

main runs.

It should also be noted that these experiments gave a measure of the accuracy of the chosen value for the quenched stream pH. Since the runs agreed to better than 99% with the theoretical, the pH value chosen was correct. (For methods used to obtain the pH, see Appendix vi.5).

APPENDIX v

The residence time distribution measurements enabled a value to be obtained for the reactor holdup volume, and it was felt that this should be checked by weighing.

1 EXPERIMENTAL PROCEDURE

All air bubbles were removed from the top of the column and the column flow lines. The column outlet plug was removed and the column was flushed out with pure distilled water for 10 minutes at maximum flow. The temperature of the water was recorded. The outlet plug was replaced, the inlet lines were removed and the column inlets were plugged. The column was then detached from the supporting frame, all water was drained from the quench vessel, and this was carefully dried together with all column externals. The entire reactor was then weighed, together with an empty 5 litre beaker.

The beads and water were emptied into the beaker, and the beaker was placed in an oven at 150°C. The column interior and all the fittings were carefully dried with compressed air. When all the water had been evaporated from the beads, these were weighed again together with the column.

Unfortunately the only balance available for weighing the column was a 0-500 lb. scale, which could be read in increments of 4 ounces.

2 EXPERIMENTAL RESULTS

The results obtained on the 149.8 cm. and 73.0 cm. lengths are given in Table v.1 below.

TABLE v.1

<u>Length of Reactor</u>	<u>Holdup Volume (litre)</u>					<u>Mean (litre)</u>
149,8	1,41	1,50	1,44	1,38	1,43	
73,0	0,718	0,722	0,710	0,715	0,716	

The holdup volume obtained from residence time distribution measurements on the 149,8 cm. length was 1,472 litres, which differs from the above by 2,8%. It was thus verified that the two methods gave holdup volumes in the same region, and since the residence time distribution measurements appeared to be more accurate than those above, the holdup was taken to be 1,472 litres for the 149,8 cm. length.

For the 73,0 cm. column, however, holdup volumes from residence time distribution measurements were not available, (see 6.1), so the above value of 0,716 litres was used. This value did not cause the experimental results to differ greatly from those predicted by the plug flow model (see Chapter 9).

APPENDIX vi

1. COMPLETE ANALYSIS PROCEDURE1.1 Diacetate Feed (Stream 1)

Referring to the reactor flowsheet on figure 5.2, it will be seen that the composition of this stream was:-

Diacetate	X_{1s} moles/litre
Monoacetate	X_{3s} moles/litre
Glycol	X_{5s} moles/litre
Acetic acid	X_{6s} moles/litre
Volumetric flow rate	Q_1 litres/second
Mass flow rate	G_1 kg/second
Density	ρ_1 kg/litre

The analysis procedure was as follows:-

(i) Acetic Acid (X_{6s})

A sample containing a grams of stream 1 was titrated with c molar standard caustic soda. Assume the density of the caustic was ρ_7 , and that b grams were required for the titration. Then:-

$$\frac{(X_{6s})(a)}{\rho_1} = \frac{bc}{\rho_7}$$

Hence,

$$X_{6s} = \frac{bc\rho_1}{a\rho_7} \text{ moles/litre}$$

(ii) Total Acetate ($2X_{1s} + X_{3s} + X_{6s}$)

e grams of c molar standard caustic soda were added to a sample containing d grams of stream 1. This was allowed to stand until saponification was complete, then backtitrated with f molar standard hydrochloric acid. Assume that the density of the hydrochloric acid was ρ_8 and that h grams were required for the back-titration.

$$\text{Mole equivalents of NaOH used up} = \frac{(2X_{1s} + X_{3s} + X_{6s})d}{\rho_1}$$

$$\text{Hence mole equivalents left} = \frac{ea}{\rho_7} - \frac{(2X_{1s} + X_{3s} + X_{6s})d}{\rho_1}$$

This is equivalent to the HCl used.

Hence,

$$\frac{hj}{\rho_8} = \frac{ea}{\rho_7} - \frac{(2X_{1s} + X_{3s} + X_{6s})d}{\rho_1}$$

Hence,

$$2X_{1s} + X_{3s} + X_{6s} = \left(\frac{ea}{\rho_7} - \frac{hj}{\rho_8} \right) \frac{\rho_1}{d}$$

2

$$(iii) \quad \text{Total Glycol } (X_{1s} + X_{3s} + X_{6s})$$

If the sample was saponified to completion, all the diacetate and monoacetate would be converted to glycol and this could be analysed for by periodate oxidation. Sufficient caustic soda was added to a sample of l grams of stream 1 to cause complete saponification. When the saponification was complete, the solution was neutralised with hydrochloric acid, and m grams of periodic acid solution were added. Assume that the periodic acid solution contained p' gm.moles per litre of periodic acid and p'' gm.moles per litre of iodic acid. (This is unavoidable, because the periodic acid decomposes on standing.) After the oxidation was complete, tg grams of r molar potassium iodide solution were added, followed by tg grams of r molar hydrochloric acid. (g and r must be large enough to liberate all the iodine.) The liberated iodine was then backtitrated using a grams of u molar standard sodium thiosulphate solution.

$$\text{As before, sample density} = \rho_1$$

$$\text{periodic acid density} = \rho_8$$

$$\text{thiosulphate density} = \rho_{10}$$

Put

$$X_{1s} + X_{3s} + X_{6s} = X'_6$$

Hence, mole equivalents of glycol in sample = $\frac{(X' \cdot 5)L}{\rho_1}$

Hence, from reaction I, (see 8.4),

$$\text{periodic acid reduced} = \frac{(X' \cdot 5)L}{\rho_1} = \text{iodic acid formed} \quad 3$$

$$\text{Now, mole equivalents of periodic acid in solution} = \frac{P'm}{\rho_g} \quad 4$$

$$\text{and mole equivalents of iodic acid in solution} = \frac{P''m}{\rho_g} \quad 5$$

Periodic acid left after oxidation, from equations 3 and 4:-

$$\frac{P'm}{\rho_g} - \frac{(X' \cdot 5)L}{\rho_1}$$

Iodic acid left after oxidation, from equations 3 and 5:-

$$\frac{P''m}{\rho_g} + \frac{(X' \cdot 5)L}{\rho_1}$$

When acidic potassium iodide is added, from equation II, (section 8.4), 1 gram molecule of periodic acid is equivalent to 8 gram atoms of iodine. When acidic potassium iodide is added, from equation III, section 8.4), 1 gram molecule of iodic acid is equivalent to 6 gram atoms of iodine. Hence atom equivalents of iodine liberated in solution =

$$8\left(\frac{P'm}{\rho_g} - \frac{L(X' \cdot 5)}{\rho_1}\right) + 6\left(\frac{P''m}{\rho_g} + \frac{L(X' \cdot 5)}{\rho_1}\right) \quad 6$$

Now each atom of iodine is equivalent to a molecule of thiosulphate

$$\text{From above, mole equivalents of thiosulphate} = \frac{8M}{\rho_{TC}} \quad 7$$

Hence from 6 and 7 above:-

$$\frac{8M}{\rho_{TC}} = 8\left(\frac{P'm}{\rho_g} - \frac{L(X' \cdot 5)}{\rho_1}\right) + 6\left(\frac{P''m}{\rho_g} + \frac{L(X' \cdot 5)}{\rho_1}\right) \quad 8$$

Hence, mole equivalents of glycol in sample = $\frac{(X'_5)L}{\rho_1}$

Hence, from reaction I, (see 8.4),

periodic acid reduced = $\frac{(X'_5)L}{\rho_1}$ = iodic acid formed

Now, mole equivalents of periodic acid in solution = $\frac{P'm}{\rho_g}$

and mole equivalents of iodic acid in solution = $\frac{P'm}{\rho_g}$

Periodic acid left after oxidation, from equations 3 and 4:-

$$\frac{P'm}{\rho_g} - \frac{(X'_5)L}{\rho_1}$$

Iodic acid left after oxidation, from equations 3 and 5:-

$$\frac{P'm}{\rho_g} + \frac{(X'_5)L}{\rho_1}$$

When acidic potassium iodide is added, from equation II, (section 8.4), 1 gram molecule of periodic acid is equivalent to 8 gram atoms of iodine. When acidic potassium iodide is added, from equation III, section 8.4), 1 gram molecule of iodic acid is equivalent to 6 gram atoms of iodine. Hence atom equivalents of iodine liberated in solution =

$$8\left(\frac{P'm}{\rho_g} - \frac{L(X'_5)}{\rho_1}\right) + 6\left(\frac{P'm}{\rho_g} + \frac{L(X'_5)}{\rho_1}\right)$$

Now each atom of iodine is equivalent to a molecule of thiosulphate

From above, mole equivalents of thiosulphate = $\frac{8M}{\rho_{10}}$

Hence from 6 and 7 above:-

$$\frac{8M}{\rho_{10}} = 8\left(\frac{P'm}{\rho_g} - \frac{L(X'_5)}{\rho_1}\right) + 6\left(\frac{P'm}{\rho_g} + \frac{L(X'_5)}{\rho_1}\right)$$

$$\text{Hence } \frac{\partial u}{\partial p_{10}} = \frac{m}{\rho_g} (\partial p' + \partial p'') - \frac{\partial I(X', S)}{\partial p_1}$$

$$\text{Put } I = \partial p' + \partial p''$$

$$\text{Hence, } X'_S = \left(\frac{m}{\rho_g} - \frac{\partial u}{\partial p_{10}} \right) \frac{\partial I}{\partial I}$$

In order to determine $\frac{\partial u}{\rho_g}$ a blank was done as follows:-

v grams of periodic acid were taken, and after liberating all the iodine with hydrochloric acid and potassium iodide according to reactions II and III, (see 8.4), the solution was titrated with w molar standard sodium thiosulphate solution. Assume that w grams of thiosulphate were used.

As above, periodic acid in sample = $\frac{P'v}{\rho_g}$ mole equivalents

As above, iodic acid in sample = $\frac{P''v}{\rho_g}$ mole equivalents

thiosulphate used = $\frac{wM}{\rho_{10}}$ mole equivalents

As before, therefore, $\frac{wM}{\rho_{10}} = \frac{\partial p'v}{\rho_g} + \frac{\partial p''v}{\rho_g}$

Hence, $\frac{wM}{\rho_{10}} = \frac{v}{\rho_g} (\partial p' + \partial p'')$

From equation 9, $\partial p' + \partial p'' = P$

Hence, $\frac{P}{\rho_g} = \frac{wM}{v\rho_{10}}$

This can now be substituted back into equation 10 to give:-

$$X'_S = \left(\frac{wM}{v\rho_{10}} - \frac{\partial u}{\rho_{10}} \right) \frac{\partial I}{\partial I}$$

$$\text{Hence } X'_S = \frac{w\rho_1}{\partial I\rho_{10}} \left(\frac{wM}{v} - \rho \right)$$

This equation will apply to any sample analysed for glycol.

In this case, $X'_S = X_{1g} + X_{3g} + X_{6g}$, hence,

$$X_{1g} + X_{3g} + X_{5g} = \frac{40}{2160} \left(\frac{100}{10} - a \right)$$

13

See notes on insensitivity of method in section 2 of this appendix.

(iv) Free Glycol (X_{5g})

The concentration of the free glycol was obtained from equation 12 in exactly the same way as for the total glycol, except of course that the feed stream was analysed directly for glycol with no prior saponification. (See 8.4 for discussion of saponification of diacetate in solution during this analysis, and section 3 of this appendix for the test of the method.)

(v) Final Calculation of all concentrations in Stream 1

$$\begin{array}{ll} \text{From (i) } X_{6g} & = \text{known} \\ \text{From (ii) } 2X_{1g} + X_{3g} + X_{6g} & = \text{known} \\ \text{From (iii) } X_{1g} + X_{3g} + X_{5g} & = \text{known} \\ \text{From (iv) } X_{5g} & = \text{known} \end{array}$$

14

Equations 14 are four independent algebraic equations in four unknowns. They were solved to give X_{1g} , X_{3g} , X_{5g} and X_{6g} .

1.2 Caustic Feed (Stream 2)

From Figure 5.2 it will be seen that the composition of this stream was:-

Caustic soda	X_{2g} moles/litre
Volumetric flowrate	Q_g litre./second
Mass flowrate	G_g kg/second
Density	ρ_g kg/litre

A sample of x grams of stream 2 was titrated with f molar standard hydrochloric acid. Assume that y grams of hydrochloric acid, density ρ_g , were used.

As before,

$$\frac{x_{2s} x}{\rho_s} = \frac{y_i}{\rho_g}$$

Hence

$$x_{2s} = \frac{y_i \rho_g}{x \rho_s}$$

15

1.3 Reactor Inlet Stream (Stream 3)

From Figure 5.2, this stream contained:-

Diacetate	x_{10} moles/litre
Caustic soda	x_{20} moles/litre
Monoacetate	x_{30} moles/litre
Glycol	x_{60} moles/litre
Sodium acetate	Not analysed for
Volumetric flow rate	Q_3 litres/second
Mass flow rate	G_3 kg/second
Density	ρ_3 kg/litre

Since the feed concentrations used were low and the feeds were at the same temperatures, it was assumed that temperature and density changes when the streams mixed were negligible (3.1). The inlet line was too short to permit sampling, so the composition of Stream 3 had to be obtained by mole balance, (see Figure 5.2).

Balance for caustic

$$Q_3 x_{20} = Q_2 x_{2s} - Q_1 x_{2s}$$

therefore

$$x_{20} = \frac{Q_2 x_{2s} - Q_1 x_{2s}}{Q_3}$$

Balance for diacetate

$$Q_3 x_{10} = Q_1 x_{1s}$$

therefore

$$x_{10} = \frac{Q_1}{Q_3} x_{1s}$$

Balance for monoacetate similarly

$$x_{30} = \frac{Q_1}{Q_3} x_{3s}$$

Balance for glycol similarly

$$x_{60} = \frac{Q_1}{Q_3} x_{6s}$$

Since all the right hand sides of these expressions were known, Stream 3 composition was known.

16

1.4 Reactor Outlet Stream (Stream 4)

From Figure 5.2, the composition of this stream was:-

Diacetate	X_{1f} moles/litre
Caustic soda	X_{2f} moles/litre
Monoacetate	X_{3f} moles/litre
Glycol	X_{5f} moles/litre
Sodium acetate	Not analysed for
Volumetric flow rate	Q_4 litres/second
Mass flow rate	G_4 kg/second
Density	ρ_4 kg/litre

The outlet line was also too short to permit sampling, so the concentration in this stream had to be calculated by mole balances too.

(i) Caustic Soda (X_{2f})

The stream leaving the reactor was neutralised exactly with the acetic acid quench stream, which flowed at Q_5 litres per minute and contained X_{6s} molar acetic acid.

Hence by acid-base balance $Q_4 X_{2f} = Q_5 X_{6s}$

Therefore
$$X_{2f} = \frac{Q_5 X_{6s}}{Q_4}$$

17

The analysis for X_{6s} is given in the following section, the flows were known, hence X_{2f} could be obtained.

(ii) Total Glycol ($X_{1f} + X_{3f} + X_{5f}$)

The total glycol present in the inlet would be the same as that present in the outlet, since this quantity would be unchanged by the reaction. If it is assumed that the feed streams were sufficiently dilute to cause negligible density and temperature changes on reaction, then $Q_4 = Q_5$

18

and
$$X_{1f} + X_{3f} + X_{5f} = X_{1o} + X_{3o} + X_{5o}$$

19

X_{1o} , X_{3o} and X_{5o} were known (see section 1.3 in this appendix), and hence the left hand side of equation 19 was known.

(iii) Saponifiable Acetate ($2x_{1f} + x_{3f}$)

If the saponifiable acetate present in Stream 6 were known, then the above could be calculated by simple mole balance:-

$$(2x_{1f} + x_{3f})Q_g = (2x_{1n} + x_{3n})Q_g$$

From 18:- $2x_{1f} + x_{3f} = \frac{Q_g}{Q_s}(2x_{1n} + x_{3n})$

20

The total acetate $2x_{1n} + x_{3n}$ was obtained in the same way as for Stream 1, section 1.1 (ii) of this appendix.

(iv) Free Glycol (x_{5f})

As above,

$$x_{5f} = \frac{Q_g}{Q_s} x_{5n}$$

21

x_{5n} was analysed for in the same way as x_{5a} for Stream 1, (see section 1.1 (iv) of this appendix).

