATA FOR ABSORBANCE REGRESSION ON BATCH RUN A.2

Ao = 0.21 Time(min)	Absorbance	Date: 12-1-87 ln Ao/A
0	0.21	0.0
0.5	0.16	0.2719
1	0.14	0.4055
2	0.12	0.5596
3	0.10	0.7419
4	0.08	0.9651
5	0.07	1.0986
6	0.052	1.3959
7	0.044	1.5629
8	0.04	1.6582
9	0.03	1.9459

Correlation coeff. = 0.9955, Intercept = 0.1835
Slope = 0.1928

TABLE XL

DATA FOR ABSORBANCE REGRESSION ON BATCH RUN A.3

Ao = 0.23		Date: 12-1-87
Time(min)	Absorbance	ln Ao/A
0	0.23	0.0
1	0.14	0.4964
2	0.11	0.7376
3	0.08	1.0561
4	0.06	1.3437
5	0.04	1.7492
6	0.03	2.0369
7	0.02	2.4423
8	0.018	2.5477
9	0.008	3,3586
10	0.002	4.7449

Correlation coeff. = 0.9184, Intercept = -0.2028
Slope = 0.4098

TABLE XLI

DATA FOR ABSORBANCE REGRESSION ON BATCH RUN A.4

Ao = 0.21		Date: 12-1-87
Time(min)	Absorbance	ln Ao/A
0	0.21	0.0
1	0.13	0.4796
2	0.085	0.9045
3	0.055	1.3398
4	0.035	1.7918
5	0.02	2.3514
6	0.018	2.4567
7	0.01	3.0445

Correlation coeff. = 0.9897, Intercept = 0.0797
Slope = 0.4218

TABLE XLII

ABSORBANCE DATA ON BATCH RUN A.5

Ao = 0.23 Time(min)	Absorbance	Date: 12-1-87 ln Ao/A
0	0.23	0.0
1	0.16	0.3629
2	0.135	0.5328
3	0.115	0.6931
4	0.09	0.9383
5	0.082	1.0314
6	0.072	1.1614
7	0.065	1.2637
8	0.052	1.4868
9	0.05	1.5261
10	0.048	1.5669

Correlation coeff. = 0.9766, Intercept = 0.2942
Slope = 0.1386

TABLE XLIII

ABSORBANCE DATA ON BATCH RUN A.6

Ao = 0.21 Time(min)	Absorbance	Date: 12-1-87 ln Ao/A
0	0.21	0.0
1	0.13	0.4796
2	0.11	0.6466
3	0.09	0.8473
4	0.075	1.0296
5	0.06	1.2528
6	0.05	1.4351
7	0.042	1.6094
8	0.035	1.7918
9	0.03	1.9459
10	0.025	2.1282

Correlation coeff. = 0.9988, Intercept = 0.2974
Slope = 0.1853

TABLE XLIV

ABSORBANCE DATA ON BATCH RUN A.7

Ao = 0.23		Date: 12-1-87
Time(min)	Absorbance	ln Ao/A
0	0.23	0.0
1	0.14	0.4964
2	0.11	0.7376
3	0.08	1.0561
5	0.055	1.4307
6	0.048	1.5669
7	0.042	1.7004
8	0.038	1.8005
9	0.02	2.4423
10	0.01	3.1355

Correlation coeff. = 0.9185, Intercept = 0.1847
Slope = 0.2491

TABLE XLV

ABSORBANCE DATA ON BATCH RUN A.8

AO = 0.21		Date: 12-1-87
Time(min)	Absorbance	ln Ao/A
0	0.21	0.0
1	0.12	0.5596
2	0.085	0.9045
3	0.065	1.1727
4	0.05	1.4351
5	0.032	1.8814
6	0.03	1.9459
7	0.02	2.3514
9	0.01	3.0445
10	0.009	3.1499

Correlation coeff. = 0.9929, Intercept = 0.2935
Slope = 0.2937

TABLE XLVI

ABSORBANCE DATA ON BATCH RUN A.9

Ao = 0.23 Time(min)	Absorbance	Date: 12-1-87 ln Ao/A
0	0.23	0.0
1	0.17	0.3023
2	0.13	0.5705
3	0.10	0.8329
4	0.085	0.9954
5	0.068	1.2186
6	0.05	1.5261
7	0.04	1.7492
8	0.035	1.8827
9	0.03	2.0369
10	0.02	2.4423

Correlation coeff. = 0.9932, Intercept = 0.1109, Slope = 0.2263

TABLE XLVII

ABSORBANCE DATA ON BATCH RUN A.10

Ao = 0.23 Time(min)	Absorbance	Date: 12-1-87 ln Ao/A
0	0.23	0.0
1	0.19	0.1911
2	0.17	0.3023
3	0.15	0.4274
4	0.12	0.6506
5	0.102	0.8131
6	0.09	0.9383
7	0.075	1.1206
8	0.065	1.2637
9	0.06	1.3437
10	0.05	1.5261

