

If accurate potential data are not available the porous electrode equation may still be solved using a numerical technique. The mathematical derivation of the underlying equations and details of the calculation sequence are given in Section B.3 of Appendix B.

A Fortran program to solve the porous electrode equation on the basis of the theory outlined in Appendix B was developed. A simplified flowchart and detailed listing of the program are given in Appendix C.

The version given in the appendix is that used as a subroutine in fitting the experimental results for the removal of nickel from dilute solution. As such it contains a loop for calculating the sum of squared errors between experimental and theoretical results. Other slightly modified versions of the program were used in fitting the results of copper removal experiments, for producing plots of potential and current density profiles and for predicting the performance of a complete electrochemical reactor system.

3. EXPERIMENTAL APPARATUS AND PROCEDURE

3.1 Introduction

The apparatus and experimental techniques used in the investigation of nickel removal from dilute solution are discussed in some detail in this section. A brief reference is made to the apparatus and techniques used by Young (11) in his study of copper removal in order to clarify the experimental basis of the results presented in Section 4.

3.2 Electrochemical Removal of Nickel from Dilute Solution

3.2.1 Description of Apparatus

The process of nickel removal was studied by means of a batch recirculation system.

The apparatus used in the investigation is shown schematically in Figure 3.1. The entire flow system was constructed either of glass or of plastic to avoid any contamination of the circulating solutions.

Catholyte circulation was by means of an Iwaki MDH-25 glandless magnet pump of polypropylene construction. The solution was circulated from a fifty litre tank through a system of valves and rotameters into the base of the cathode compartment of the packed bed cell. The catholyte leaving the cell returned by gravity to the catholyte tank. Sampling points were provided at the cell inlet and outlet.

Three accurately calibrated rotameters in parallel were provided for measurement of the catholyte flowrate, which was controlled by means of plastic diaphragm valves. A side stream was diverted through a flow-through conductivity cell. This cell had been previously calibrated in a water bath at 25°C using standard potassium chloride solutions of known