(v) Final Calculation of all concentrations in Stream 4

$$\begin{array}{ll} \text{From (i)} & x_{2f} = \text{known} \\ \text{(ii)} & x_{1f} + x_{3f} + x_{5f} = \text{known} \\ \text{(iii)} & 2x_{1f} + x_{3f} = \text{known} \\ \text{(iv)} & x_{5f} = \text{known} \end{array}$$

22

As for Stream 1, equations 22 present four equations in four unknowns, so x_{1f} , x_{2f} , x_{3f} and x_{5f} could be calculated.

1.5 Acetic Acid Quench (Stream 5)

From Figure 5.2, the composition of this stream was:-

Acetic acid	x_{6a} moles/litre
Volumetric flowrate	Q_5 litres/second
Density	ρ_5 kg/litre

A sample of a grams of Stream 5 was titrated with σ molar standard caustic soda. Assume that β grams of caustic (density ρ_p) were required.

As before,
$$\frac{(X_{6a})_a}{\rho_5} = \frac{80}{\rho_7}$$

Therefore,
$$X_{6a} = \frac{80\rho_5}{\rho_7}$$

23

1.6 Neutralised Stream (Stream 6)

From Figure 5.2, the composition of this stream was:-

Diacetate X_{1n} moles/litre

Monoacetate X_{3n} moles/litre

Glycol X_{5n} moles/litre

Sodium acetate Not analysed for.

Note: If the quench is not perfect, this stream could contain sodium hydroxide or acetic acid, so initially an acid-base titration was performed on it. The acidity or alkalinity was always found to be negligible, however, so this procedure was later discontinued.)

It will be seen from sections 1.4 (iii) and (iv) above that this stream only had to be analysed for total acetate and free glycol.

(i) Saponifiable Acetate ($2X_{1n} + X_{3n}$)

This was measured in exactly the same way as for Stream 1, see section 1.1 (ii) of this appendix.

(ii) Free Glycol (X_{5n})

This was measured in exactly the same way as for Stream 1, section 1.1 (iv) of this appendix. The above quantities could now be used to solve equations 22.

2. INSENSITIVITY OF GLYCOL ANALYSIS

If sufficient periodic acid is added to oxidise all the glycol in a stream 1 sample, then from equations 3 and 4:-

$$\frac{p'm}{\rho_g} \geq \frac{(x'_s) l}{\rho_l}$$

Substitute this into equation 8:

$$\text{Hence } \frac{wm}{\rho_{10}} \geq \frac{\delta m}{\rho_g}(p' + p'') \quad 24$$

From equation 11:-

$$\frac{wm}{\rho_{10}} = \frac{v}{\rho_g}(\delta p' + \delta p'') \quad 11$$

$$\frac{\text{equation 24}}{\text{equation 11}}: \quad \frac{g}{w} \geq \frac{\delta m(p' + p'')}{v(\delta p' + \delta p'')} \quad 25$$

now g = weight of thiosulphate used to titrate liberated iodine
after oxidation of sample

w = weight of thiosulphate used to titrate iodine liberated
from periodic acid blank

m = weight of periodic acid added for oxidation of sample

v = weight of periodic acid taken for blank

p' = moles per litre of periodic acid present in periodic
acid solution

p'' = moles per litre of iodic acid present in periodic
acid solution.

m and v were made equal so that one pipette could be used for adding periodic acid for oxidising and blanks. Also, the iodic acid was liable to be present in small amounts compared to the periodic acid.

Hence equation 25 can be approximated by:-

$$\frac{g}{w} \geq \frac{3(p')}{8(p')}$$

Therefore,

$$\frac{g}{w} \geq 0.75 \quad 26$$

Now, from equation 12,

$$X'_s = \frac{w}{210} \left(\frac{100}{v} - a \right)$$

If $m = v$ as above, then,

$$X'_s = \frac{w}{210} (w - a)$$

27

An example using equations 26 and 27 will be used to demonstrate the insensitivity of the method.

Assume that $w = 20$ grams.

then $a_{min.} = (0.75)(w) = 15$ grams.

The balance could be read to an accuracy of 0.01 grams. Hence each weighing can have a maximum error of 0.02 grams.

$$\text{Percentage error on } w = \frac{(0.02)(100)}{20} = 0.1\%$$

$$\text{Percentage error on } a = \frac{(0.02)(100)}{15} = 0.133\%$$

When these two are subtracted, however, $(w - a)_{max} = 5$ grams, and maximum error is 0.04 grams. From 27, percentage error on $X'_s =$

$$\frac{(0.04)(100)}{5} = 0.8\%$$

The percentage error on X'_s is thus nearly 8 times that on w and a .

In order to insure that there was always sufficient periodic acid present to complete the oxidation, it was decided that a/w would be set at 0.8. Since the limit on the balance without a range change was 100 grams, it was decided that

$$w = 100 \text{ grams}$$

$$\text{Hence for } a/w = 0.8$$

$$a = 80 \text{ grams}$$

The percentage error on glycol concentration would thus be reduced to:-

$$\frac{(0.04)(100)}{20} = 0.2\%$$

3. CHOICE OF OXIDATION TIME AND TEST OF ACCURACY OF ANALYSES

As discussed earlier (section 8.4) the periodic oxidation time had to be chosen so as to achieve maximum oxidation of glycol in the samples and minimum saponification of mono- and diacetate. The method would be expected to be least accurate when solutions with high diacetate, and low glycol concentrations were being measured.

A solution was thus made up in which the diacetate concentration was roughly four times that of the glycol, and this was analysed repeatedly with varying oxidation times. The results are given in Table A.vi.1 below:-

TABLE A.vi.1

Solution made up accurately to contain 0.103 molar diacetate

0.02455 molar glycol

<u>Oxidation Time</u>	<u>Measured Glycol Concentration (Molar)</u>
15	0.02463
20	0.02459
30	0.02450
45	0.02439
60	0.02453
90	0.02446
120	0.02460

Two points become evident from the table:-

- (i) Since the standard deviation of the above concentrations from the correct value is only 0.00008, the glycol analysis procedure was sufficiently accurate for use in this work.
- (ii) The oxidation times above have no effect on the glycol concentration, and the oxidation time during analy-

sis is therefore unimportant provided it is greater than 15 minutes. In practice, due to the fact that many analyses were done in succession, the oxidation time was roughly 30 minutes per sample.

All other analyses were tested similarly and found to be acceptable, except the analysis of mixtures of diacetate and monoacetate and glycol. Since pure monoacetate could not be obtained to make up solutions with known concentrations, the analysis methods for these mixtures could not be tested.

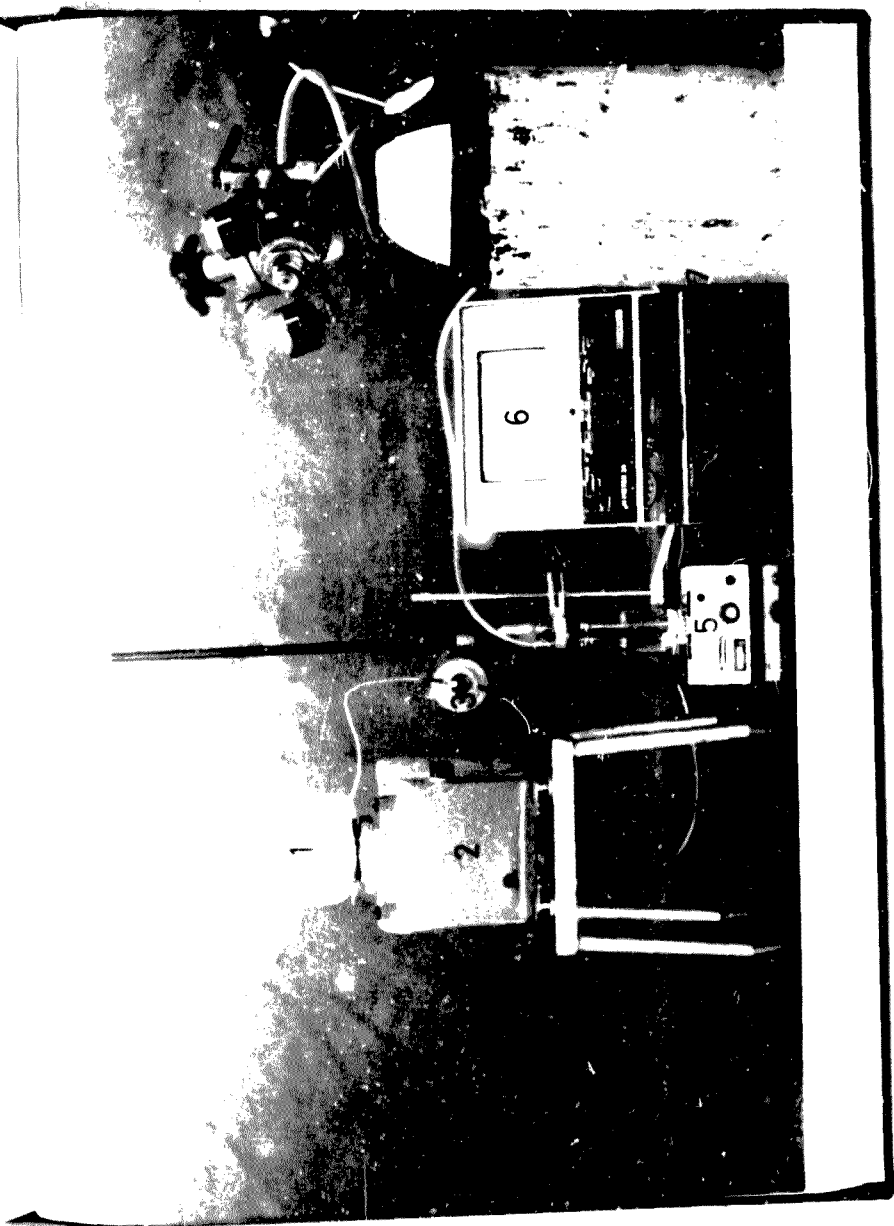
A discussion of possible errors and error growth in the analyses is given in section 8.5.

4. TITRATION APPARATUS

The photograph on the following page shows the experimental apparatus used for titrations.

The polythene bottle, 1, was filled with the standard solution to be used in the titration and was placed on the balance, 2. The standard solution was supplied through a 2mm. polythene tube welded into the bottom of the bottle. This tube, in turn, was inserted into a 3mm. diameter rubber tube which terminated in a nylon titration nozzle. The rubber tubing passed through an on-off solenoid valve, 3, which could pinch the tubing closed to cut off the flow of standard solution through the titration nozzle into the titration beaker, 4. A screw on the solenoid valve also enabled the delivery rate of standard solution to be controlled when the tube was open. Nylon was chosen for the titration nozzle because this substance is non-wettable, and the nozzle was carefully shaped and drilled to produce the smallest drop size possible.

The titration electrodes, nitrogen bleed tube, and titration



nozzle, were all fitted into a holder which could be raised and lowered into the titration beaker. Teflon covered stirring bars were placed in the titration beakers, and a magnetic stirrer, 5, with adjustable speeds was used to stir the titration mixture.

The pH meter, 6, read both pH and mV. For pH measurements a combined glass-calomel electrode was used in conjunction with a temperature sensing element which enabled the meter to compensate for pH changes due to temperature increases or decreases during the titrations. For mV measurements a calomel-platinum electrode pair was used.

The pH scale was calibrated from 0 to 14 in steps of 0.1 pH, and the reading could be expanded by 10 at any pH, so that the meter could be read to 0.01 pH. Similarly, the mV scale was calibrated from 0-1400 mV in steps of 10 mV, and could also be expanded by 10 to give steps of 1 mV. The pH meter was provided with two output sockets, one giving 0.1 mA per pH or per 100 mV, and the other giving a 10 mV per pH or per 100 mV output for a recorder.

The recorder output from the pH meter provided the input for the automatic titrator, 7. The method for determining the titration endpoints is given below, and once these were known, an "end point" knob on the titrator enabled the pH meter scale to be used to set the titrator to the desired endpoint of the titration. Both the pH and mV normal and expanded scales could be used for this purpose, so the pH titration endpoint could be set to within 0.01 pH and the potentiometric titration endpoints to within 1 mV. It was found that the mV titration range was so narrow that all potentiometric titrations had to be done on expanded scale. The pH titrations, however, provided a wide enough range to enable them to be carried out using the normal scale.

During the titrations, the pH meter output is fed to the

automatic titrator and the difference between this value and the set point above gives rise to an error signal in the titrator which is amplified and applied to a switch circuit. The switch circuit energizes the solenoid valve, and so controls the flow of standard solution into the titration beaker. The design of the titrator provides for an average flow of standard solution which is proportional to the error in the proportional band range. The solenoid valve only provides on-off control. This meant, in practice, that when the pH meter approached to within the proportional band of the desired titration endpoint, the solenoid valve would switch on and off with increasing frequency, finally switching off when the endpoint was reached.

The choice of proportional band setting was influenced both by the delivery rate of standard solution when the solenoid valve was open, and the stirring rate of the titration mixture. When the level of standard solution in the supply bottle dropped, the flow from the titration nozzle diminished proportionately, and it was found that a narrower proportional band could be used than when the supply bottle was full. Further, if the titration mixture had to be stirred slowly to avoid splashing, then the proportional band width had to be increased to prevent the standard solution from being added too rapidly and so overshooting the endpoint. The proportional band width and setting of the screw on the solenoid valve were thus both altered continuously during titrations in order to obtain the desired endpoints.

5. DETERMINATION OF TITRATION ENDPOINTS

A 0,715 ohm resistor was connected across the 0,1 mA per pH or per 100 mV socket on the pH meter. This provided a voltage of 0 to 1,0 mV for pH meter scale readings of 0 to 14 pH or 0 to

1400 mV. It was found that this output voltage depended on the position of the pH meter needle on the scale, so that even when the scale of the pH meter was expanded, the recorder still read 0 to 1.0 mV for fullscale meter deflection.

The automatic titrator was disconnected and the correct electrodes were placed in the pH meter, and it was set to read in the desired range. If pH titrations were being done, it was zeroed using standard buffer solution supplied by Radiometer. The recorder was zeroed at the same time using a zero suppression potentiometer. Solutions were then made up which corresponded to the concentrations of standards and unknowns to be used in any of the other experiments.

The standard solutions were placed in the supply bottle and the unknowns in the titration beaker. The nitrogen bleed, stirrer, and recorder were all turned on and standard solution was allowed to flow into the beaker until the complete titration curve had been traced out by the recorder. It was assumed that the drop in the liquid level in the supply bottle would be small enough to ensure a constant flow of standard solution during the titration. The time scale on the recorder trace would thus be directly proportional to the volume of titre added.

Several typical titration curves are shown in Figures A.vi.1-5. The midpoint of the section with the steepest slope on these curves was taken as the endpoint of the titration. It should be noted that the recorder chart drive was only turned on when the pen began to accelerate, so the position of each curve with respect to the time axis is different in each case. The flows through the solenoid valve were kept constant for each titration curve, and all the curves are plotted on the same time scale to give an idea of the sharpness of the endpoints.

Due to the presence of sodium acetate buffer, and also due

to the dilution effect, the saponification backtitrations, and final quench titration endpoints were less steep than the straight acid-base titration for stream 5. The potentiometric titration endpoints were less sharp than the pH titration endpoints with the dilution effect making the glycol determination titration endpoints the least steep of all. Figure A.vi.1 shows the titration curve used to obtain the pH which was to be maintained in the outlet quench vessel.

6. TITRATION PROCEDURE FOR MAIN RUNS

"Volucon" primary standard solutions were used throughout this work. They are guaranteed accurate to 99.8%. The solutions used were 0.1 and 1 molar sodium hydroxide and 0.1 and 1 molar hydrochloric acid and 0.1 molar iodine. Certificated A grade standard flasks were used to make up the solutions and they were always made up in the constant temperature room at 20°C. These primary standards were used in as many of the analyses as possible, but if a large volume of standard solution was required for a large number of titrations, then this solution was made up and standardised with the Volucon solution.

All standards were used for a maximum of two days, before being discarded, and fresh ones being prepared.

For solutions where the composition had to be known very accurately, as, for example, the periodic acid oxidising solution, eight samples were taken, otherwise four samples were taken of all unknown solutions. If the results did not agree to better than 99%, the analysis was repeated. All titration beakers were fitted with rubber stoppers wrapped in aluminium foil, and where necessary, the samples were weighed out and allowed to stand in a nitrogen atmosphere. If a large number of samples all of approximately the same concentration were to be titrated, these were all weighed out,

stoppered, and then titrated one after the other, so that the standard solution did not have to be removed from the balance between titrations.