Correlation coeff. = 09952, Intercept = 0.0236, Slope = 0.1516

TABLE XLVIII

ABSORBANCE DATA ON BATCH RUN B.1

10 ml of 7.353 M dye
300 ml of 0.02 M NaOH
Temperature 24° C

Date: 12-12-87
Ao = 0.1152

Time(min)	Transmittance	Absorbance	-ln A/Ao
1	0.773	0.1118	0.0300
2	0.799	0.0975	0.1668
3	0.832	0.0799	0.3659
4	0.844	0.0737	0.4467
5	0.862	0.0645	0.5800
6	0.881	0.0550	0.7393
7	0.896	0.0477	0.8817
8	0.912	0.0400	1.0578
9	0.918	0.0372	1.1304
10	0.920	0.0362	1.1576

Correlation coeff. = 0.9873, Intercept = 0.0716, Slope = 0.1322

TABLE XLIX

ABSORBANCE DATA ON BATCH RUN B.2

10 ml of 7.353 M dye
350 ml of 0.02 M NaOH
Temperature = 24° C

Date: 12-12-87
Ao = 0.1124

Time(min)	Transmittance	Absorbance	-ln A/Ao
1	0.810	0.0915	0.2057
2	0.834	0.0788	0.3552
3	0.850	0.0706	0.4650
4	0.869	0.0610	0.6112
5	0.882	0.0545	0.7239
6	0.904	0.0438	0.9424
7	0.906	0.0429	0.9632
8	0.916	0.0381	1.0818
9	0.931	0.0311	1.2849
10	0.943	0.0255	1.4834

Correlation coeff. = 0.9896, Intercept = 0.0661, Slope = 0.1355

TABLE L

ABSORBANCE DATA ON BATCH RUN C.1

10 ml of $7.353\text{x}10^{5}$ M dye
300 ml of 0.04 M NaOH
Temperature = 24^{o} C

Date: 12-12-87
Ao = 0.1152

Time(min)	Transmittance	Absorbance	-ln A/Ao
1	0.820	0.0862	0.2900
2	0.861	0.0650	0.5723
3	0.891	0.0501	0.8326
4	0.924	0.0343	1.2115
5	0.954	0.0205	1.7262
6	0.960	0.0177	1.8731
7	0.963	0.0164	1.9494
8	0.983	0.0075	2.7318
9	0.986	0.0061	2.9384
10	0.990	0.0044	3.2651

Correlation coeff. = 0.9846, Intercept = -0.1008, Slope = 0.3345

TABLE LI

ABSORBANCE DATA ON BATCH RUN C.2

10 ml of $7.353\text{x}10^{-5}$ M dye
350 ml of 0.04 M NaOH
Temperature = 24^{o} C

Date: 12-12-87
Ao = 0.1124

Time(min)	Transmittance	Absorbance	-ln A/Ao
1	0.847	0.0721	0.4440
2	0.879	0.0560	0.6967
3	0.921	0.0357	1.1469
4	0.936	0.0287	1.3652
5	0.958	0.0186	1.7989
6	0.968	0.0141	2.0759
7	0.975	0.0110	2.3242
8	0.986	0.0061	2.9138
10	0.998	0.0009	4.8274

Correlation coeff. = 0.9268, Intercept = -0.2801, Slope = 0.4373

TABLE LII

DATA FOR BATCH RUN D.1

10 ml dye solutions	Date: 12-14-87
300 ml of 0.02 M NaOH	Initial temp. = 10^{o} C
Ao = 0.2506	Final temp. = 16^{o} C

Time(min)	Transmittance	Absorbance	-ln A/Ao
1	0.586	0.2321	0.0767
2	0.600	0.2218	0.1221
3	0.614	0.2118	0.1682
4	0.624	0.2048	0.2018
5	0.643	0.1918	0.2674
6	0.660	0.1805	0.3281
7	0.672	0.1726	0.3729
8	0.682	0.1662	0.4107
10	0.714	0.1463	0.5382

Correlation coeff. = 0.9955, Intercept = 0.0159, Slope = 0.0509

TABLE LIII

DATA FOR BATCH RUN D.2

10 ml of dye solution	Date: 12-14-87
300 ml of 0.02 M NaOH	Initial temp. = 35^{o} C
Ao = 0.2506	Final temp. = 42^{o} C

Time(min)	Transmittance	Absorbance	-ln A/Ao
1	0.760	0.1192	0.7431
2	0.884	0.0535	1.5442
3	0.956	0.0195	2.5534
4	0.984	0.0070	3.5780
5	0.992	0.0035	3.2711
6	0.996	0.0017	4.9932