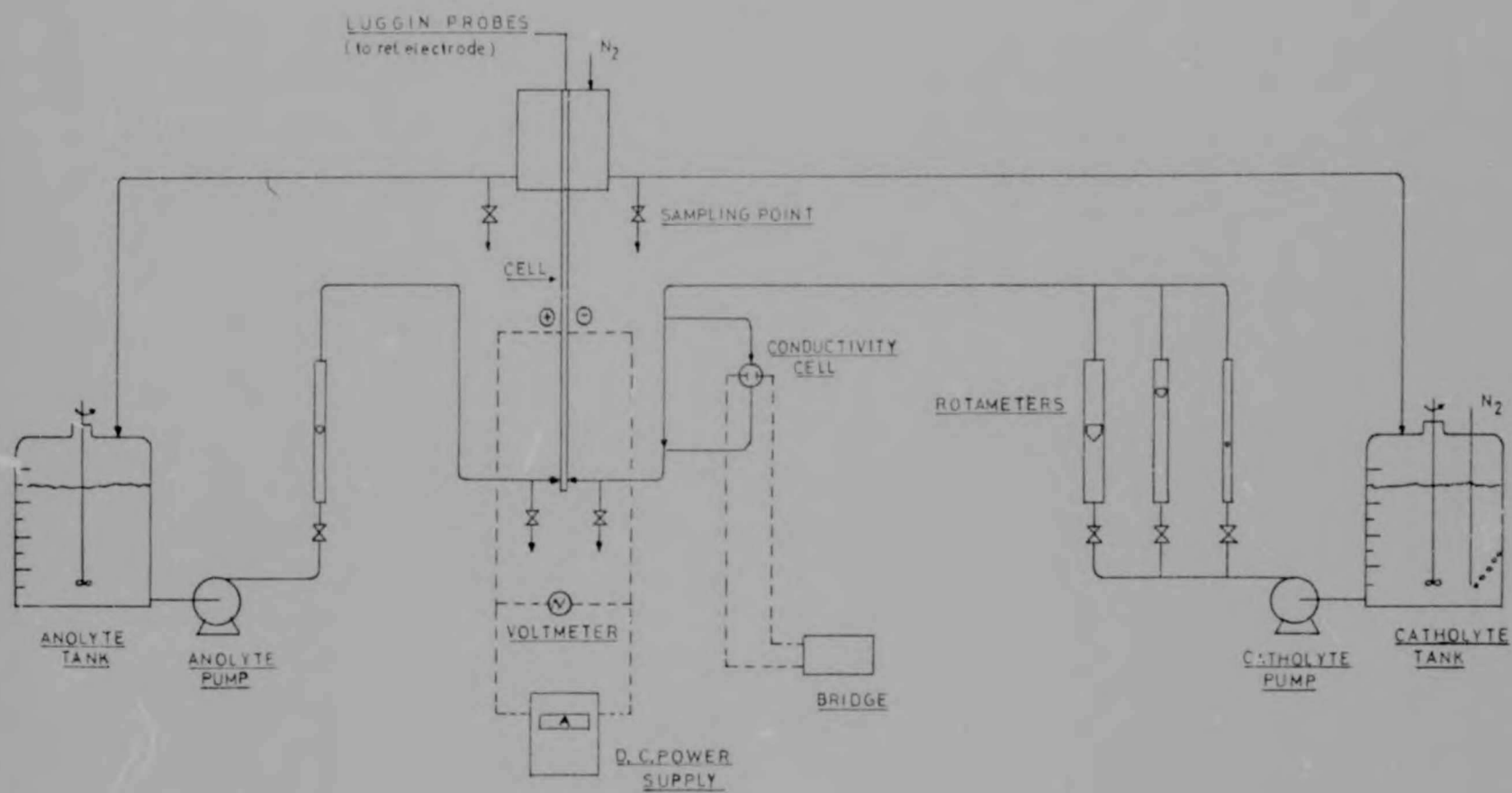


FIGURE 3.1 SCHEMATIC DIAGRAM SHOWING THE LAYOUT OF APPARATUS USED IN NICKEL EXPERIMENTS

specific conductivity.

The catholyte tank was marked with graduations at five litre intervals so that any desired volume of catholyte could be circulated. This indicated volume included the volume of solution in the packed bed cell and remainder of the flow system. A three litres per minute stream of commercial nitrogen gas ($>95\% \text{N}_2$) was bubbled through the catholyte which was stirred continuously. A stream of nitrogen was also introduced into the top of the packed bed cell.

The anolyte circulation system was similar but less complex and comprised a tank, MD-30 magnet pump and single plastic diaphragm valve and rotameter. Sampling points were provided and the solution in the anolyte tank was stirred continuously.

Electric current to the packed bed cell was provided by a Kepco JQE 15-50 fifty amp power supply operated in the constant current mode. The cell current was determined by measurement of the potential drop across a precision ($\pm 1\%$) ten milliohm resistor by means of a Radiometer PHM64 digital voltmeter. The cell voltage was measured on a Comark Type 120 D.C. voltmeter.

The conductance of the catholyte flowing through the conductivity cell was measured on a Wayne-Kerr B530 universal bridge. Multiplication of the measured conductance by the cell constant then gave the solution specific conductivity.

The potential of the packed bed cathode was measured at the current feeder and membrane by means of Luggin probes connected to two Radiometer B530 saturated calomel reference electrodes. A Radiometer PHM 64 digital millivoltmeter, of input impedance greater than

10^{12} ohms, was used for the potential measurements. The reference electrodes were periodically compared against each other and against a third electrode and were always found to be within five millivolts of each other.

3.2.2. Description of Packed Bed Electrolytic Cell

The packed bed cell used in the investigation and illustrated in Figure 3.2 was constructed of perspex, which has the advantages of being easily machined and of being electrically non-conducting.

Catholyte was pumped into the cathode compartment through two inlets at the base of the cell and then flowed upward, first through an entrance region 100 millimetres long packed with glass beads and then through the packed bed cathode itself. The cathode was made up of uniformly sized nickel-plated lead spheres and was supplied with electrical current through a current feeder made of copper plate which ran the length and breadth of the packed bed. Catholyte leaving the top of the bed overflowed a weir and was collected in a perspex collecting tank, from which the solution returned by gravity through four outlet pipes to the catholyte tank.

The cell was constructed to facilitate variation of the bed thickness, which was adjusted by inserting rubber gaskets of different thickness between the perspex backing of the cathode compartment and the Ionac MA3475 anion permeable membrane separating the anode and cathode compartments. The membrane effectively prevented Ni^{2+} cations transferring from the catholyte to the anolyte but did permit some limited diffusion and migration of highly mobile H^+ ions from anolyte to catholyte.

Pertinent details of the packed bed cathode are given in Table 3.1 and further details of the cell construction are shown in Figure 3.3. The bed thickness