The automatic titrator was connected to the pH meter and the desired end point and proportional band set. The rest of the titration procedure was the same as that given in 5 above, except that the titrator turned the solenoid valve off at the end of the titration and the end pH or mV reading was recorded, together with the weight of standard solution used in the titration.

The titration procedure was found to work extremely well, giving better results and enabling the titrations to be done quicker than those done by hand.

11 - pH

TITRATION OF REACTOR OUTLET STREAM WITH ACETIC ACID(to find pH for outlet
(quench vessel))

10

9

8,38

8

7

6

5

0

10

20

30

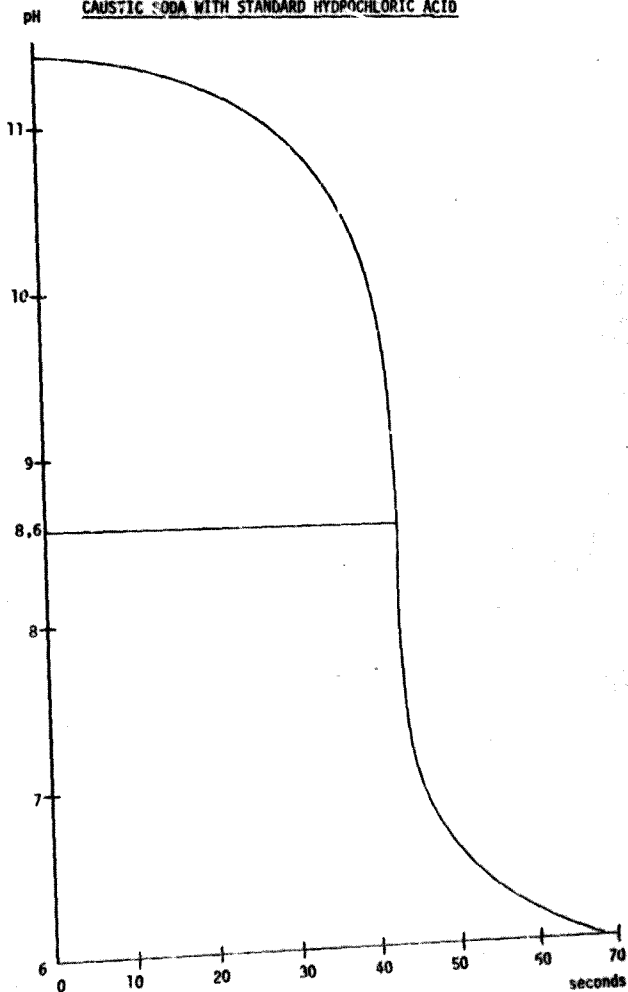
40

50

60

seconds

FIGURE A.vi.1

CAUSTIC SODA WITH STANDARD HYPOCHLORIC ACIDFIGURE A.v1.2

TITRATION OF 5% ACETIC ACID WITH
STANDARD CAUSTIC SODA

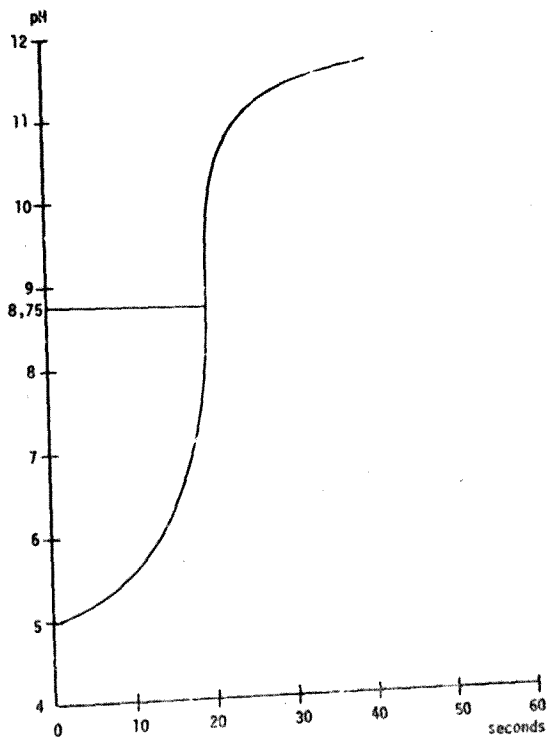
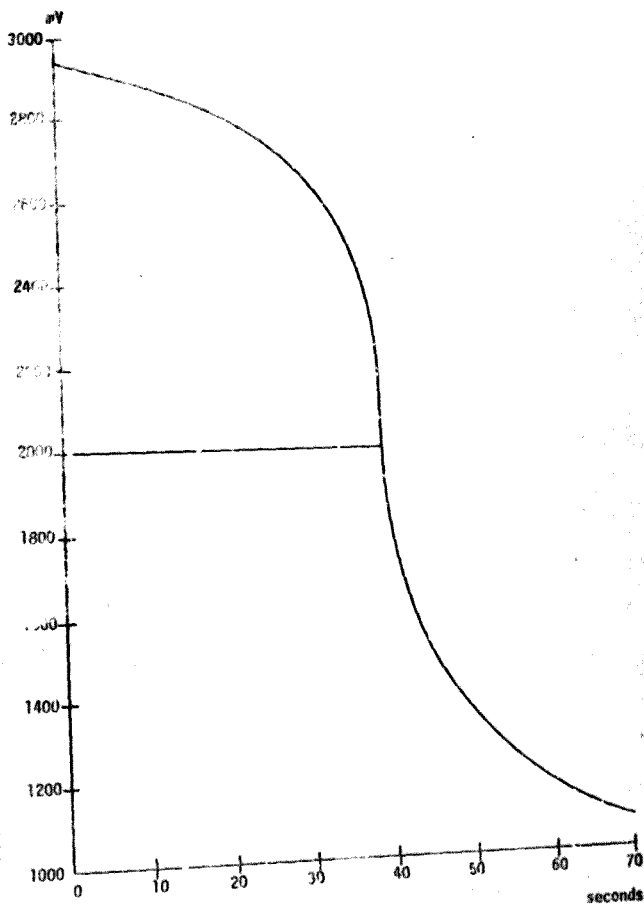


FIGURE A.v1.3

TITRATION OF THIOSULPHATE WITH STANDARD IODINE

(iodine in titration beaker)

FIGURE A.v1.4

TITRATION OF IODINE LIBERATED DURING GYCOL
ANALYSIS WITH THIOSULPHATE

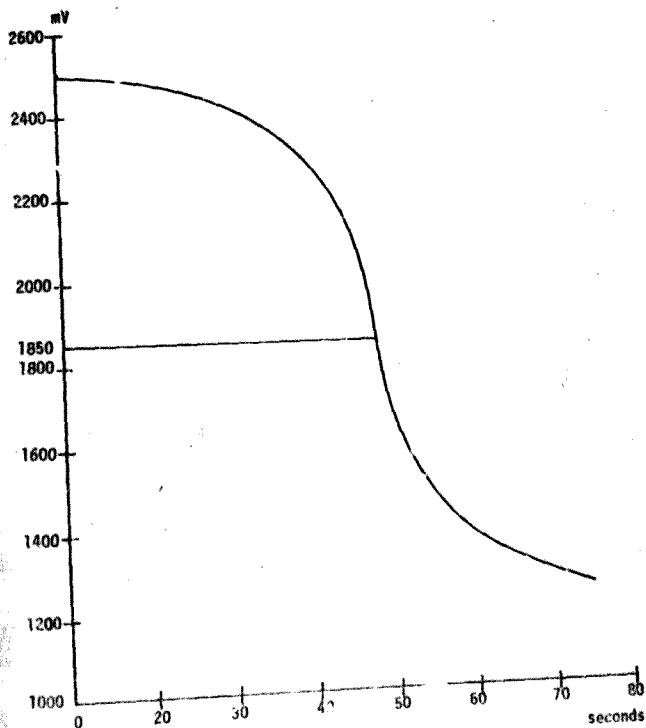


FIGURE A.vi.5

APPENDIX vii

Note: Meyer's nomenclature and equation numbers will be used throughout this appendix.

The consecutive reactions may be written:-



Let the initial concentration of diester be A moles per litre, and of caustic soda be B moles per litre.

After time t , assume that moles per litre of diester reacted = x , and that moles per litre of caustic soda reacted = w .

Hence diester concentration = $(A - x)$

caustic concentration = $(B - w)$

Further, assume that y moles per litre of monoester have formed during time t , and z moles per litre of alcohol. Then, by mole balance,

$$x = y + z \quad 6$$

$$\text{and } w = y + 2z = x + z = 2x - y \quad 7$$

The rate equations for diester and monoester become:-

$$\frac{d(A - x)}{dt} = -\frac{dx}{dt} = -k_1(A - x)(B - w) \quad 8$$

$$\text{and } \frac{dy}{dt} = k_1(A - x)(B - w) - k_2(B - w)y \quad 9$$

with initial conditions at $t = 0$, $x = 0$, $w = 0$, $y = 0$.

Meyer was able to use equations 7, 8 and 9 with the above boundary conditions to show that:-

$$y = \frac{k_1}{k_1 - k_2} \left(A + \frac{k_1 - 2k_2}{k_1} x - A \frac{k_1 - k_2}{k_2} (A - x) \frac{k_2}{k_1} \right) \quad 25$$

Meyer was not able to solve this equation for k_1 or k_2 , and he thus assumed that $k_1 = 2k_2$.

Equation 25 could thus be reduced to:

$$A - x = \frac{1}{4A}(2A - w)^2 \quad (27)$$

and when this was substituted into equation 8, the latter could be solved to give:-

$$k_2 = \frac{1}{t} \frac{1}{(2A - B)} \ln \frac{B(2A - w)}{2A(B - w)} \quad (28)$$

In this equation:-

A = initial concentration of diester at $t = 0$

B = initial concentration of caustic at $t = 0$

w = caustic used up in reaction at $t = t$

k_2 = rate constant of reaction II

Hence Meyer was able to solve for k_2 from his experimental runs.

APPENDIX viiiEXPERIMENTAL RESULTS OF RESIDENCE TIME DISTRIBUTIONMEASUREMENTS

As discussed in 6.1 and 6.2, these results were obtained in the form of a recorder trace of millivolts versus time, and were equivalent to the $C(t)$ versus time functions discussed by Danckwerts (1953).

The millivolt scale on the chart paper contained 100 divisions, so one division was taken as one unit of $C(t)$, and the first decimal place was estimated. An overlay was milled out of perspex, by means of which the time scale could be read in intervals of 1/20 inch. Since the chart speeds were varied for some of the runs, the interval at which points were read off the recorder traces was varied as well.

The tables on the following pages give the data read off the above recorder curves. In the tables, the time intervals between successive points are given, as well as the baseline $C(t)$ value at the start of each run. The number of base points for each run represents the number of points on the recorder trace between the injection point and the point where the $C(t)$ curve left the baseline.

The $C(t)$ values reported for Runs 27-36 had to be multiplied by 10 to convert them to the same scale as those for Runs 1-20.

RUN NO. 1

Baseline: 3,3. No. of base points: 201. Total no. of points: 420.

Flowrate: 0,5509 litres/minute. Seconds per point: 0,0572.

3,3	3,3	3,4	3,5	3,9	4,3	4,6	5,3	6,1	6,7	7,2	7,7	8,3	9,0
9,8	10,4	11,3	12,1	13,1	14,0	15,1	16,0	16,7	18,2	19,1	20,6	22,5	24,6
26,3	27,8	29,4	31,3	32,9	34,3	36,6	38,3	40,1	43,0	45,0	47,0	49,3	51,2
52,8	55,3	57,9	60,3	62,7	65,0	67,2	68,9	70,6	72,7	74,8	77,2	79,4	81,3
83,1	84,7	86,0	87,3	88,4	90,1	91,1	92,3	93,3	94,5	96,0	96,9	97,5	97,9
98,4	98,9	99,0	99,1	99,0	99,0	98,9	98,4	98,0	97,5	97,0	96,2	95,5	94,7
94,1	93,2	91,7	90,7	89,6	88,3	86,0	83,2	82,8	81,3	79,3	77,8	76,5	75,3
73,4	71,2	70,3	67,9	66,0	64,3	62,3	60,3	58,8	57,4	56,3	54,4	52,5	50,4
48,7	47,3	45,9	43,7	42,0	40,6	40,0	38,9	37,8	35,7	34,8	32,9	31,1	29,9
28,9	27,8	26,6	25,5	24,6	23,7	22,9	22,2	21,7	21,2	20,7	20,0	19,2	18,1
17,5	16,9	15,9	15,0	14,3	13,6	12,9	12,3	11,9	11,4	11,0	10,4	10,0	9,7
9,3	9,0	8,6	8,2	8,1	7,9	7,6	7,3	7,1	6,9	6,7	6,4	6,3	6,1
6,0	6,0	5,9	5,7	5,4	5,3	5,2	5,1	5,1	5,1	5,0	5,0	5,0	5,0
5,0	4,9	4,9	4,8	4,8	4,8	4,8	4,7	4,7	4,7	4,6	4,6	4,5	4,5
4,4	4,4	4,3	4,3	4,3	4,2	4,2	4,2	4,2	4,2	4,2	4,1	4,1	4,1
4,1	4,1	4,0	4,0	4,0	4,0	4,0	4,0	4,0	4,0	4,0			

RUN NO. 2

Baseline: 3,6. No. of base points: 199. Total no. of points: 414

Flowrate: 0,5509 litres/minute. Seconds per point: 0,0572

3,6	3,6	3,7	3,9	4,1	4,3	4,8	5,2	5,7	6,3	6,6	7,1	7,9	8,3
6,9	9,6	10,2	10,9	11,6	12,5	13,3	14,3	15,3	16,2	17,0	18,1	19,3	20,5
22,3	24,3	26,1	27,3	29,1	30,7	32,3	34,0	35,4	37,5	39,2	41,5	44,9	46,1
48,3	50,0	51,7	53,5	56,0	58,0	60,5	62,9	65,1	67,2	68,9	70,5	72,4	74,1
76,2	78,8	80,5	82,1	83,7	85,0	86,6	87,6	88,3	89,6	90,7	91,5	92,4	93,3
94,2	95,2	95,8	96,3	96,5	96,8	96,9	96,9	96,6	96,4	96,1	95,6	95,1	94,5
94,2	95,2	95,8	96,3	96,5	96,8	96,9	96,9	96,6	96,4	96,1	95,6	95,1	94,5
94,3	93,7	92,8	91,7	90,8	89,7	88,3	86,8	85,0	83,6	82,4	80,7	79,0	77,2
76,2	75,1	73,2	71,1	70,0	68,2	66,0	64,3	62,6	60,7	59,1	57,9	56,6	55,2
52,9	50,8	49,3	47,8	46,3	44,8	43,0	41,6	40,3	39,5	38,5	37,5	35,5	34,3
32,3	30,9	29,7	28,7	27,6	26,3	25,3	24,7	23,6	22,8	22,3	21,9	21,2	20,7
20,1	19,4	18,2	17,6	17,0	16,2	15,3	14,7	13,7	13,1	12,6	12,2	11,7	11,2
10,9	10,4	10,0	9,8	9,3	9,0	8,7	8,3	8,1	7,9	7,7	7,5	7,2	7,0
6,9	6,6	6,3	6,3	6,1	6,0	6,0	5,8	5,7	5,6	5,3	5,3	5,2	5,2
5,2	5,2	5,1	5,1	5,0	5,0	5,0	5,0	4,9	4,9	4,8	4,8	4,8	4,8
4,7	4,7	4,7	4,7	4,6	4,6	4,6	4,5	4,5	4,4	4,3	4,3	4,2	4,2
4,1	4,1	4,1	4,1	4,0	4,0								

RUN NO. 3

Baseline: 3,2. No. of base points: 139. Total no. of points: 322.

Flowrate: 0,7819 litres/minute. Seconds per point: 0,0572

3,2	3,2	3,3	3,5	3,7	4,2	4,7	5,5	6,3	7,0	7,7	8,2	9,3	10,2
11,3	12,3	13,5	15,0	16,1	17,3	19,0	20,8	23,5	25,7	28,0	30,0	32,4	34,3
37,0	39,3	42,8	45,5	48,4	51,1	53,3	56,2	59,0	62,1	65,2	67,8	70,0	72,1
74,6	76,7	79,5	81,3	83,0	84,6	85,9	86,9	87,9	88,5	89,2	89,6	90,1	90,2

[illegible]

RUN NO. 4

Baseline: 4,0. No. of base points: 145. Total no. of points: 326.