Correlation coeff. = 0.9949, Intercept = 0.0984, Slope = 0.8702

TABLE LIV

DATA FOR BATCH RUN D.3

10 ml of 7.35x10^{-5} M dye solution
300 ml of 0.02 M NaOH solutions
Ao = 0.2506

Date: 12-16-87
Initial temp. = 15° C
Final temp. = 18° C

Time(min)	Transmittance	Absorbance	-ln A/Ao
1	0.587	0.2314	0.0797
2	0.612	0.2132	0.1616
3	0.624	0.2048	0.2018
4	0.649	0.1878	0.2885
5	0.670	0.1739	0.3654
6	0.679	0.1681	0.3993
7	0.705	0.1518	0.5013
8	0.722	0.1415	0.5716
9	0.741	0.1302	0.6548
10	0.755	0.1221	0.7190

Correlation coeff. = 0.9961, Intercept = 0.0034, Slope = 0.0711

TABLE LV

DATA FOR BATCH RUN D.4

10 ml of dye solution
300 ml of 0.02 M NaOH
Ao = 0.2506

Date: 12-16-87
Initial temp. = 25° C
Final temp. = 34° C

Time(min)	Transmittance	Absorbance	-ln A/Ao
1	0.619	0.2083	0.1849
2	0.648	0.1884	0.2853
3	0.702	0.1537	0.4889
4	0.755	0.1221	0.7190
5	0.796	0.0991	0.9277
6	0.831	0.0804	1.1368
7	0.871	0.0600	1.4295
8	0.891	0.0501	1.6098
9	0.908	0.0419	1.7886
10	0.932	0.0306	2.1029

Correlation coeff. = 0.9949, Intercept = -0.1237, Slope = 0.2165

TABLE LVI

DATA FOR BATCH RUN D.5

10 ml of dye solution 300 ml of 0.02 M NaOH Ao = 0.2506		Date: 12-16-87 Initial temp. = 20^{o} C Final temp. = 24^{o} C	
Time(min)	**Transmittance**	**Absorbance**	**-ln A/Ao**
1	0.601	0.2211	0.1252
2	0.631	0.2000	0.2255
3	0.662	0.1791	0.3359
4	0.672	0.1726	0.3729
5	0.714	0.1463	0.5382
6	0.745	0.1278	0.6734
7	0.772	0.1124	0.8018
8	0.779	0.1085	0.8371
9	0.801	0.0964	0.9554
10	0.826	0.0830	1.1050

Correlation coeff. = 0.9915, Intercept = 0.0019, Slope = 0.1082

TABLE LVII

DATA ON Ao VALUES FOR GIVEN TEMPERATURE

Date: 12-16-87

Temp(^{o}C)	Transmittance	Absorbance (Ao)
10	0.558	0.2534
15	0.566	0.2472
20	0.560	0.2518
25	0.554	0.2565
35	0.570	0.2441

TABLE LVIII

DATA FOR BATCH RUN E.1

10 ml of 7.353x10^{-5} M dye
300 ml of 0.02 M NaOH
Temperature = 25^{o} C

Date: 1-19-88
Ao = 0.2377
C_{Bo} = 0.0194

Time(min)	Transmittance	Absorbance	-ln A/Ao
1	0.636	0.1965	0.1903
2	0.690	0.1612	0.3884
3	0.738	0.1319	0.5890
4	0.771	0.1129	0.7445
5	0.794	0.1002	0.8638
6	0.801	0.0964	0.9025
7	0.850	0.0706	1.2140
8	0.882	0.0545	1.4528
9	0.899	0.0462	1.6380
10	0.915	0.0386	1.8178

Correlation coeff. = 0.9851, Intercept = 0.0067, Slope = 0.1773

TABLE LIX

DATA FOR BATCH RUN E.2 (CO_2 FREE)

10 ml of 7.353x10^{-5} M dye
300 ml of 0.02 M NaOH
Temperature 25^{o} C

Date: 1-20-88
Ao = 0.2377
C_{Bo} = 0.0194

Time(min)	Transmittance	Absorbance	-ln A/Ao
1	0.639	0.1945	0.2006
2	0.698	0.1561	0.4205
3	0.729	0.1373	0.5488
4	0.774	0.1113	0.7588
5	0.793	0.1007	0.8589
6	0.832	0.0799	1.0902
7	0.854	0.0685	1.2442
8	0.882	0.0545	1.4728
9	0.904	0.0438	1.6914
10	0.910	0.0410	1.7574

Correlation coeff. = 0.9950, Intercept = 0.0305, Slope = 0.1771

VITA

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Master of Science

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