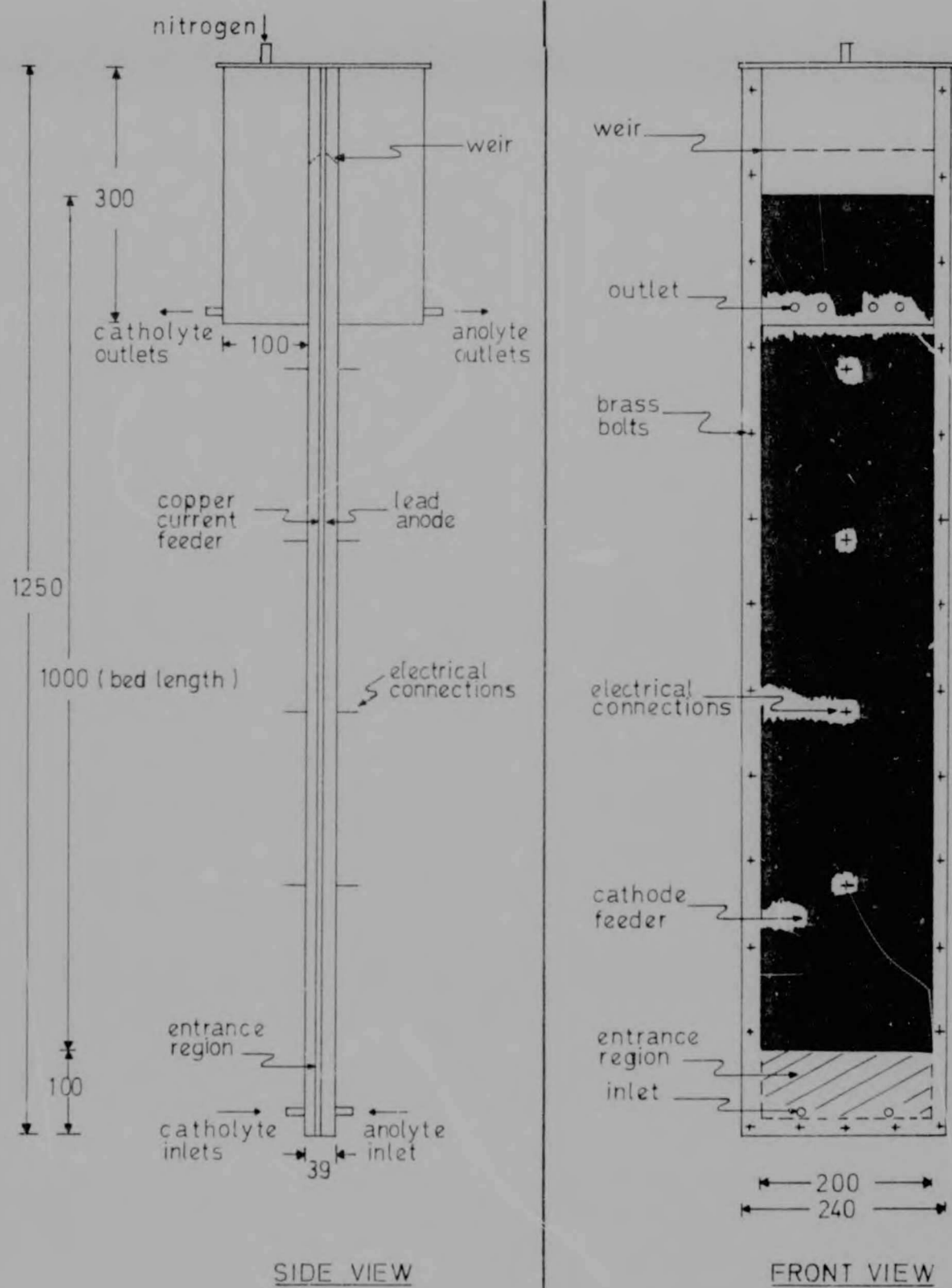
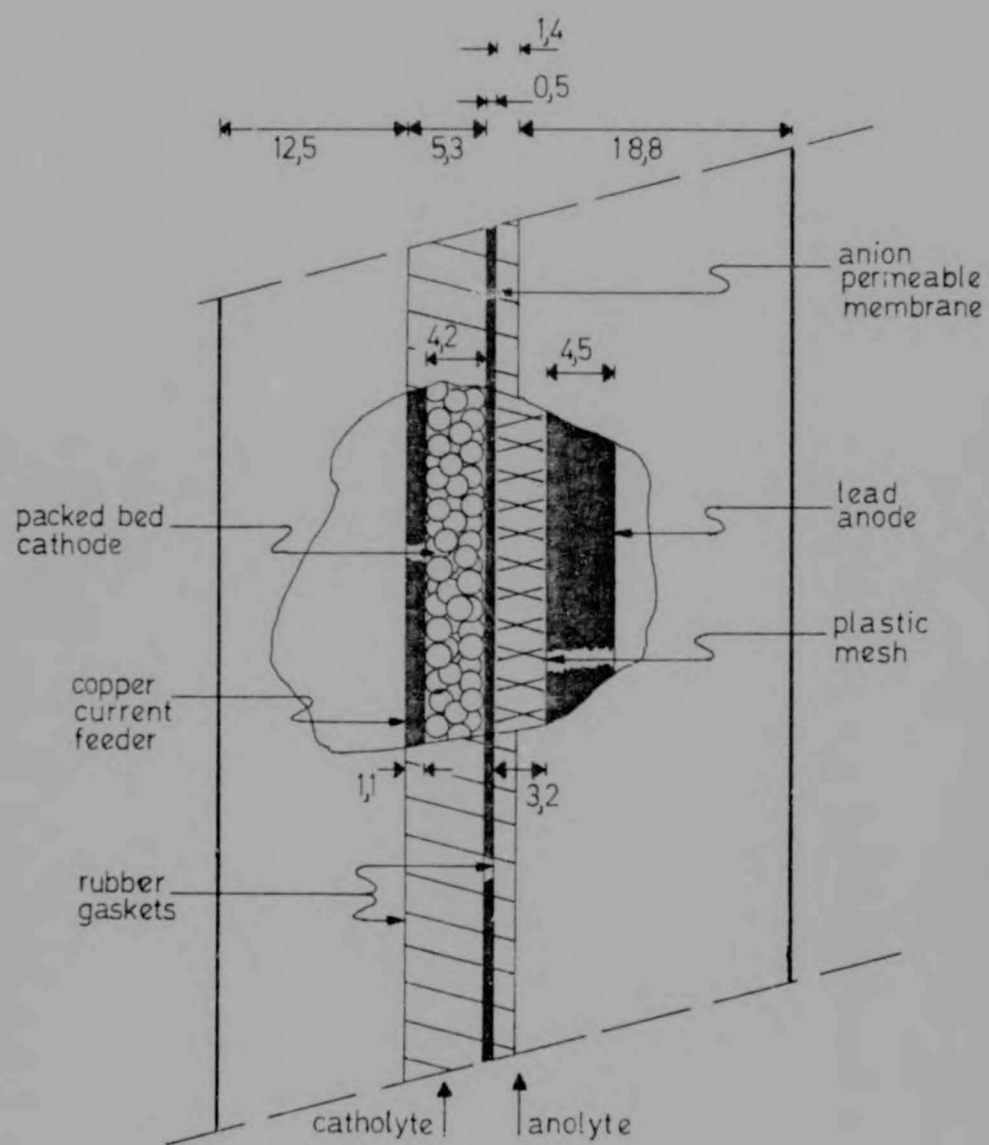