Flowrate: 0,7819 litres/minute. Seconds per point: 0,0572

[illegible]

RUN NO. 5

Baseline: 3,7. No. of base points: 221. Total no. of points: 460.

Flowrate: 1,1944 litres/minute. Seconds per point: 0,0572.

3,7	3,7	3,7	3,7	3,7	3,7	3,7	3,7	3,7	3,7	3,7	3,8	3,8	3,8
3,8	3,8	3,8	3,8	3,8	3,9	4,0	4,1	4,3	4,6	5,0	5,3	5,8	6,3
6,6	7,0	7,5	8,0	8,5	9,1	9,7	10,2	10,9	11,6	12,2	13,0	13,7	14,5
15,4	16,2	17,0	18,0	19,0	20,0	21,3	23,0	24,5	26,0	27,3	28,8	30,4	31,7
33,3	34,5	36,0	37,8	39,0	40,8	43,5	46,4	46,4	48,2	50,0	51,1	52,1	54,2
56,1	57,5	59,3	63,0	63,0	64,8	66,0	67,2	68,6	69,9	70,9	71,8	73,0	74,0
75,2	76,1	77,3	78,4	79,3	79,8	80,3	80,8	81,3	81,9	82,1	82,1	82,4	82,6
82,8	82,5	82,9	82,3	82,1	82,0	81,7	81,0	80,5	80,0	79,2	78,8	78,0	77,3
76,7	76,1	75,3	74,3	73,3	72,6	71,8	70,5	69,2	67,8	66,6	65,2	64,0	62,6
61,4	60,1	58,9	58,0	57,4	56,6	55,2	52,9	51,9	50,5	49,0	47,8	46,7	45,9
44,1	42,9	41,8	40,4	39,5	39,9	39,3	38,2	37,4	36,0	34,7	33,0	32,3	30,9
28,7	28,3	27,4	26,4	25,7	24,9	24,2	23,6	22,7	22,2	21,8	21,3	20,9	20,2
20,2	19,7	18,8	17,9	17,5	17,0	16,3	15,5	15,1	14,7	14,0	13,7	13,3	12,9
12,2	12,0	11,7	11,4	11,1	10,8	10,2	10,1	9,9	9,7	9,2	9,0	8,8	8,5
8,2	8,0	7,8	7,6	7,4	7,2	7,0	6,9	6,8	6,5	6,4	6,3	6,2	6,1
6,0	5,9	5,8	5,8	5,6	5,4	5,3	5,3	5,3	5,3	5,2	5,2	5,2	5,2
5,1	5,1	5,1	5,1	5,1	5,0	5,0	5,0	5,0	4,9	4,9	4,9	4,9	4,9

4,2 4,2 4,2 4,2 4,2 4,2 4,1 4,1 4,1 4,1 4,1 4,1 4,1 4,1
4,1 4,1 4,1 4,1

RUN NO. 8

Baseline: 4,1. No. of base points: 141. Total no. of points: 396.

Flowrate: 1,5613 litres/minute. Seconds per point: 0,0572

4,1 4,1 4,1 4,1 4,2 4,3 4,6 5,3 6,1 6,6 7,2 8,0 8,6 9,5
10,2 11,3 12,4 13,3 14,5 15,8 17,1 18,8 20,1 22,1 24,5 26,6 28,8 30,8
33,0 35,0 37,4 40,1 43,4 46,1 48,6 51,0 53,0 56,1 58,5 61,9 65,3 68,0
69,9 72,6 74,9 77,5 79,7 82,5 84,3 86,1 87,7 89,0 91,0 91,8 93,3 94,5
96,0 97,2 98,0 98,6 98,7 99,0 99,2 99,2 99,0 98,8 98,3 97,3 96,5 95,6
94,7 93,7 92,6 91,1 90,0 88,6 86,3 84,2 82,5 80,7 78,7 76,9 75,2 73,2
71,0 67,1 66,5 64,8 62,5 60,5 58,7 57,2 56,0 53,1 51,0 49,2 47,4 45,6
43,6 41,9 40,4 39,5 38,3 36,6 34,9 33,3 31,1 29,6 28,3 27,6 26,2 25,0
24,2 23,0 22,3 21,9 21,1 20,6 19,9 19,0 18,0 17,0 16,1 15,3 14,5 13,9
13,2 12,8 12,2 11,9 11,3 10,9 10,4 10,0 9,7 9,3 9,0 8,7 8,2 8,0
7,8 7,4 7,2 7,0 6,8 6,6 6,5 6,3 6,2 6,0 6,0 5,8 5,7 5,5
5,4 5,3 5,3 5,2 5,2 5,2 5,1 5,1 5,1 5,1 5,0 5,0 5,0 5,0
5,0 5,0 5,0 5,0 5,0 4,9 4,9 4,9 4,9 4,9 4,8 4,8 4,8 4,8
4,8 4,7 4,7 4,7 4,7 4,7 4,6 4,6 4,6 4,6 4,5 4,5 4,5 4,5
4,5 4,4 4,4 4,4 4,4 4,4 4,4 4,3 4,3 4,3 4,3 4,3 4,3 4,3
4,3 4,3 4,3 4,3 4,2 4,2

RUN NO. 9

Baseline: 4,2. No. of base points: 146. Total no. of points: 336.

Flowrate: 1,9257 litres/minute. Seconds per point: 0,0572

4,2 4,2 4,3 4,6 4,7 5,3 6,0 6,6 7,2 7,9 8,7 9,6 10,2 11,5
12,8 13,0 15,3 16,8 17,7 20,2 22,5 25,2 27,7 30,5 32,9 35,2 37,5 40,5
44,3 47,2 50,3 52,8 55,8 58,8 62,0 65,0 67,5 69,9 71,9 74,5 76,6 79,1
80,6 82,4 83,9 94,7 85,4 86,3 86,7 87,0 87,1 87,1 87,0 86,4 85,8 84,6
83,5 82,3 81,1 79,3 77,5 76,2 74,5 72,4 7,10 67,7 66,0 63,3 61,0 58,7
57,2 55,5 52,4 50,2 48,0 46,2 44,0 41,8 40,3 39,0 37,8 35,3 33,0 31,0
29,2 28,0 26,5 25,2 23,7 22,6 21,9 21,0 20,2 19,5 18,2 17,3 16,0 15,1
14,5 13,6 13,0 12,3 11,4 11,1 10,7 10,1 9,6 9,2 8,9 8,3 8,0 7,7
7,4 7,1 6,8 6,6 6,3 6,1 6,0 5,9 5,9 5,7 5,5 5,3 5,2 5,2
5,2 5,1 5,1 5,0 5,0 5,0 5,0 5,0 5,0 4,9 4,9 4,9 4,9 4,9
4,8 4,3 4,8 4,8 4,8 4,8 4,8 4,7 4,7 4,7 4,7 4,7 4,6 4,6
4,6 4,6 4,6 4,5 4,5 4,5 4,5 4,4 4,4 4,4 4,4 4,4 4,3 4,3
4,3 4,3 4,3 4,3 4,3 4,3 4,3 4,3 4,3 4,3 4,3 4,3 4,3 4,3
4,3 4,3 4,3 4,2 4,2 4,2 4,2 4,2 4,2

RUN NO. 10

Baseline: 4,0. No. of base points: 146. Total no. of points: 324.

Flowrate: 1,9257 litres/minute. Seconds per point: 0,0572

4,0 4,0 4,1 4,2 4,5 5,1 5,7 6,1 6,8 7,3 8,1 8,9 10,0 10,7
12,1 13,2 14,2 15,9 17,3 18,9 21,0 23,6 26,2 28,4 30,8 33,3 35,6 38,3

42,0	45,1	48,0	51,0	52,8	56,0	58,7	62,2	65,3	67,9	69,9	72,0	73,8	75,9
78,3	79,9	81,2	82,6	83,3	84,1	84,7	85,1	85,2	85,3	85,2	84,8	84,3	83,4
82,2	81,1	80,0	78,3	76,8	75,7	74,8	72,0	70,5	67,6	65,3	63,2	61,0	58,5
57,2	55,5	52,2	50,4	48,2	46,0	43,8	41,8	40,1	38,8	37,4	35,1	33,0	31,0
29,4	28,2	26,4	25,3	23,9	22,7	22,0	21,3	20,5	19,6	18,5	17,1	16,2	15,3
14,4	13,6	13,0	12,3	11,5	11,1	10,6	10,2	9,7	9,3	8,9	8,5	8,0	7,6
7,4	7,1	6,9	6,6	6,4	6,2	6,0	5,9	5,7	5,5	5,4	5,3	5,2	5,2
5,1	5,0	5,0	5,0	5,0	5,0	4,9	4,9	4,8	4,8	4,8	4,7	4,7	4,7
4,7	4,7	4,7	4,6	4,6	4,6	4,6	4,6	4,5	4,5	4,5	4,5	4,5	4,5
4,5	4,5	4,4	4,4	4,4	4,4	4,3	4,3	4,3	4,3	4,3	4,3	4,3	4,3
4,3	4,3	4,3	4,2	4,2	4,2	4,2	4,2	4,2	4,1	4,1			

RUN NO. 11

Baseline: 4,1. No. of base points: 251. Total no. of points: 536.

Flowrate: 2,2851 litres/minute. Seconds per point: 0,0572.

4,1	4,1	4,2	4,3	4,5	4,7	5,2	5,5	5,9	6,3	6,7	7,0	7,6	8,0
8,3	9,0	9,6	10,2	10,8	11,8	12,5	13,2	14,1	15,0	15,9	17,0	18,2	19,0
19,9	21,3	23,1	25,0	26,2	27,9	29,6	31,1	32,5	33,9	35,4	37,5	38,8	40,6
43,5	45,2	46,9	49,0	50,5	52,1	53,4	55,1	57,0	58,9	61,5	63,3	64,8	66,6
68,1	69,3	70,2	71,5	73,2	75,0	76,0	77,7	79,0	80,0	81,5	82,2	83,1	84,0
84,6	85,1	85,6	86,1	86,5	86,7	86,9	87,0	87,1	87,1	87,2	87,2	87,2	87,0
86,8	86,3	85,3	84,5	84,0	83,5	82,5	81,8	81,2	80,2	79,3	77,9	76,9	76,2
75,5	74,2	73,0	72,0	71,2	69,7	68,0	66,4	64,8	63,3	62,2	60,9	59,2	58,0
57,3	56,2	54,9	53,0	51,6	50,1	48,8	47,6	46,3	45,0	43,2	42,1	41,7	40,1
39,3	38,6	37,5	36,4	35,3	33,9	32,4	31,3	30,2	29,5	28,3	27,6	26,7	26,0
25,2	24,7	23,9	23,0	22,4	22,1	21,6	20,8	20,4	20,1	19,7	18,9	18,9	17,4
16,8	16,1	15,4	15,1	14,7	14,2	13,5	13,0	12,5	12,3	11,9	11,6	11,4	11,1
10,7	10,3	10,0	9,9	9,7	9,3	9,0	8,9	8,8	8,5	8,2	8,0	7,8	7,5
7,3	7,2	7,1	6,9	6,6	6,5	6,3	6,2	6,1	6,0	6,0	5,9	5,9	5,8
5,7	5,6	5,4	5,3	5,3	5,2	5,2	5,2	5,1	5,1	5,1	5,1	5,1	5,1
5,1	5,0	5,0	5,0	5,0	5,0	5,0	5,0	4,9	4,9	4,9	4,9	4,9	4,9
4,8	4,8	4,8	4,8	4,8	4,8	4,7	4,7	4,7	4,7	4,7	4,6	4,6	4,6
4,6	4,6	4,6	4,6	4,6	4,6	4,6	4,6	4,6	4,5	4,5	4,5	4,5	4,5
4,5	4,5	4,5	4,5	4,5	4,5	4,5	4,5	4,5	4,5	4,4	4,4	4,4	4,4
4,4	4,4	4,4	4,4	4,4	4,4	4,4	4,4	4,3	4,3	4,3	4,3	4,3	4,3
4,3	4,3	4,3	4,3	4,3	4,3	4,3							

RUN NO. 12

Baseline: 4,0. No. of base points: 251. Total no. of points: 546.

Flowrate: 2,2851 litres/minute. Seconds per point: 0,0572.

4,0	4,1	4,2	4,3	4,4	4,6	5,0	5,7	6,0	6,3	6,7	7,0	7,4	8,0
0,5	9,0	9,5	10,3	10,8	11,5	12,5	13,3	14,0	14,9	15,6	16,3	17,5	18,6
19,6	20,7	22,0	24,0	25,7	27,1	28,6	30,0	31,5	33,0	34,4	36,1	37,9	39,1
41,6	43,9	45,6	47,2	48,7	50,3	51,6	52,8	54,9	56,2	58,0	60,0	62,0	63,5
65,2	66,8	68,0	69,0	70,1	71,2	72,3	73,5	75,0	75,9	76,7	78,0	79,1	79,7
80,2	81,0	81,5	81,8	82,2	82,3	82,8	82,9	83,0	82,9	82,8	82,7	82,5	82,3
82,1	81,8	81,0	80,4	79,8	79,2	78,5	77,5	76,9	76,5	75,5	74,6	73,6	72,9
72,0	71,0	69,3	67,8	66,5	65,2	64,0	62,9	61,5	60,0	58,7	57,9	57,0	56,0
54,8	52,5	51,4	50,0	48,8	47,8	46,8	45,4	44,1	42,6	41,7	40,5	39,9	39,1
38,1	37,2	36,0	34,9	33,3	32,2	30,9	29,8	29,1	28,2	27,5	26,4	25,9	25,0

24,0 23,2 22,7 22,3 21,7 21,5 20,7 20,6 19,9 19,3 18,6 18,1 17,0 16,3
 15,9 15,3 14,9 14,3 13,8 13,4 13,0 12,7 12,3 12,0 11,7 11,3 11,2 10,6
 10,3 10,0 9,8 9,7 9,4 9,1 8,9 8,7 8,3 8,1 7,9 7,7 7,5 7,3
 7,1 7,0 6,9 6,8 6,7 6,6 6,4 6,3 6,2 6,1 6,0 5,8 5,7 5,6
 5,6 5,5 5,4 5,3 5,3 5,2 5,2 5,1 5,1 5,1 5,0 5,0 5,0 5,0
 5,0 5,0 5,0 5,0 5,0 5,0 4,9 4,9 4,9 4,8 4,8 4,8 4,8 4,8
 4,8 4,8 4,8 4,8 4,8 4,7 4,7 4,7 4,7 4,7 4,7 4,7 4,7 4,7
 4,7 4,7 4,7 4,6 4,6 4,6 4,6 4,6 4,6 4,6 4,6 4,6 4,5
 4,5 4,5 4,5 4,5 4,5 4,5 4,5 4,4 4,4 4,4 4,4 4,4 4,4 4,4
 4,4 4,4 4,4 4,4 4,4 4,3 4,3 4,3 4,3 4,3 4,3 4,3 4,3 4,3
 4,3 4,3 4,3 4,3 4,3 4,3 4,2 4,2 4,2 4,2 4,2 4,2 4,2 4,2
 4,2 4,2

RUN NO. 13

Baseline: 4,1. No. of base points: 216. Total no. of points: 456.

Flowrate: 2,6450 litre/minute. Seconds per point: 0,0572.

4,1 4,1 4,1 4,2 4,2 4,3 4,5 4,9 5,5 5,9 6,3 6,7 7,2 7,8
 8,5 9,0 9,7 10,8 11,6 12,5 13,3 14,2 15,6 16,6 18,0 19,3 20,2 22,0
 24,8 26,5 28,0 29,7 31,4 33,2 34,3 36,8 38,5 41,0 44,0 46,1 48,3 50,0
 52,0 53,8 56,2 58,3 61,1 63,6 65,5 67,1 69,3 70,8 72,3 74,0 75,9 78,0
 79,5 80,5 82,1 83,3 84,0 85,2 86,0 86,8 87,2 87,7 88,1 88,3 88,6 88,7
 88,8 88,9 88,9 88,9 88,9 88,7 88,2 87,7 87,2 85,8 84,5 83,6 82,8 81,5
 80,1 79,0 78,1 76,5 75,5 74,3 73,0 71,5 70,3 68,2 66,6 65,1 64,0 61,8
 60,2 58,5 57,5 56,5 55,2 53,1 51,8 50,2 48,3 47,0 46,5 44,1 42,6 41,7
 40,3 39,2 38,6 37,5 36,0 34,5 33,7 31,9 30,3 29,5 28,4 27,4 26,5 25,7
 24,8 24,0 23,3 22,6 22,0 21,5 21,0 20,5 19,8 19,4 18,5 17,9 16,9 16,0
 15,4 15,0 14,3 13,7 13,3 12,9 12,6 12,1 11,7 11,3 10,9 10,7 10,2 9,9
 9,7 9,5 9,2 8,9 8,6 8,3 8,1 7,9 7,7 7,4 7,3 7,2 7,0 6,9
 6,7 6,5 6,4 6,3 6,2 6,1 6,0 5,9 5,8 5,7 5,6 5,4 5,3 5,3
 5,3 5,2 5,2 5,2 5,1 5,1 5,1 5,1 5,1 5,0 5,0 5,0 5,0 5,0
 5,0 5,9 4,9 4,9 4,9 4,9 4,8 4,8 4,8 4,8 4,8 4,8 4,8 4,8
 4,8 4,8 4,8 4,8 4,8 4,8 4,8 4,7 4,7 4,7 4,7 4,7 4,7
 4,7 4,6 4,6 4,6 4,6 4,6 4,6 4,6 4,6 4,6 4,5 4,5 4,5
 4,5 4,5 4,5

RUN NO. 14

Baseline: 4,2. No. of base points: 216. Total no. of points: 476.