FIGURE 3.2 PACKED BED CELL FOR NICKEL EXPERIMENTS



dimensions in mm

FIGURE 3.3 ENLARGED SECTION OF THE NICKEL CELL

was calculated from the volume of water required to fill the cathode compartment (but excluding the entrance region) before the cell was packed: the bed voidage was then calculated from the volume required to fill the cathode compartment after packing. A micrometer was used to measure the diameter of a large number of particles and the average particle diameter was calculated from these measurements.

Considerable care was taken in the assembly of the cell to ensure that the packed bed was of uniform thickness throughout its entire length and breadth.

Table 3.1 Dimensions and details of the packed bed cathode used for nickel removal

Dimensions	Length : 100 cm Breadth : 20 cm Thickness : 0,42 cm
Current feeder area	2000 cm ²
Bed construction	Lead spheres - nickel plated Average diameter : 0,244 cm (standard deviation 0,015 cm) Bed voidage : 0,54

The anolyte was pumped into the base of the anode compartment, from which it flowed upwards and over a weir into a collecting box. The solution then returned by gravity through two outlet pipes to the anolyte tank.

The lead anode was of the same length and breadth as the cathode current feeder. Flow in the anode compartment was upwards through a plastic mesh support

which held the anion permeable membrane firmly at a constant distance from the anode surface itself.

Two Luggin probes were inserted to a depth of ten centimetres into the packed bed to measure the potentials of the electrode at the current feeder and at the separating membrane. The probes were connected to the reference electrodes by saturated potassium chloride salt bridges which passed through a hole cut in the lid of the cell. A stream of nitrogen was continually fed to the cathode compartment to prevent atmospheric oxygen dissolving in the catholyte: excess nitrogen, as well as hydrogen gas evolved at the cathode was vented through the same hole. Oxygen generated in the anode compartment was vented via the anolyte outlet pipes which were operated in a slug-flow mode.

3.2.3 Experimental Procedure

Before the start of an experimental run the anolyte and catholyte tanks were filled with distilled water which was circulated for an hour at the desired flowrates. The period of circulation served to raise the temperature of the catholyte to $25^{\circ}\text{C} \pm 1^{\circ}\text{C}$ which was $3 - 4^{\circ}\text{C}$ above the temperature of the constant temperature room in which the experiments were conducted. At the end of this period a small volume of nickel sulphate solution, containing sufficient nickel to produce a starting concentration of about 120 parts per million, was added to the catholyte. Sulphuric acid was then added to the catholyte and anolyte to produce a catholyte pH of 3,0 - 3,5 and an anolyte acid concentration of about 1,5 grams per litre. All reagents used were of analytical quality.

Preliminary experiments had shown that boric acid, used as a buffer in commercial nickel electroplating solutions, had no effect on experimental results when the plating solution was diluted down to wash-water effluent concentration and no boric acid was therefore added to the solutions used in the main experimental program.

At the end of a further ten minute period of circulation and mixing the power supply was connected to the cell and the experimental run started. The cell current and

the flowrates of the two circulating streams were kept constant throughout the run. At intervals 100 millilitre samples of the catholyte entering and leaving the cell were taken in polythene bottles. The catholyte conductance, bed potentials and cell voltage were measured at regular intervals and a check was kept on the temperatures of the circulating anolyte and catholyte. Anolyte samples were taken at the start and end of the run.

As the run progressed the nickel concentration of the catholyte was reduced. At the same time the pH of the catholyte rose as hydrogen ions were consumed in the competing reaction. Once pH8 was reached the current was switched off. If the nickel concentration, as indicated by the conductance of the solution, was still high, sufficient sulphuric acid was added to again produce a catholyte pH of 3,0 - 3,5 and, after a ten minute period of circulation and mixing, the power supply was reconnected and the run continued.

Once the nickel concentration had reached a sufficiently low level the run was ended. Sulphuric acid was added to the now depleted catholyte to produce a pH of 1,0 and this strongly acidic solution was circulated for ten minutes to wash the cathode. The anolyte and catholyte systems were then washed with at least three complete changes of distilled water to leave the cell ready for another run.

The pH of all the samples was measured immediately and the samples were allowed to stand for at least twenty four hours before being analysed for nickel. The nickel analyses were performed at 352,4 nanometres on a Varian Techtron atomic absorption spectrometer.

Each sample was analysed twice for nickel. The first analysis was performed on the sample as taken, and the second after the sample had been acidified with a few drops of sulphuric acid and then vigorously shaken. This procedure provided a check on whether any precipi-

tation of nickel hydroxide had occurred during the experimental run. Any hydroxide which had been produced settled while the samples were standing and had no effect on the first analysis; after acidification and agitation any hydroxide precipitate redissolved and a higher nickel concentration was measured in the second analysis.

3.2.4 Treatment of Results

A mass balance for nickel in the catholyte yields

$$\frac{dC_N}{dt} = \frac{58,71 \times 10^3 I \epsilon_N}{nFV} \quad [3.1]$$

where C_N is the nickel concentration (ppm) of the circulating catholyte, t is the time (secs), V is the volume of circulating catholyte (litres), I is the cell current (amps) and ϵ_N the current efficiency for nickel reduction. The assumptions inherent in the form of equation [3.1] are considered in section 5.5.

Provided the concentration change over a time interval is not too great the average current efficiency over the time interval may be approximated by

$$\epsilon_N = \frac{nFV}{58,71 \times 10^3 I} \frac{\Delta C_N}{\Delta t} \quad [3.2]$$

where ΔC_N is the change in nickel concentration over the interval Δt .

Equation [3.2] gives the result for the calculation of current efficiency from measured changes in the concentration of the bulk catholyte. Provided the concentration changes over the cell are not too great, it is reasonable to consider the current efficiency calculated in this manner to be the current efficiency determined at the average hydrogen ion and nickel concentrations existing within the cell itself at the time the measurements were made.

A pseudo steady-state mass balance for the nickel concentration difference over the cell yields

$$\Delta C_N = \frac{58,71 \times 10^3 \times 60 I_{\epsilon_N}}{n F Q} \quad [3.3]$$

Where $\Delta C_N = C_{N,IN} - C_{N,OUT}$ is the difference between the nickel concentrations (ppm) of the catholyte at the inlet and outlet of the cell and Q is the catholyte flow rate (l/min).

Hence the average nickel concentration in the cell may be calculated as

$$\bar{C}_N = C_{N,IN} - \frac{\Delta C_N}{2} \quad [3.4]$$

and the arithmetic mean hydrogen ion concentration may be calculated from the measured values at the inlet and outlet of the cell.

Equation [3.2] is therefore used to calculate current efficiencies from the experimental concentration versus time results. This current efficiency is considered as determined at the mean nickel concentration calculated using equation [3.4] and mean hydrogen ion concentration calculated from the catholyte pH measured at the inlet and outlet of the cell. Allowance is made in the calculations for volume changes after sampling.

3.2.5 Related Experimentation

Two other topics, namely an independent examination of the kinetics of the side reaction and the determination of the rate of diffusion of hydrogen ions through the cell membrane from anolyte to catholyte, were investigated by means of additional experiments. Brief summaries of the theory, experimental apparatus and procedure and of the experimental results are presented in Appendices D and E respectively.

3.3 Electrochemical Removal of Copper from Dilute Solution

3.3.1 Description of Apparatus and Packed Bed Electrolytic Cell

The apparatus and packed bed cell used by Young (11) in his investigation of copper removal from dilute solution are shown in Figure 3.4.