Flowrate: 2,6450 litre/minute. Seconds per point: 0,0572.

4,2 4,2 4,2 4,3 4,4 4,6 4,9 5,3 6,0 6,3 7,0 7,2 7,9 8,4
 9,0 9,7 10,4 11,3 11,9 13,1 14,0 15,0 16,3 17,1 18,4 19,6 21,2 22,3
 25,5 26,8 28,5 30,3 32,0 34,0 36,0 37,6 39,2 42,7 44,9 46,9 49,3 51,1
 52,5 55,0 57,1 59,2 62,3 64,3 66,2 68,0 69,4 70,8 72,3 74,0 75,9 77,5
 79,6 80,8 81,9 82,8 84,1 84,6 85,2 85,8 86,4 86,9 87,0 87,1 87,3 87,4
 87,5 87,4 87,3 87,2 86,9 86,3 85,2 84,2 83,7 82,8 81,5 80,5 79,5 78,1
 76,9 76,0 74,7 73,6 72,0 71,1 69,4 67,1 65,7 64,2 62,9 61,0 59,0 57,9
 57,0 55,6 53,5 51,5 50,3 48,3 47,2 45,8 44,3 42,5 41,4 40,1 39,3 38,4
 37,0 36,2 34,5 32,5 31,2 30,2 29,1 28,2 27,3 26,3 25,3 24,5 23,4 22,6
 22,2 21,6 21,1 20,5 19,8 19,5 18,5 17,6 17,1 16,3 15,4 15,0 14,2 13,7
 13,3 12,6 12,5 11,9 11,4 11,2 11,0 10,6 10,4 9,9 9,7 9,4 9,2 8,9
 8,5 8,3 8,1 7,9 7,6 7,4 7,3 7,1 7,0 6,9 6,8 6,6 6,4 6,3

6,1	6,0	5,9	5,8	5,7	5,6	5,6	5,5	5,4	5,3	5,2	5,2	5,2	5,2
5,1	5,1	5,1	5,1	5,1	5,0	5,0	5,0	5,0	5,0	5,0	5,0	5,0	5,0
5,0	5,0	5,0	5,0	4,9	4,9	4,9	4,9	4,9	4,8	4,8	4,8	4,8	4,8
4,8	4,8	4,8	4,8	4,7	4,7	4,7	4,7	4,7	4,7	4,7	4,7	4,6	4,6
4,6	4,6	4,6	4,6	4,6	4,6	4,6	4,6	4,6	4,6	4,6	4,6	4,6	4,6
4,5	4,5	4,5	4,5	4,5	4,5	4,5	4,5	4,5	4,5	4,5	4,4	4,4	4,4
4,4	4,4	4,4	4,4	4,3	4,3	4,3	4,3	4,3	4,3	4,3			

RUN NO. 15

Baseline: 4,2. No. of base points: 196. Total no. of points: 432.

Flowrate: 2,9940 litre/minute. Seconds per point: 0,0572.

4,3	4,7	5,2	5,9	6,2	6,8	7,3	8,0	8,9	9,3	10,4	11,5	12,2	13,3
14,6	16,0	17,0	18,5	20,0	22,0	24,2	26,5	28,3	31,1	33,0	34,8	37,4	39,1
43,0	44,3	48,1	50,8	52,6	55,0	58,2	61,1	63,6	65,3	69,0	70,7	72,7	75,0
77,9	80,2	82,3	83,8	85,1	86,8	88,1	88,0	90,0	91,1	91,7	92,4	93,0	93,3
93,6	93,8	93,9	94,0	94,0	93,9	93,8	93,3	92,5	91,6	90,0	89,6	88,0	86,0
84,2	83,1	81,5	79,5	77,6	76,0	74,5	72,9	70,6	68,6	66,2	64,5	62,5	60,4
58,5	57,0	56,0	54,1	52,0	49,8	48,3	46,6	44,7	42,7	41,1	40,0	39,7	37,7
36,5	34,6	32,5	30,8	29,0	27,3	26,2	25,3	24,4	23,3	22,3	21,8	21,4	
20,7	20,2	19,2	18,3	17,5	17,0	15,8	15,2	14,3	13,6	13,1	12,4	12,1	11,6
11,4	10,9	10,3	10,1	9,9	9,5	9,2	8,9	8,5	8,3	8,1	7,9	7,6	7,3
7,1	7,0	6,9	6,7	6,5	6,3	6,2	6,1	6,0	5,9	5,8	5,7	5,6	5,4
5,3	5,3	5,2	5,2	5,2	5,2	5,1	5,1	5,1	5,1	5,0	5,0	5,0	5,0
5,0	5,0	5,0	5,0	5,0	5,0	5,0	5,0	5,0	4,9	4,9	4,9	4,9	4,9
4,9	4,9	4,9	4,9	4,9	4,9	4,9	4,9	4,8	4,8	4,8	4,8	4,8	4,8
4,8	4,8	4,8	4,8	4,8	4,7	4,7	4,7	4,7	4,7	4,7	4,7	4,7	4,7
4,7	4,6	4,6	4,6	4,6	4,6	4,6	4,6	4,6	4,6	4,6	4,6	4,6	4,6
4,6	4,6	4,5	4,5	4,5	4,5	4,5	4,5	4,5	4,5	4,5	4,5	4,5	4,5

RUN NO. 16

Baseline: 4,1. No. of base points: 196. Total no. of points: 350.

Flowrate: 2,9924 litres/minute. Seconds per point: 0,0572.

4,2	4,3	4,6	5,3	5,8	6,3	6,7	7,3	8,0	8,6	9,5	10,5	11,3	12,3
13,4	14,7	16,1	17,0	18,7	20,0	22,0	24,5	26,4	28,5	30,2	32,4	34,2	37,0
38,5	41,2	44,7	47,2	49,4	51,6	53,6	56,1	58,3	61,2	64,0	66,0	67,6	70,0
71,4	73,0	75,0	76,8	79,0	80,0	81,4	82,8	83,6	84,3	85,1	85,6	86,0	86,2
86,4	86,6	86,5	86,5	86,3	86,1	85,0	84,0	82,9	82,3	81,0	79,9	78,3	77,1
76,2	74,8	73,1	71,7	70,1	68,4	66,2	64,1	62,4	60,8	58,5	57,6	56,1	54,4
52,3	51,7	48,3	46,7	45,2	43,3	41,7	40,4	39,1	38,5	36,9	35,1	33,8	31,9
30,0	29,0	28,0	27,0	25,5	24,6	23,6	22,6	22,0	21,4	20,7	20,2	19,4	18,2
17,3	16,7	15,6	15,0	14,7	13,6	13,1	12,6	12,0	11,7	11,2	10,6	10,4	10,0
9,7	9,4	8,9	8,8	8,4	8,2	7,9	7,5	7,3	7,1	6,9	6,7	6,4	6,3
6,2	6,1	6,0	5,8	5,7	5,6	5,4	5,3	5,3	5,3	5,2	5,2	5,2	5,2
5,2													

NOTE: The excessive baseline drift on the recorder during Run No. 16 meant that this run could not be used to obtain a residence time distribution.

RUN NO. 17

Baseline: 4.2. No. of base points: 191. Total no. of points: 412.

Flowrate: 2,9924 litres/minute. Seconds per point: 0,0572

4,2 4,2 4,2 4,3 4,4 4,6 5,0 5,6 6,3 6,8 7,3 8,0 8,8 9,5
 10,2 11,0 12,2 13,3 14,5 15,7 16,8 18,5 19,8 21,1 23,2 25,9 27,8 29,3
 31,8 34,0 35,6 38,1 40,3 44,1 46,7 49,2 51,1 52,9 55,8 58,1 60,9 63,5
 66,0 68,0 70,0 71,2 73,0 75,2 77,5 79,2 80,7 81,7 83,2 84,5 85,1 86,0
 86,6 88,8 87,0 87,2 87,3 87,4 87,4 87,3 87,1 86,6 85,6 84,7 83,5 82,6
 81,5 80,3 78,5 77,3 76,3 74,9 73,5 71,6 70,4 68,1 66,0 64,1 62,3 60,6
 58,4 57,5 56,1 54,0 51,9 50,2 48,0 46,4 44,9 43,1 41,6 40,3 39,1 37,9
 37,0 34,7 32,8 31,5 29,8 28,8 27,8 26,5 25,5 24,5 23,6 22,3 22,0 21,2
 21,7 20,0 19,5 18,5 17,2 16,8 15,5 15,0 14,6 13,6 13,0 12,5 12,1 11,4
 11,0 10,7 10,3 10,0 9,8 9,4 9,0 8,7 8,5 8,1 7,9 7,7 7,4 7,1
 7,0 6,8 6,7 6,5 6,3 6,2 6,0 5,9 5,8 5,5 5,5 5,4 5,3 5,2
 5,2 5,2 5,1 5,1 5,1 5,0 5,0 5,0 5,0 5,0 5,0 5,0 5,0 5,0
 4,9 4,9 4,9 4,9 4,9 4,9 4,9 4,9 4,9 4,9 4,9 4,9 4,9 4,9
 4,8 4,8 4,8 4,8 4,8 4,8 4,8 4,8 4,8 4,8 4,8 4,7 4,7 4,7
 4,7 4,7 4,6 4,6 4,6 4,6 4,6 4,5 4,5 4,5 4,5 4,5 4,5 4,5
 4,5 4,4 4,4 4,4 4,4 4,4 4,4 4,3 4,3 4,3 4,3 4,3 4,3 4,3
 4,3

RUN NO. 18

Baseline: 4,1. No. of base points: 171. Total no. of points: 371.

Flowrate: 3,3342 litres/minute. Seconds per point: 0,0572

4,1 4,1 4,1 4,1 4,1 4,3 4,6 5,1 5,8 6,2 6,8 7,5 8,2 9,0
 10,0 11,0 12,2 13,4 14,5 15,9 17,5 19,1 20,9 23,0 25,1 28,2 30,4 33,0
 35,0 37,4 39,3 43,1 46,0 48,3 50,3 53,0 55,8 58,0 60,7 63,8 66,2 68,0
 69,8 71,2 73,5 75,2 77,0 78,8 80,1 81,3 82,1 82,8 83,1 83,7 83,6 84,0
 83,9 83,7 83,2 82,6 82,0 81,9 80,0 79,0 77,5 76,3 75,0 73,6 72,0 70,6
 68,0 65,9 63,3 62,0 60,0 58,1 57,1 55,2 52,2 50,5 48,3 47,0 45,0 43,0
 41,3 39,9 38,6 37,3 35,2 34,0 32,0 30,0 28,5 27,5 26,1 25,0 23,9 22,8
 22,1 21,3 20,6 20,1 19,1 18,0 17,0 16,0 15,2 14,4 13,3 13,0 12,5 12,0
 11,2 10,9 10,6 10,1 9,8 9,4 9,2 8,9 8,5 8,2 7,8 7,4 7,3 7,0
 6,9 6,6 6,3 6,2 6,0 5,9 5,8 5,6 5,4 5,3 5,2 5,1 5,1 5,1
 5,0 5,0 5,0 5,0 5,0 5,0 5,0 5,0 4,9 4,9 4,9 4,9 4,9 4,9
 4,9 4,9 4,8 4,8 4,8 4,8 4,8 4,7 4,7 4,7 4,6 4,6 4,6 4,6
 4,6 4,6 4,6 4,6 4,6 4,5 4,5 4,5 4,5 4,4 4,4 4,4 4,4 4,4
 4,4 4,3 4,3 4,3 4,3 4,3 4,3 4,3 4,3 4,3 4,3 4,3 4,2 4,2
 4,2 4,2 4,2 4,2 4,2 4,2

RUN NO. 19

Baseline: 4,0. No. of base points: 176. Total no. of points: 361.

Flowrate: 3,3342 litres/minute. Seconds per point: 0,0572.

4,0 4,1 4,3 4,6 5,3 5,9 6,4 7,0 7,7 8,4 9,5 10,9 10,7 12,0
 13,0 14,5 15,7 16,7 18,2 19,6 21,0 23,5 26,0 27,5 30,0 31,2 33,0 34,7
 36,7 38,2 40,2 43,2 45,0 47,0 49,0 50,2 51,3 52,8 54,3 55,2 56,4 57,6
 58,4 59,3 60,6 61,4 61,9 62,3 62,6 62,8 62,9 62,7 62,6 62,3 61,9 61,0
 60,3 59,5 58,8 58,2 57,1 56,2 55,0 53,8 52,8 51,4 49,0 47,8 46,3 45,0

43,2	41,8	40,5	39,7	38,4	37,4	35,2	33,8	32,5	30,6	29,5	28,4	27,0	26,0
25,0	24,0	23,0	22,2	21,7	20,8	20,3	19,5	19,0	17,8	16,8	15,7	14,7	14,2
13,4	13,0	12,3	11,9	11,3	11,0	10,6	10,2	9,8	9,5	9,2	8,9	8,5	8,1
7,8	7,5	7,3	7,0	6,9	6,7	6,4	6,2	6,1	6,0	5,9	5,7	5,5	5,4
5,3	5,2	5,1	5,1	5,1	5,0	5,0	5,0	5,0	5,0	5,0	5,0	4,9	4,9
4,9	4,9	4,9	4,9	4,8	4,8	4,8	4,8	4,8	4,8	4,8	4,8	4,8	4,7
4,7	4,7	4,7	4,7	4,7	4,7	4,6	4,6	4,6	4,5	4,5	4,5	4,5	4,4
4,4	4,4	4,3	4,3	4,3	4,3	4,2	4,2	4,2	4,2	4,2	4,2	4,2	4,2
4,2	4,2	4,2	4,2	4,2									

RUN NO. 20

Baseline: 4,1. No. of base points: 176. Total no. of points: 341.

Flowrate: 3,3342 litres/minute. Seconds per point: 0,0572.

4,2	4,5	5,0	5,8	6,2	6,8	7,6	8,3	9,5	10,2	11,5	13,0	14,1	15,5
16,6	18,7	20,0	22,0	25,0	27,0	29,3	31,8	34,1	36,1	39,0	41,7	45,0	48,0
50,8	52,6	55,2	58,1	61,1	64,3	67,0	69,0	71,0	73,4	75,2	78,0	79,9	81,2
82,9	84,1	85,0	85,6	86,5	86,9	87,3	87,6	87,8	87,9	87,9	87,8	87,3	86,7
85,3	84,0	82,9	81,9	80,0	78,5	76,9	75,5	73,8	72,3	71,1	68,0	66,2	64,0
62,0	59,7	58,0	56,6	54,5	51,9	50,1	47,8	46,2	44,2	42,5	40,8	39,5	38,1
36,9	34,8	32,7	31,0	29,5	28,2	27,1	25,8	24,6	23,7	22,5	21,8	21,4	20,7
19,9	18,6	17,9	16,5	15,6	15,1	14,2	13,6	12,7	12,5	12,1	11,5	10,9	10,5
99,8	9,6	9,3	9,0	8,7	8,3	8,0	9,7	7,3	2	6,9	6,7	6,6	6,4
6,3	6,0	5,9	5,8	5,7	5,5	5,3	5,3	5,2	5,2	5,1	5,1	5,0	5,0
5,0	4,9	4,9	4,9	4,9	4,9	4,9	4,9	4,9	4,9	4,9	4,9	4,9	4,9
4,8	4,8	4,8	4,8	4,8	4,8	4,7	4,7	4,7	4,6	4,6	4,5	4,5	

Note: Runs 21-26 were discarded because it was discovered that the recorder had been overdamped and the resulting $B(t)$ curves had been affected by the recorder dynamics.