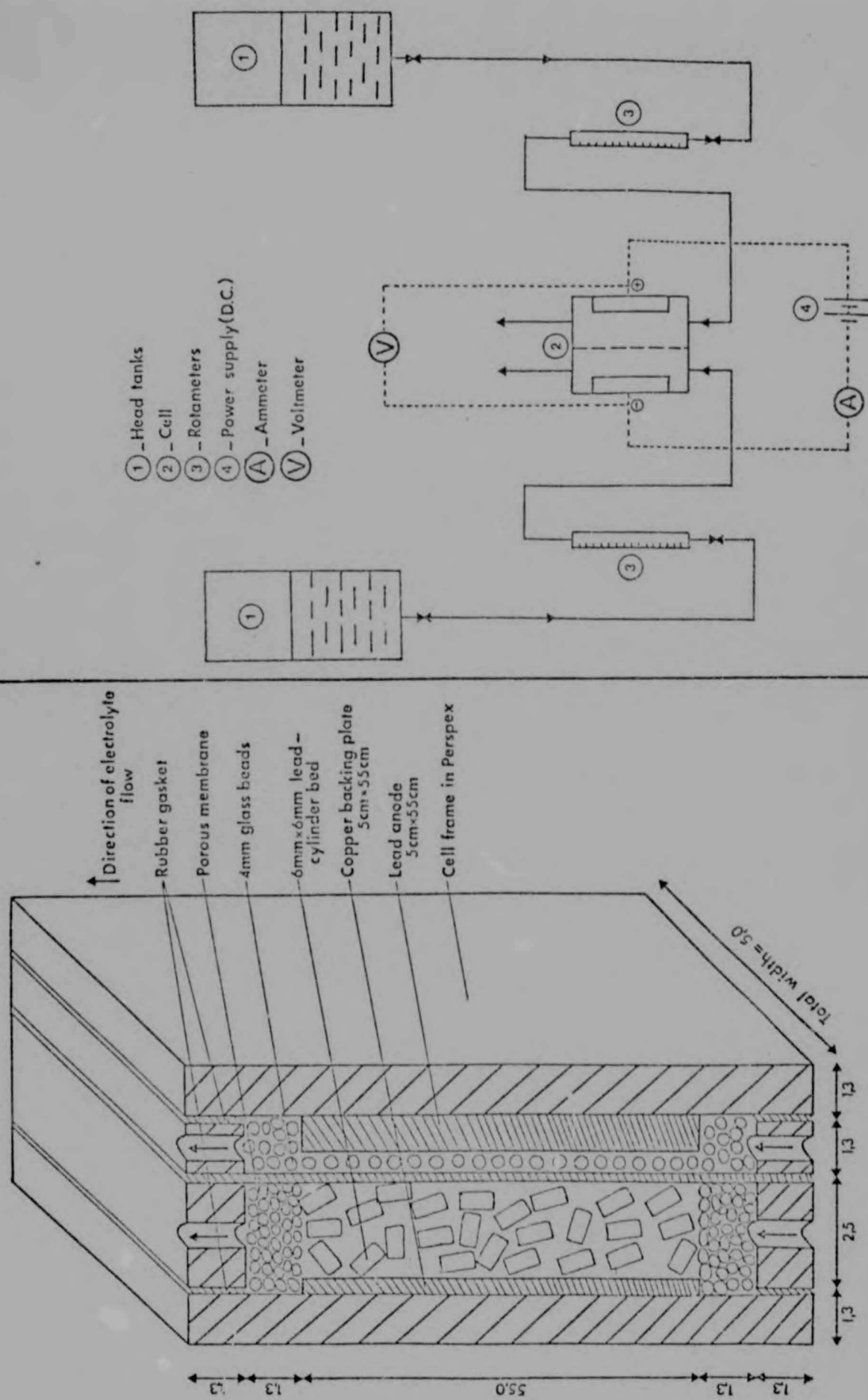
Two 225 litre head tanks were used to feed the anolyte and catholyte to four of the cells in series. Solution entered each cell at the bottom, passed out at the top and was discarded after leaving the fourth cell. Each cell was divided into two compartments by a one millimetre thick membrane of microporous rubber sheeting. Glass beads served as flow distributors at the inlet of each compartment and as a support between the lead anode and membrane. The cathode current feeder was a copper sheet and the cathode itself consisted of a bed of six millimetre by six millimetre lead cylinders. Other significant details are given in Table 3.2.

Table 3.2 Dimensions & details of the packed bed cathode used for copper removal

Dimensions	Four cells in series
	Length: 4 x 55 cm
	Breadth: 5 cm
	Thickness: 2,5 cm
Current feeder area	1100 cm ²
Bed construction	Lead cylinders: 6mm x 6mm
	Bed voidage : 0,407

3.3.2 Experimental Procedure and Treatment of Results

Fresh solutions - copper sulphate and sulphuric acid in tap water for the catholyte and sulphuric acid in tap water for the anolyte - were run through the apparatus which was operated at constant flowrate and current. Head and effluent



All dimensions in cm
Not drawn to scale

Schematic diagram showing the layout of apparatus.