RUN NO. 27

Baseline: 0,71. No. of base points: 181. Total no. of points: 420.

Flowrate: 0,4878 litres/minute. Seconds per point: 0,6000.

0,71	0,71	0,71	0,71	0,71	0,71	0,72	0,72	0,72	0,74	0,75	0,80	0,85	0,86
0,87	0,88	0,89	0,90	0,90	0,91	0,91	0,92	0,93	0,93	0,93	0,94	0,95	0,95
0,96	0,97	0,97	0,98	0,99	1,00	1,01	1,02	1,03	1,04	1,05	1,11	1,13	1,15
1,19	1,22	1,31	1,35	1,38	1,42	1,48	1,55	1,60	1,65	1,69	1,77	1,89	1,92
1,97	2,05	2,15	2,20	2,34	2,38	2,49	2,61	2,65	2,70	2,82	2,95	3,07	3,19
3,34	3,42	3,58	3,68	3,77	3,90	4,10	4,30	4,35	4,50	4,65	4,81	5,00	5,10
5,15	5,25	5,29	5,40	5,50	5,60	5,70	5,85	6,01	6,17	6,23	5,34	6,50	6,59
6,60	6,69	6,72	6,81	6,90	7,13	7,25	7,35	7,32	7,35	7,46	7,52	7,53	7,57
7,60	7,61	7,62	7,62	7,62	7,61	7,61	7,61	7,61	7,60	7,06	7,59	7,57	7,59
7,47	7,37	7,30	7,26	7,21	7,13	7,00	6,97	6,95	6,86	6,75	6,62	6,51	6,35
6,29	6,20	6,09	6,01	5,90	5,78	5,62	5,48	5,36	5,20	5,10	5,00	4,91	4,87
4,77	4,64	4,50	4,39	4,30	4,12	4,01	3,94	3,89	3,80	3,71	3,59	3,40	3,30
3,21	3,17	3,07	3,00	2,94	2,90	2,77	2,70	2,67	2,60	2,51	2,40	2,35	2,31
2,25	2,21	2,20	2,12	2,00	1,98	1,93	1,87	1,81	1,78	1,73	1,71	1,63	1,60
1,49	1,43	1,41	1,40	1,39	1,38	1,37	1,36	1,35	1,33	1,32	1,30	1,25	1,15

1,13 1,09 1,05 1,04 1,03 1,02 1,01 1,00 1,00 0,99 0,97 0,93 0,92 0,91
0,91 0,90 0,90 0,89 0,88 0,87 0,86 0,85 0,84 0,83 0,82 0,81 0,80 0,79
0,76 0,74

RUN NO. 28

Baseline: 0,40. No. of base points: 190. Total no. of points: 407.
Flowrate: 0,4878 litres/minute. Seconds per point: 0,6000.

0,40 0,41 0,43 0,44 0,45 0,47 0,48 0,50 0,52 0,55 0,56 0,58 0,60 0,62
0,63 0,67 0,69 0,73 0,75 0,77 0,80 0,85 0,87 0,90 0,93 0,95 1,00 1,10
1,13 1,17 1,33 1,40 1,52 1,58 1,62 1,69 1,76 1,88 1,97 2,06 2,16 2,26
2,36 2,51 2,65 2,72 2,89 2,94 3,16 3,23 3,40 3,56 3,71 3,80 3,97 4,12
4,30 4,40 4,53 4,72 4,84 4,98 5,15 5,26 5,40 5,55 5,75 5,86 5,97 6,17
6,25 6,34 6,50 6,63 6,71 6,83 6,95 7,07 7,15 7,27 7,37 7,38 7,50 7,58
7,64 7,71 7,75 7,78 7,81 7,85 7,89 7,90 7,90 7,90 7,90 7,90 7,90 7,89
7,88 7,87 7,85 7,85 7,82 7,81 7,80 7,80 7,77 7,70 7,65 7,57 7,48 7,35
7,29 7,19 7,07 6,94 6,82 6,70 6,63 6,47 6,32 6,15 6,01 5,95 5,87 5,72
5,68 5,49 5,41 4,28 5,19 5,03 4,90 4,81 4,67 4,50 4,43 4,32 4,20 4,08
4,00 3,87 3,76 3,65 3,53 3,33 3,28 3,20 3,10 3,03 3,00 2,93 2,85 2,77
2,67 2,55 2,51 2,41 2,35 2,27 2,20 2,10 2,08 2,00 1,97 1,95 1,92 1,87
1,80 1,78 1,73 1,67 1,60 1,50 1,46 1,45 1,40 1,35 1,33 1,32 1,30 1,29
1,26 1,25 1,23 1,20 1,18 1,15 1,11 1,10 1,09 1,07 1,05 1,03 1,01 1,00
0,99 0,97 0,93 0,90 0,85 0,85 0,83 0,81 0,80 0,80 0,80 0,80 0,80 0,79
0,79 0,78 0,77 0,76 0,76 0,75 0,75 0,73 0,72 0,71 0,71

RUN NO. 29

Baseline: 1,10. No. of base points: 73. Total no. of points: 169.
Flowrate: 1,2018 litres/minute. Seconds per point: 0,6000.

1,10 1,10 1,10 1,10 1,10 1,10 1,10 1,10 1,10 1,11 1,12 1,14 1,19 1,21
1,24 1,26 1,30 1,32 1,40 1,47 1,50 1,70 1,77 1,96 2,09 2,29 2,53 2,82
3,08 3,41 3,84 4,11 4,62 4,97 5,42 5,85 6,17 6,58 6,96 7,40 7,72 7,90
8,15 8,36 8,53 8,68 8,71 8,79 8,77 8,75 8,68 8,53 8,35 8,09 7,90 7,53
7,17 6,75 6,34 6,12 5,80 5,50 5,23 4,79 4,55 4,23 3,97 3,85 3,53 3,36
3,13 2,80 2,61 2,50 2,44 2,33 2,16 2,09 2,00 1,93 1,87 1,76 1,70 1,67
1,59 1,56 1,50 1,49 1,48 1,47 1,45 1,38 1,31 1,30 1,25 1,21 1,20

RUN NO. 30

Baseline: 1,60. No. of base points: 73. Total no. of points: 181.
Flowrate: 1,2018 litres/minute. Seconds per point: 0,6000

1,60 1,60 1,60 1,60 1,60 1,60 1,60 1,60 1,60 1,60 1,61 1,62 1,64
1,66 1,69 1,72 1,82 1,83 1,90 1,99 2,07 2,23 2,40 2,57 2,77 2,98 3,29
3,77 4,02 4,37 4,70 5,08 5,53 6,00 6,41 6,67 7,12 7,48 7,92 8,21 8,50
8,74 8,72 9,10 9,18 9,29 9,42 9,44 9,42 9,40 9,29 9,13 8,99 8,73 8,43
8,05 7,77 7,45 7,00 6,62 6,30 6,00 5,60 5,41 5,06 4,67 4,51 4,20 3,92
3,77 3,61 3,40 3,30 3,16 3,01 2,92 2,82 2,70 2,61 2,57 2,47 2,40 2,36
2,32 2,28 2,21 2,20 2,19 2,15 2,14 2,13 2,12 2,11 2,10 2,10 2,09 2,08
2,08 2,07 2,05 2,04 2,04 2,04 2,04 2,04 2,04 2,04 2,04

RUN NO. 31

Baseline: 0.75. No. of base points: 97. Total no. of points: 216.
Flowrate: 1,9584 litres/minute. Seconds per point: 0,3000.

0,75	0,75	0,75	0,75	0,75	0,76	0,76	0,76	0,77	0,77	0,78	0,78	0,79
0,79	0,84	0,85	0,91	0,96	1,00	1,07	1,20	1,35	1,46	1,62	1,86	2,09
2,53	2,73	3,00	3,36	3,80	4,00	4,37	4,54	4,87	5,13	5,50	5,73	6,04
6,45	6,65	6,80	6,89	6,92	6,95	6,98	7,00	6,99	6,98	6,93	6,89	6,77
6,45	5,15	5,91	5,69	5,51	5,31	5,06	4,32	4,63	4,30	4,10	3,88	3,63
3,30	3,08	2,97	2,73	2,55	2,47	2,26	2,13	2,01	1,87	1,70	1,60	1,49
1,38	1,30	1,25	1,20	1,15	1,08	1,00	0,99	0,97	0,95	0,91	0,87	0,85
0,80	0,79	0,77	0,76	0,73	0,72	0,72	0,71	0,71	0,70	0,70	0,67	0,65
0,63	0,62	0,62	0,61	0,61	0,61	0,61	0,61					

RUN NO. 32

Baseline: 0.61. No. of base points: 97. Total no. of points: 221.
Flowrate: 1,9584 litres/minute. Seconds per point: 0,3000.

0,61	0,61	0,61	0,61	0,62	0,62	0,63	0,63	0,64	0,65	0,66	0,67	0,68	0,69
0,69	0,70	0,71	0,75	0,79	0,85	0,91	1,05	1,18	1,23	1,39	1,62	1,79	1,96
2,19	2,50	2,72	3,02	3,38	3,81	4,19	4,42	4,95	5,17	5,49	5,80	6,17	6,45
6,75	6,94	7,29	7,47	7,59	7,72	7,80	7,90	7,93	7,32	7,91	7,90	7,78	7,69
7,59	7,32	7,05	6,81	6,60	6,40	5,96	5,73	5,52	5,10	4,81	4,52	4,27	4,02
3,80	3,52	3,33	3,07	2,99	2,81	2,53	2,42	2,30	2,16	2,00	1,90	1,72	1,67
1,60	1,50	1,45	1,35	1,28	1,22	1,15	1,12	1,11	1,10	1,05	1,01	1,00	0,99
0,98	0,97	0,96	0,92	0,90	0,88	0,87	0,84	0,81	0,80	0,79	0,78	0,74	0,73
0,71	0,70	0,69	0,68	0,67	0,66	0,65	0,64	0,63	0,62	0,61	0,61	0,61	

RUN NO. 33

Baseline: 0.32. No. of base points: 73. Total no. of points: 160.
Flowrate: 2,6694 litres/minute. Seconds per point: 0,3000.

0,32	0,32	0,33	0,35	0,36	0,37	0,38	0,39	0,40	0,40	0,41	0,42	0,47	0,60
0,68	0,75	1,02	1,21	1,55	1,83	2,23	2,65	3,15	3,80	4,37	4,82	5,40	6,00
6,50	6,95	7,30	7,61	7,78	8,06	8,17	8,21	8,21	8,19	8,15	7,96	7,70	7,29
6,85	6,43	5,92	5,50	5,16	4,65	4,28	3,80	3,48	3,31	3,00	2,71	2,50	2,29
2,08	1,98	1,72	1,47	1,38	1,29	1,16	1,00	0,95	0,92	0,87	0,81	0,79	0,70
0,65	0,60	0,50	0,47	0,45	0,40	0,39	0,37	0,36	0,36	0,35	0,35	0,35	0,34
0,33	0,33	0,33	0,32										

RUN NO. 34

Baseline: 0.30. No. of base points: 73. Total no. of points: 162.
Flowrate: 2,6694 litres/minute. Seconds per point: 0,3000.

0,30	0,30	0,30	0,30	0,30	0,30	0,30	0,30	0,31	0,32	0,37	0,39	0,41	
0,60	0,73	0,85	1,15	1,47	1,85	2,23	2,61	3,15	3,60	4,30	4,73	5,30	5,72
6,05	6,55	6,84	7,15	7,39	7,47	7,53	7,62	7,62	7,54	7,50	7,20	6,85	6,60
5,10	5,75	5,40	4,93	4,39	4,12	3,70	3,48	3,19	2,72	2,60	2,40	2,2	2,00

1,83 1,65 1,59 1,37 1,23 1,18 1,11 1m08 0,98 0,90 0,85 0,81 0,70 0,65
 0,59 0,55 0,52 0,50 0,45 0,42 0,40 0,39 0,38 0,37 0,35 0,32 0,31 0,31
 0,30 0,30 0,30 0,30 0,30 0,30

RUN NO. 35

Baseline: 0,23. No. of base points: 49. Total no. of points: 136.

Flowrate: 3,3374 litres/minute. Seconds per point: 0,3000.

0,23 0,23 0,23 0,23 0,23 0,23 0,23 0,23 0,24 0,24 0,25 0,25 0,26 0,27
 0,28 0,30 0,31 0,32 0,37 0,44 0,57 0,80 1,05 1,50 1,91 2,38 3,19 4,09
 4,60 5,35 6,30 6,80 7,40 7,82 8,15 8,32 8,42 8,43 8,42 8,36 7,95 7,41
 6,85 6,03 5,50 5,00 4,60 3,95 3,47 3,02 2,75 2,42 2,15 1,90 1,68 1,47
 1,25 1,12 1,10 1,05 0,90 0,80 0,75 0,63 0,58 0,55 0,51 0,40 0,49 0,45
 0,44 0,40 0,39 0,38 0,37 0,36 0,35 0,34 0,33 0,32 0,31 0,30 0,29 0,28
 0,27 0,26 0,25 0,23

RUN NO. 36

Baseline: 0,11. No. of base points: 49. Total no. of points: 146.

Flowrate: 3,3374 litres/minute. Seconds per point: 0,3000.

0,11 0,11 0,11 0,11 0,11 0,12 0,12 0,13 0,13 0,14 0,14 0,15 0,15 0,16
 0,16 0,17 0,18 0,19 0,20 0,25 0,30 0,39 0,60 0,82 1,18 1,57 2,09 2,75
 3,46 4,30 4,85 5,55 6,30 6,83 7,35 7,80 8,09 8,19 8,22 8,21 8,10 7,73
 7,35 6,65 6,10 5,65 5,02 4,45 4,00 3,53 3,12 2,75 2,30 2,15 1,80 1,50
 1,40 1,19 1,01 0,99 0,90 0,85 0,75 0,68 0,63 0,59 0,52 0,44 0,41 0,39
 0,35 0,29 0,26 0,25 0,23 0,22 0,21 0,21 0,20 0,20 0,19 0,19 0,18 0,18
 0,17 0,17 0,16 0,16 0,15 0,15 0,15 0,14 0,13 0,13 0,12 0,12 0,12 0,11

APPENDIX ix

The tables given below summarise the reaction experiments carried out in the packed tubular reactor. The experimental outlet concentrations are compared with those predicted by the ideal tubular reactor model for each component. All concentrations are in gram molecules per litre, and all times in seconds.

It was decided in 3.1 that the temperature rise in the reactor would be held to 1°C. In practice this was possible for all runs except 4 and 5, where the inlet-outlet temperature difference rose to 1.8°C. These runs were included, however, and from Figure 9.7 it is seen that the results are compatible with those of the other runs.

The reaction temperatures reported are the arithmetic mean of inlet and outlet temperatures recorded during the experiments.

For some of the experiments the inlet concentrations were assumed to be sufficiently reproducible so as not to have to be calculated at each flowrate. The inlet concentrations used for all flows are thus reported, together with the residence times for each run. The agreement between ideal and actual x_{sf} values for these cases shows that this approach was justified.

As discussed in 7.1, for several experiments only the caustic soda outlet concentration was measured. For these, therefore, only the experimental and predicted outlet caustic concentrations are given in the table.