FIGURE 3.4 APPARATUS USED TO INVESTIGATE COPPER REMOVAL

samples were taken at regular intervals and the cell voltage and pressure drop were regularly monitored.

A number of runs under identical conditions were performed on a bed which had initially been stripped free of copper. As a fresh copper surface built upon the bed the measured current efficiency increased to a steady value. An average value of the current efficiency was then taken from the final runs with consistent values. Once consistent values had been obtained the bed was stripped of copper and a new series of runs at different operating conditions was commenced.

The average current efficiency of the cell was calculated from a steady state mass balance analogous to that of equation [3.3]. Once again the calculated current efficiency was considered as determined at the mean of the measured inlet and effluent copper concentration - an assumption which is valid provided the change in concentration over the cell is not too great.

4. EXPERIMENTAL RESULTS

4.1 Investigation of Nickel Removal

4.1.1 Primary Experimental Program - Results obtained

A program of twenty experimental runs was conducted using the procedure detailed in Section 3. The range of experimental conditions investigated in the course of this program is detailed in Table 4.1 and detailed results for all runs are presented graphically in Appendix A.

Each set of results includes

- (a) the measured nickel concentration versus time curve,
- (b) the catholyte inlet and outlet pH versus time curves,
- (c) the current efficiency versus time curve calculated using equation [3.2],
- (d) the bed potential versus time curves measured at current feeder and anion permeable membrane, and
- (e) the catholyte specific conductivity versus time curve.

Dotted curves indicating the arithmetic average of the catholyte inlet and outlet hydrogen ion concentrations as well as the mean nickel concentration have been included wherever feasible.

As has previously been mentioned all samples were analysed twice for nickel in order to check whether precipitation of nickel hydroxide had taken place. In only two cases, namely in runs 2 and 7 was there a significant difference between

Table 4.1 Summary of experimental conditions in the investigation of nickel removal

Run No.	Cell Current (amps)	Catholyte Flowrate (litres/min)	Nickel Concentrations(ppm)	
			Initial	Final
1	1,0	3,0	108,5	3,2
2	1,0	1,0	109,6	4,4
3	1,0	3,0	98,0	2,0
4	1,0	2,0	98,4	3,9
5	1,0	8,0	94,7	3,6
6	1,0	1,0	95,4	5,6
7	10,0	8,0	107,8	4,9
8	5,0	8,0	111,8	2,2
9	7,5	8,0	131,0	4,2
10	15,0	3,0	110,6	3,2
11	7,5	3,0	154,1	7,1
12	15,0	3,0	308	23,4
13	10,0	3,0	308	9,7
14	5,0	3,0	103,3	5,0
15	15,0	5,0	115,6	2,0
16	10,0	5,0	109,1	1,9
17	7,5	5,0	107,4	3,5
18	5,0	5,0	107,2	2,0
19	7,5	1,0	106,4	21,1
20	5,0	1,0	107,9	18,9

the nickel concentrations measured before and after acidification of the samples. This difference, which amounted to 1,6 parts per million at the end of run 2 and 0,4 parts per million at the end of run 7, increased steadily from the apparent onset of precipitation at about pH7.5. In the case of runs 12-14 a slight turbidity was observed in the circulating catholyte at higher pH values but no measurable difference in the nickel analyses was obtained. There was therefore very little apparent precipitation of nickel hydroxide and even when precipitation did occur it was very limited in extent.

In the majority of runs the initial nickel concentration was about 110 parts per million and was generally reduced to a level of five parts per million or less. The concentrations listed in Table 4.1 are for the bulk catholyte and the concentrations of the stream leaving the cell were naturally even smaller.

4.1.2 Experimental Reproducibility

Two additional pairs of experimental runs were conducted under identical conditions in order to gauge the reproducibility of the experiments. From the results of these runs (shown in Figure 4.1) it is apparent that a high degree of reproducibility was attained. Such slight differences as do exist may be ascribed to the different pH versus time behaviour caused by slightly different rates of hydrogen ion diffusion and migration from the anolyte to the catholyte. These differences do not affect the mathematical modelling of the process as the effect of pH is taken into account by the model.

As the reproducibility of the experiments was satisfactory it was decided that none of the experiments in the primary