EXPERIMENT 1

Column length: 149.8 cm. Inlet type: T. Temperature: 24.6°C.

x_{10}	x_{20}	x_{30}	x_{40}	Residence Time
0.00862	0.02463	0.00160	0.00007	87.3
0.00861	0.02465	0.00160	0.00007	74.3
0.00861	0.02464	0.00160	0.00007	57.0
0.00874	0.02425	0.00152	0.00007	30.0

x_{1f}		x_{2f}		x_{3f}		x_{5f}	
Actual	Ideal	Actual	Ideal	Actual	Ideal	Actual	Ideal
0,00385	0,00326	0,01853	0,01771	0,00504	0,00539	0,00140	0,00163
0,00409	0,00369	0,01888	0,01843	0,00486	0,00522	0,00132	0,00136
0,00459	0,00441	0,01966	0,01950	0,00466	0,00486	0,00103	0,00101
0,00617	0,00606	0,02127	0,02115	0,00380	0,00388	0,00047	0,00049

EXPERIMENT 2

Column length: 149,9 cm. Inlet type: T. Temperature: 22,4°C.

x_{10}	x_{20}	x_{30}	x_{50}	Residence time
0,05181	0,1068	0,00000	0,00024	103,8
0,05175	0,1070	0,00000	0,00024	75,9
0,05162	0,1072	0,00000	0,00024	46,6
0,05159	0,1073	0,00000	0,00024	34,2
0,05138	0,1067	0,00000	0,00024	26,8

x_{1f}		x_{2f}		x_{3f}		x_{5f}	
Actual	Ideal	Actual	Ideal	Actual	Ideal	Actual	Ideal
0,00	0,0367	0,03897	0,03455	0,02072	0,02399	0,02381	0,02439
0,006	0,0587	0,04411	0,04168	0,02314	0,02647	0,02010	0,01964
0,01261	0,01088	0,05451	0,05386	0,02531	0,02812	0,01394	0,01286
0,01660	0,0502	0,06224	0,06166	0,02495	0,02753	0,01029	0,00928
0,02006	0,01884	0,06729	0,06693	0,02423	0,02632	0,00783	0,00696

EXPERIMENT 3

Column length: 149,8 cm. Inlet type: T. Temperature: 22,2°C.

x_{10}	x_{20}	x_{30}	x_{50}	Residence time
0,05090	0,1070	0,00048	0,00332	103,5
0,05092	0,1070	0,00048	0,00032	76,0
0,05073	0,1073	0,00048	0,00032	46,6
0,05095	0,1069	0,00048	0,00032	34,0
0,05122	0,1063	0,00048	0,00032	26,9

x_{1f}		x_{2f}		x_{3f}		x_{5f}	
Actual	Ideal	Actual	Ideal	Actual	Ideal	Actual	Ideal
0,01004	0,00361	0,04277	0,03569	0,01799	0,02375	0,02367	0,02433
0,00832	0,00583	0,04531	0,04258	0,02304	0,02629	0,01986	0,01959
0,012	0,01079	0,05574	0,05485	0,02561	0,02790	0,01355	0,01284
0,01646	0,01514	0,06225	0,06220	0,02520	0,02740	0,01008	0,00920
0,01950	0,01893	0,06739	0,06727	0,02459	0,02621	0,00773	0,00693

EXPERIMENT 4

Column length: 149,8 cm. Inlet type: T. Temperature: 22,8°C.

X_{1o}	X_{2o}	X_{3o}	X_{5o}	Residence time
0,09952	0,2416	0,00755	0,00031	46,0
0,09824	0,2447	0,00746	0,00031	34,2
0,09890	0,2434	0,00750	0,00031	76,5
0,09921	0,2424	0,00753	0,00031	32,7

X_{1f}		X_{2f}		X_{3f}		X_{5f}	
Actual	Ideal	Actual	Ideal	Actual	Ideal	Actual	Ideal
0,00512	0,00531	0,09143	0,09083	0,04615	0,04518	0,05612	0,05689
0,01036	0,00849	0,1100	0,1082	0,04851	0,05044	0,01714	0,04707
0,00261	0,00160	0,07813	0,07225	0,03465	0,03079	0,06935	0,07421
0,01158	0,00963	0,1103	0,1079	0,05074	0,05225	0,04474	0,04518

EXPERIMENT 5a

Column length: 149,8 cm. Inlet type: T. Temperature: 21,5°C.

X_{1o}	X_{2o}	X_{3o}	X_{5o}	Residence time
0,09757	0,2439	0,00906	0,00032	46,5
0,09911	0,2426	0,00911	0,00032	34,1
0,09862	0,2413	0,00916	0,00032	76,9
0,09767	0,2437	0,00907	0,00032	46,5

X_{1f}		X_{2f}		X_{3f}		X_{5f}	
Actual	Ideal	Actual	Ideal	Actual	Ideal	Actual	Ideal
0,00468	0,00588	0,09826	0,09783	0,04928	0,04642	0,05302	0,05465
0,01079	0,01041	0,1118	0,1114	0,05297	0,05338	0,04378	0,04375
0,00532	0,00216	0,08105	0,07373	0,03546	0,03447	0,06732	0,07146
0,00544	0,00590	0,09835	0,09754	0,04823	0,04650	0,05339	0,05465

EXPERIMENT 5b

Column length: 149,8 cm. Inlet type: T. Temperature: 21,2°C.

X_{1o}	X_{2o}	X_{3o}	X_{5o}	Residence time
0,09791	0,2430	0,00909	0,00032	57,5
0,09841	0,2419	0,00913	0,00032	39,1
0,09783	0,2432	0,00908	0,00032	76,3
0,09806	0,2428	0,00909	0,00032	113,7

X_{3f}	
Actual	Ideal
0,09100	0,08929
0,1061	0,1068
0,08134	0,07677
0,07331	0,06475

x_{1f}		x_{2f}		x_{3f}		x_{4f}	
Actual	Ideal	Actual	Ideal	Actual	Ideal	Actual	Ideal
0,00583	0,00590	0,01710	0,01701	0,00479	0,00455	0,00050	0,00066
0,00807	0,00833	0,01921	0,01925	0,00332	0,00283	0,00000	0,00018
0,00508	0,00482	0,01537	0,01494	0,00528	0,00537	0,00094	0,00111
0,00849	0,00888	0,01960	0,01971	0,00307	0,00239	0,00000	0,00012
0,00727	0,00749	0,01864	0,01860	0,00393	0,00344	0,00000	0,00030

EXPERIMENT 8b

Column length: 149,8 cm. Inlet type: T. Temperature: 21,1°C.

x_{1c}	x_{2c}	x_{3c}	x_{4c}
0,01096	0,02222	0,00030	0,00000

x_{2f}		Residence time
Actual	Ideal	
0,01793	0,01785	57,8
0,01937	0,01953	29,8
0,01886	0,01888	38,8
0,01587	0,01598	90,1
0,01948	0,01969	25,2

EXPERIMENT 9

Column length: 149,8cm. Inlet type: T. Temperature: 22,4°C.

x_{1c}	x_{2c}	x_{3c}	x_{4c}	Residence time
0,02524	0,05846	0,00138	0,00004	46,5
0,02505	0,05888	0,00137	0,00004	75,0
0,02535	0,05821	0,00139	0,00004	26,4
0,02529	0,05834	0,00139	0,00004	112,5
0,02520	0,05854	0,00138	0,00004	33,6

x_{1f}		x_{2f}		x_{3f}		x_{4f}	
Actual	Ideal	Actual	Ideal	Actual	Ideal	Actual	Ideal
0,01037	0,00934	0,05364	0,03894	0,01230	0,01366	0,00399	0,00367
0,00617	0,00576	0,03350	0,03333	0,01233	0,01440	0,00646	0,00630
0,01438	0,01379	0,04537	0,04494	0,01060	0,01124	0,00185	0,00175
0,00657	0,00356	0,03049	0,02751	0,01097	0,01401	0,00917	0,00914
0,01214	0,01183	0,04309	0,04277	0,01204	0,01237	0,00243	0,00243

EXPERIMENT 10a

Column length: 149,8 cm Inlet type: T. Temperature: 23,2°C.

X_{10}	X_{20}	X_{30}	X_{50}	Residence time
0,02503	0,05887	0,00136	0,00005	75,1
0,02509	0,05872	0,00136	0,00005	33,8
0,02527	0,05833	0,00137	0,00005	112,7
0,02505	0,05882	0,00136	0,00005	46,0
0,02527	0,05832	0,00137	0,00005	26,7

X_{1f}		X_{2f}		X_{3f}		X_{5f}	
Actual	Ideal	Actual	Ideal	Actual	Ideal	Actual	Ideal
0,00554	0,00535	0,03254	0,03251	0,01401	0,01436	0,00688	0,00612
0,01152	0,01122	0,04236	0,04223	0,01215	0,01262	0,00284	0,00267
0,00610	0,00325	0,02952	0,02871	0,01090	0,01379	0,00969	0,00965
0,01051	0,00880	0,04027	0,03871	0,01187	0,01373	0,00097	0,00392
0,01332	0,01317	0,04430	0,04432	0,01125	0,01157	0,00000	0,00195

EXPERIMENT 10b

Column length: 149,8 cm. Inlet type: T. Temperature: 23,2°C.

X_{10}	X_{20}	X_{30}	X_{50}
0,02514	0,05861	0,00136	0,00005

X_{2f}		Residence time
Actual	Ideal	
0,03610	0,03517	36,9
0,04343	0,04328	29,8
0,03067	0,02999	90,0
0,04411	0,04382	28,1

EXPERIMENT 11

Column length: 73,0 cm. Inlet type: Ring main. Temperature: 21,7°C.

X_{10}	X_{20}	X_{30}	X_{50}
0,04773	0,1195	0,00205	0,00000

X_{2f}		Residence time
Actual	Ideal	
0,08870	0,08900	17,4
0,08252	0,08346	23,4
0,07113	0,07174	37,4
0,08951	0,08916	17,6
0,07864	0,07888	28,4
0,06629	0,06663	45,0
0,08788	0,08757	19,2
0,06735	0,06663	45,0
0,08164	0,08171	25,3
0,08624	0,08573	20,8
0,07511	0,07544	32,2

x_{sf}		Residence time
Actual	Ideal	
0,07319	0,07150	37,5
0,08152	0,08145	25,3
0,08600	0,08560	20,9
0,07503	0,07544	32,2
0,07840	0,07871	28,3
0,08788	0,08370	19,2
0,06015	0,06070	56,5

EXPERIMENT 12

Column length: 149,8 cm. Inlet type: Ring main. Temperature: 25,0°C.

x_{10}	x_{20}	x_{30}	x_{50}
0,05485	0,1163	0,00285	0,00000

x_{sf}		Residence time
Actual	Ideal	
0,06793	0,06741	26,9
0,05237	0,05297	46,4
0,03603	0,03465	104,9
0,04282	0,04305	74,2
0,06074	0,06663	33,2
0,04725	0,04851	55,5
0,06433	0,06466	29,0
0,05705	0,05795	37,9
0,03747	0,03760	88,7
0,05105	0,05126	9,6
0,06279	0,06248	31,8
0,06537	0,06517	8,2
0,04434	0,04472	65,2
0,03580	0,03440	104,7
0,05336	0,05297	46,4
0,04111	0,04111	75,8
0,06074	0,06063	33,9

EXPERIMENT 13

Column length: 149,8. Inlet type: Ring main. Temperature: 26,0°C.

x_{10}	x_{20}	x_{30}	x_{50}	Residence time
0,04669	0,09694	0,00065	0,00000	42,5
0,04673	0,09686	0,00085	0,00000	26,7
0,04692	0,09646	0,00085	0,00000	51,9
0,04669	0,09694	0,00085	0,00000	33,0

x_{1f}		x_{2f}		x_{3f}		x_{5f}	
Actual	Ideal	Actual	Ideal	Actual	Ideal	Actual	Ideal
0,00795	0,00890	0,04606	0,04626	0,02509	0,02574	0,01450	0,01289
0,01387	0,01447	0,05705	0,05665	0,02453	0,02517	0,00918	0,00794
0,00462	0,00559	0,03694	0,03742	0,02341	0,02448	0,01974	0,01770
0,01103	0,01178	0,05166	0,05199	0,02527	0,02574	0,01124	0,01002

APPENDIX A

The tables below contain the complete set of kinetic data used to obtain the reaction velocity constants reported in section 2.3

4. All concentrations are in gram moles per litre, and all times in minutes, and temperatures in °C.

1. Kinetic Data from J. Meyer (1909).

TABLE 1

Temp.	x_{10}	x_{20}	x_{30}
18	0	0,01	0,01268

Time	x_p
2	0,008152
5	0,006112
8	0,005000
11	0,004256
15	0,003320
20	0,002560
25	0,002000
30	0,001656
45	0,000872
60	0,000616

TABLE 2

Temp.	x_{10}	x_{20}	x_{30}
18	0	0,05	0,04906

Time	x_2
2	0,02880
5	0,01850
8	0,01368
11	0,01108
14	0,00832
17	0,00728
20	0,00636
25	0,00528
30	0,00460
45	0,00328

TABLE 3

Temp.	x_{10}	x_{20}	x_{30}
25	0	0,01	0,01268

Time	x_2
2	0,007024
4	0,005308
7	0,003776
10	0,002840
13	0,002272
16	0,001840
20	0,001392
30	0,000768
45	0,000420
60	0,000112

TABLE 4

Temp.	x_{10}	x_{20}	x_{30}
25	0	0,02	0,01932

Time	x_2
2	0,01227
5	0,007800
7	0,005660
11	0,004542
14	0,003740
17	0,003340
20	0,002923
25	0,002481
30	0,002140
45	0,001600

TABLE 5

Temp.	x_{10}	x_{20}	x_{30}
18	0,02000	0,02	0
Time	x_2		
2	0,01493		
5	0,01116		
8	0,008976		
11	0,007480		
15	0,006096		
20	0,005184		
25	0,004400		
30	0,003624		
45	0,002360		
60	0,002064		

TABLE 6

Temp.	x_{10}	x_{20}	x_{30}
18	0,01000	0,01	0
Time	x_2		
2	0,00640		
5	0,007272		
8	0,006200		
11	0,005512		
15	0,004620		
20	0,004048		
25	0,003472		
30	0,003040		
45	0,002304		
60	0,001800		

TABLE 7

Temp.	x_{10}	x_{20}	x_{30}
25	0,0174	0,01	0
Time	x_2		
2	0,007200		
5	0,004952		
8	0,003688		
11	0,002880		
14	0,002308		
17	0,001968		
20	0,001592		
25	0,001320		
30	0,001020		

TABLE 8

Temp.	x_{10}	x_{20}	x_{30}
25	0,05286	0,05	0
Time	x_2		
2	0,01740		
5	0,008552		
8	0,005340		
11	0,003600		
14	0,002760		
17	0,002180		
20	0,001820		
25	0,001300		
30	0,000920		
45	0,000296		

2. Kinetic Data from Batch Runs with Diacetate

Monoacetate concentration, x_{30} , assumed to be zero for all these data.

Temp.	x_{10}	x_{20}
25,0	0,0491	0,1012

Temp.	x_{10}	x_{20}
25,0	0,05029	0,09884

Time	X_2
0,25	0,07000
0,50	0,05322
0,75	0,04454
1,00	0,03842
1,50	0,03026
2,00	0,02548
3,00	0,01997
5,00	0,01410
10,00	0,00927
15,00	0,00782

Time	X_2
0,25	0,07116
0,50	0,05427
0,75	0,04253
1,00	0,03610
1,50	0,02824
2,00	0,02300
3,00	0,01737
5,00	0,01151
10,25	0,00629
15,00	0,00480

Temp.	X_{10}	X_{20}
25,0	0,004739	0,01073

Time	X_2
0,25	0,01038
0,50	0,009959
0,75	0,009586
1,00	0,009234
1,50	0,008636
2,00	0,008142
3,00	0,007323
5,00	0,006151
10,00	0,004599
15,00	0,003806

Temp.	X_{10}	X_{20}
25,0	0,004781	0,01064

Time	X_2
0,25	0,01013
0,50	0,009895
0,75	0,009445
1,00	0,009118
1,50	0,008549
2,00	0,008074
3,00	0,007217
5,00	0,006080
10,00	0,004534
15,00	0,003747

Temp.	X_{10}	X_{20}
21,5	0,04756	0,1044

Time	X_2
0,25	0,07991
0,50	0,06405
0,75	0,05443
1,00	0,04846
1,50	0,03946
2,00	0,03426
3,00	0,02666
5,00	0,02093
10,00	0,01441
15,00	0,01250

Temp.	X_{10}	X_{20}
21,5	0,04638	0,1067

Time	X_2
0,25	0,08214
0,50	0,06744
0,75	0,05758
1,00	0,04966
1,50	0,04229
2,00	0,03662
3,00	0,02979
5,00	0,02281
10,00	0,01765
15,00	0,01615

Temp.	x_{10}	x_{20}
21,5	0,005076	0,01036

Time	x_2
0,25	0,01036
0,50	0,019788
0,75	0,009430
1,00	0,009156
1,50	0,008564
2,00	0,008196
3,00	0,007376
5,00	0,006260
10,00	0,004627
15,00	0,003748

Temp.	x_{10}	x_{20}
21,5	0,005104	0,01030

Time	x_2
0,25	0,01034
0,50	0,009734
0,75	0,009559
1,00	0,009070
1,50	0,008663
2,00	0,008085
3,00	0,007394
5,00	0,006163
10,00	0,004552
15,00	0,003669

Temp.	x_{10}	x_{20}
21,5	0,09550	0,2361

Time	x_2
0,25	0,1492
0,50	0,1130
0,75	0,09496
1,00	0,08349
1,50	0,07018
2,00	0,06143
3,00	0,05380
5,00	0,04825
10,00	0,04639
15,00	0,04624

Temp.	x_{10}	x_{20}
21,5	0,09245	0,2429

Time	x_2
0,25	0,1657
0,50	0,1239
0,75	0,1042
1,00	0,09250
1,50	0,07984
2,00	0,07037
3,00	0,06356
5,00	0,05925
10,00	0,05808
15,00	0,05795

Temp.	x_{10}	x_{20}
17,0	0,09900	0,2273

Time	x_2
0,25	0,1580
0,50	0,1195
0,75	0,09755
1,00	0,08555
1,50	0,07031
2,00	0,05960
3,00	0,05000
5,00	0,03954
10,00	0,03177
15,00	0,03044

Temp.	x_{10}	x_{20}
17,0	0,09802	0,2295

Time	x_2
0,25	0,1606
0,50	0,1206
0,75	0,1014
1,00	0,08797
1,50	0,07133
2,00	0,06337
3,00	0,05195
5,00	0,04117
10,00	0,03547
15,00	0,03494

Temp.	X_{10}	X_{20}
17,0	0,04776	0,1037
Time	X_2	
0,25	0,08599	
0,50	0,07064	
0,75	0,06082	
1,00	0,05464	
1,50	0,04543	
2,00	0,03946	
3,00	0,03168	
5,00	0,02352	
10,00	0,01555	
15,00	0,01277	

Temp.	X_{10}	X_{20}
17,0	0,04891	0,1014
Time	X_2	
0,25	0,07978	
0,50	0,06787	
0,75	0,05943	
1,00	0,05240	
1,50	0,04318	
2,00	0,03738	
3,00	0,02923	
5,00	0,02056	
10,00	0,01312	
15,00	0,01024	

Temp.	X_{10}	X_{20}
17,0	0,004548	0,01107
Time	X_2	
0,25	0,01083	
0,50	0,01059	
0,75	0,01026	
1,00	0,01009	
1,50	0,009657	
2,00	0,009321	
3,00	0,008687	
5,00	0,007679	
10,00	0,006152	
15,00	0,005217	

Temp.	X_{10}	X_{20}
17,0	0,004498	0,01119
Time	X_2	
0,25	0,01095	
0,50	0,01057	
0,75	0,01033	
1,00	0,01017	
1,50	0,009752	
2,00	0,009413	
3,00	0,008798	
5,00	0,007847	
10,00	0,006250	
15,00	0,005323	

3. Kinetic Data from Batch Runs with Monoacetate

Temp.	X_{10}	X_{20}	X_{30}
24,0	0,01360	0,04733	0,02972
Time	X_2		
0,10	0,04183		
0,27	0,03726		
0,50	0,03214		
0,67	0,02782		
0,83	0,02626		
1,00	0,02368		
1,17	0,02138		
1,33	0,02003		
1,50	0,01763		

Temp.	X_{10}	X_{20}	X_{30}
24,0	0,01278	0,04982	0,02872
Time	X_2		
0,10	0,04537		
0,25	0,04087		
0,50	0,03485		
0,67	0,03165		
0,83	0,02879		
1,00	0,02700		
1,33	0,02361		
1,57	0,02065		
2,00	0,01817		

Temp.	x_{10}	x_{20}	x_{30}
24,0	0,01161	0,05473	0,02502

Time	x_2
0,10	0,05014
0,43	0,04253
0,58	0,03909
0,75	0,03624
1,00	0,03300
1,33	0,02930
1,67	0,02659
2,00	0,02459

Temp.	x_{10}	x_{20}	x_{30}
24,0	0,01173	0,05425	0,02529

Time	x_2
0,10	0,35050
0,25	0,04510
0,42	0,04156
0,58	0,03848
0,75	0,03550
1,00	0,03213
1,33	0,02870
1,67	0,02618
2,00	0,02397

4. Kinetic Data from Plug Reactor Runs

Temp.	x_{10}	x_{20}	x_{30}
25,0	0,05367	0,1188	0,00279

Time	x_2
0,43	0,06793
0,51	0,06279
0,53	0,06074
0,58	0,05973
0,62	0,05705
0,67	0,05571
0,74	0,05310
0,81	0,05105
0,91	0,04725
1,04	0,04434
1,18	0,04282
1,20	0,04119
1,45	0,02747
1,66	0,03110
1,67	0,03603

Temp.	x_{10}	x_{20}	x_{30}
21,7	0,04772	0,1195	0,00205

Time	x_2
0,28	0,03870
0,28	0,03951
0,31	0,03788
0,31	0,03787
0,33	0,03612
0,37	0,03252
0,40	0,03158
0,45	0,02864
0,45	0,02840
0,51	0,02507
0,60	0,02113
0,72	0,01682
0,90	0,01015

APPENDIX xi1. EQUIPMENT LIST

All electrical equipment 220V single phase unless otherwise stated.

Automatic Titrator	Radiometer TTT 11b.
Balances:	Mettler H 10. Used for density measurement. Mettler P 1200 N. Used for titrations. Mettler K 4 T. Used for flowmeter calibrations.
Conductivity Meter:	Wayne Kerr Universal Bridge B221 in conjunction with Wayne Kerr Autobalance Adaptor AA 221.
Conductivity Cell:	Philips PW 9510 1 cm. standard cell. Used in residence time distribution experiments.
Control Valves:	Lunkenheimer 316 stainless steel 1/4" needle valves, figure 1732.
Flowmeters:	Fischer Porter Co. precision bore flow-rator tube no. B4.17.10/77. Float no. BSVT-45. Hose clamp connections and end springs 10A 1027A.
Heaters:	1 kw electric kettle element. Four used in caustic and diacetate stock tanks.
Magnetic Stirrer:	Ruhromag MO 13 25 watt, maximum RPM = 2800 Stirrer bars 2-6cm. long, 5 mm. diameter, teflon covered. Used for titrations.
Magnetic Valve:	Radiometer MNV-1C. Used in conjunction with automatic titrator.
pH Meter:	Radiometer PHM 26 with expanded scale.
pH Meter electrodes:	Calomel - Radiometer K 401 TS. Combined glass-calomel - Radiometer GK 2311 C KR.

Pipettes:	Platinum - Radiometer P101 DW. H.J. Elliott Green Line 20 cm ³ A Grade with certificate (used for density measurement).
Recorders:	Bristols Dynamaster 0-1 mV with zero suppression, model 1P124570 x 92-51-T4C-T113A-T71B-T88-T111. Used for residence time distribution experiments 1-20. Rika Denki B024 10 with zero adjustment and attenuator. Used for residence time distribution experiments 21-36.
Scale:	Avery 3205 ABA. 500 lbs. in 1/4 lb. Used for holdup volume measurements.
Still:	Buchi Fontavapour-61 380V 3 phase. Delivery: 5 litres per hour. Fisons Loughborough Still W/SF/8A. Delivery: 10 litres per hour.
Stirrers:	1/4 HP 725 RPM belt driven by Associated Electric Motors BS 2405 856 running at 1425 RPM. Used to stir stock tanks. Heidolph Elektro KGE60, 17 watt adjustable speed stirrer. Set at 200 RPM for batch reactor runs and at 960 RPM when mounted in quench vessel.
Stopwatch:	Omega, 30 second sweep, smallest division 0.1 second.
Temperature Compensator:	Radiometer T501 SU. Used in conjunction with pH meter.
Temperature Controllers:	United Electric Co. DB-85a, 6' stainless steel capillary, stainless steel B bulb range -150 - 200°F. Temperature control to ±1°F. Used in caustic and diacetate stock tanks.
Thermometers:	Labotec 0-100°C, 15" calibrated in 0.1°C. Each thermometer was calibrated against the standard thermometers kept in the Chemical Engineering Department.

Volumetric Flasks:

H.J. Elliott Green Line, 1 litre A grade and certificated. (Used for making up standard solutions.)

2. CHEMICALS LIST

Acetic acid	Riedel de Haen A.G. A.R. Grade.
Chloroform:	Riedel de Haen A.G. A.R. Grade
Ethylene Glycol:	Riedel de Haen A.G. Chromatographic grade.
Ethylene glycol diacetate:	Dow Chemical Co. 98,9 % pure.
Ethylene glycol mono-acetate:	British Drug Houses. Technical Grade.
Hydrochloric acid ampoules:	May and Baker Volucon Standard Solution. Guaranteed accurate to $\pm 0,2\%$
Hydrochloric acid:	Riedel de Haen A.G. A.R. Grade.
Iodine ampoules:	May and Baker Volucon Standard Solution Guaranteed accurate to $\pm 0,2\%$.
Nitrogen:	African Oxygen Ltd. High Purity.
Periodic Acid:	Riedel de Haen A.G. A.R. Grade.
pH Buffer	Radiometer. pH 6,5. Type SI001.
Potassium Iodide:	Protea Laboratory Services. A.R. Grade.
Sodium Hydroxide ampoules:	May and Baker Volucon Standard Solution. Guaranteed accurate to $\pm 0,2\%$.
Sodium Hydroxide:	Konto Chemical Co. Cica brand. A.R. Grade.
Sodium Thiosulphate:	British Drug Houses. A.R. Grade. (Note: Sodium thiosulphate standard solution made up and stored according to methods in Vogel (1951) page 333.

NOMENCLATURE

a	mass of stream 1 sample taken for acetic acid determination (gm)
a_v	cross-sectional area available for flow in reactor (cm^2) (void area)
b	mass of a molar caustic soda used for stream 1 acetic acid determination (gm)
c	molarity of standard caustic soda used in analyses (gm.mole/l)
d	mass of stream 1 sample taken for total acetate determination (gm)
d_p	particle diameter in packed bed reactor (cm)
d_t	diameter of open tube reactor (cm)
e	mass of a molar caustic soda added to stream 1 sample during total acetate determination (gm)
g	mass of potassium iodide and hydrochloric acid solutions added to liberate iodine from periodic acid during glycol analyses (gm)
h	mass of j molar hydrochloric acid used for total acetate determination in stream 1 (gm)
i	used to denote reaction number
j	used to denote component number in i th reaction
j	molarity of standard hydrochloric acid used in analyses (gm.mole/l)
k	reaction rate constant (1/gm.mole min. or 1/gm.mole sec)
k_1	reaction rate constant for reaction I, section 2.2
k_2	reaction rate constant for reaction II, section 2.2
k_∞	Arrhenius constant (1/gm.mole min. or 1/gm.mole sec)
$k_{\infty 1}$	Arrhenius constant for reaction I, section 2.2
$k_{\infty 2}$	Arrhenius constant for reaction II, section 2.2
l	mass of stream 1 sample taken for total glycol determination (gm)
l'	mass of stream 6 sample taken for free glycol determination (gm)

z''	mass of stream 6 sample taken for total glycol determination (gm)
m	mass of periodic acid added to samples for glycol determinations (gm)
p'	molarity of periodic acid in periodic acid solution used for glycol determinations (gm.mole/l)
p''	molarity of iodic acid in periodic acid solution used for glycol determinations (gm.mole/l)
p	$= p' + p''$ (gm.mole/l)
r	molarity of potassium iodide and hydrochloric acid solutions used in the glycol analyses (gm.mole/l)
r_t	radius of open tube reactor (cm)
s	mass of u molar thiosulphate used to titrate liberated iodine in glycol analyses (gm)
t	residence time in reactor (seconds)
t_f	total residence time in reactor = time final (seconds)
u	molarity of thiosulphate used to titrate liberated iodine in glycol analyses (gm.mole/l)
v	mass of periodic acid taken for blank titration (gm)
w	mass of thiosulphate used to titrate liberated iodine in periodic acid blank analyses (gm)
x	mass of stream 2 sample taken for caustic soda analysis (gm)
y	mass of j molar hydrochloric acid used in Stream 2 caustic soda analysis (gm)
z	length down reactor ($z=0$ at inlet) (cm)
A	positive constant used to relate X_3 to profit in optimisation
$C(t)$	concentration distribution function measured during residence time distribution experiments
D_a	axial dispersion coefficient (Taylor diffusion coefficient, longitudinal dispersion coefficient) (cm^2/sec)
D_m	molecular diffusion coefficient (cm^2/sec)
E	activation energy (kcal/gm.mole $^\circ\text{K}$)
E_1	activation energy for reaction 1, section 2.2 (kcal/gm.mole $^\circ\text{K}$)

E_2	activation energy for reaction II, section 2.2 (kcal/gm.mole°K)
$E(t)$	residence time distribution function
G	mass flow rate in reactor (kg/sec or kg/min)
G_{1-6}	mass flows of streams 1-6 in reactor system (see Figure 5.2) (kg/sec or kg/min)
H	Hamiltonian used in optimisation
k	$= k_2/k_1$
k_∞	$= \ln k_\infty$
$k_{\infty 1}$	$= \ln k_{\infty 1}$
$k_{\infty 2}$	$= \ln k_{\infty 2}$
L	reactor length (cm)
M	profit function used in optimisation
Pe_a	axial Peclet number in packed reactor $= U_d \rho / D_a$
Q	volumetric flowrate in reactor (l/sec or l/min)
Q'	volumetric flowrate in reactor (cm ³ /sec)
Q_1-Q_6	volumetric flows of streams 1-6 in reactor system (see Figure 5.2) (l/sec or l/min)
R	gas constant (kcal/gm.mole°K)
R_i	reaction rate for i th reaction (gm.moles/l.sec)
T	reaction temperature (°C or °K)
U	fluid velocity in reactor $= Q'/a_v$ (cm/sec)
V_h	holdup volume in reactor (volume of void spaces) (l)
X	concentration of tracer in reactor (gm.moles/l)
$X_{i,j}$	concentration of j th species in i th reaction, $i=1,2$ $j=1,4$ (gm.mole/l) for species numbers see below
$X_{j0}, X_{j\theta}$ etc.	concentration of j th species in reactor system (gm.mole/l) see subscript list below
$(X_j)^{batch}$	concentration of j th species in a batch reactor (gm.mole/l)
X'_s	total glycol in stream 1 sample $= X'_{1s} + X'_{2s} + X'_{3s}$ (gm.mole/l)

Y_i	extent of i th reaction $i=1,2$ (gm.mole/l)
Y_{i0}	extent of i th reaction at time $t=0$ ($Z=0$)
Y_{if}	extent of i th reaction at time $t=t_f$ ($Z=1$)
Y_3	$\frac{dY_1}{dZ}$
Y_4	$\frac{dY_2}{dZ}$
Y_{3f}, Y_{4f}	Y_3, Y_4 at time $t=t_f$ ($Z=1$)
Z	dimensionless reactor length = z/L
α	mass of stream 5 sample taken for acetic acid analysis (gm)
α_{ij}	stoichiometric coefficient of j th component in i th reaction
β	mass of α molar standard caustic soda used in stream 5 analysis (gm)
$\delta(t)$	dirac delta function
$\lambda_{1..2}$	adjoints used in optimisation
μ	viscosity of fluid in reactor (gm/cm.sec)
ρ	density of fluid in reactor (gm/cm ³)
ρ_{1-6}	densities of streams 1-6 (see Figure 5.2) (kg/l)
ρ_7	density of α molar standard caustic soda (gm/cm ³)
ρ_8	density of j molar standard hydrochloric acid (gm/cm ³)
ρ_9	density of standard periodic acid (gm/cm ³)
ρ_{10}	density of u molar standard thiosulphate (gm/cm ³)
T	variable used in optimisation, as defined by equation 4.6
T_f	value of T at t_f

SUBSCRIPTS

These subscripts refer to reactor system concentrations, see Figure 5.2.

f	outlet stream for reactor (corresponds to time final, t_f)
n	neutralised stream leaving quench vessel

o reactor inlet stream (corresponds to time $t=0$)

a stock streams

Unsubscripted concentrations, i.e. X_j etc., are concentrations in reactor.

STREAM NUMBERS

- | | |
|---|--|
| 1 | diacetate feed |
| 2 | caustic feed |
| 3 | reactor inlet |
| 4 | reactor outlet |
| 5 | acetic acid quench stream |
| 6 | neutralised stream leaving quench vessel |

SPECIES NUMBERS

- | | |
|---|-----------------------------|
| 1 | ethylene glycol diacetate |
| 2 | ethylene glycol monoacetate |
| 3 | caustic soda |
| 4 | sodium acetate |
| 5 | ethylene glycol |
| 6 | acetic acid |

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