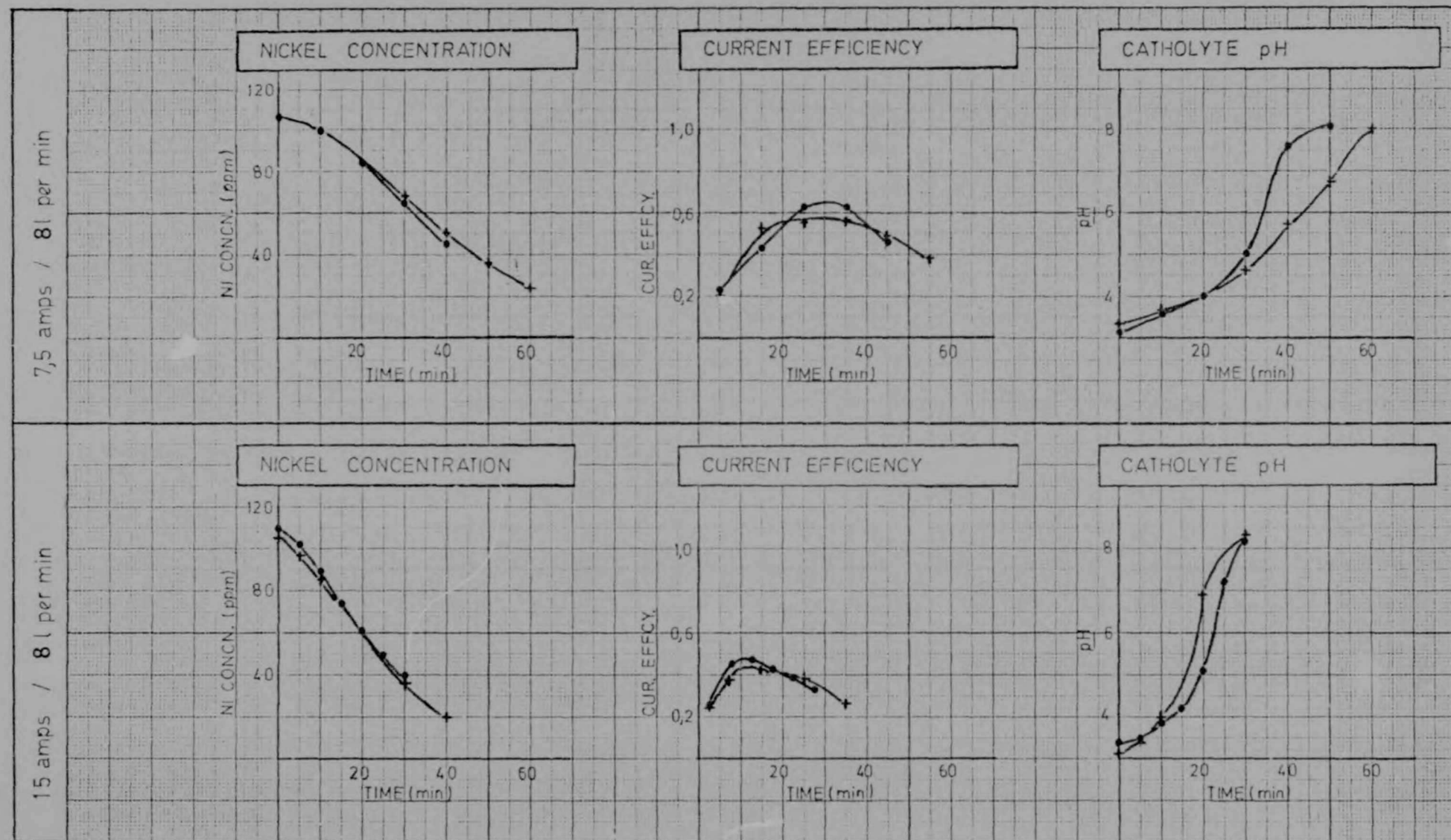


FIGURE 4.1. REPRODUCIBILITY OF NICKEL ION REMOVAL EXPERIMENTS.

program would be duplicated. A maximum of three runs, including all analyses and calculations, could be performed per week and this decision therefore considerably shortened the duration of the experimental program.

4.1.3 Catholyte Specific Conductivity

Before the mathematical model could be applied to the analysis of the experimental results the conductivity of the catholyte under the conditions of pH and nickel concentration existing within the interior of the packed bed cell had to be defined. The experimental conductivity results for the bulk catholyte were therefore used to develop a relation for specific conductivity as a function of pH and nickel concentration. The conductivity was found to be well described by the expression.

$$\kappa = (3,283 [\text{Ni}^{2+}] + 5,358 \times 10^5 [\text{H}^+] + 10,89) \times 10^{-6} \quad [4.1]$$

where κ is the specific conductivity (mho/cm); $[\text{Ni}^{2+}]$ (ppm) and $[\text{H}^+]$ (moles/litre) are the concentrations of these ions in solution

The fit of values calculated using equation [4.1] against over two hundred experimentally measured conductivity values is shown in Figure 4.2. Although at very low specific conductivities the calculated values do become slightly greater than the experimental values, too much significance should not be attached to this observation as the calibration curve of the conductivity cell was also most uncertain in this region.

The theoretical basis for the form of function chosen for fitting the experimental results is as follows.

The specific conductivity of the solution is given by

$$\kappa = F^2 \sum_i z_i^2 u_i c_i \quad [4.2]$$

Where u_i is the ionic mobility of species i ($\text{cm}^2\text{-mol/J sec}$), c_i is the ionic concentration (mol/cm^3) and κ is the specific conductivity (mho/cm).

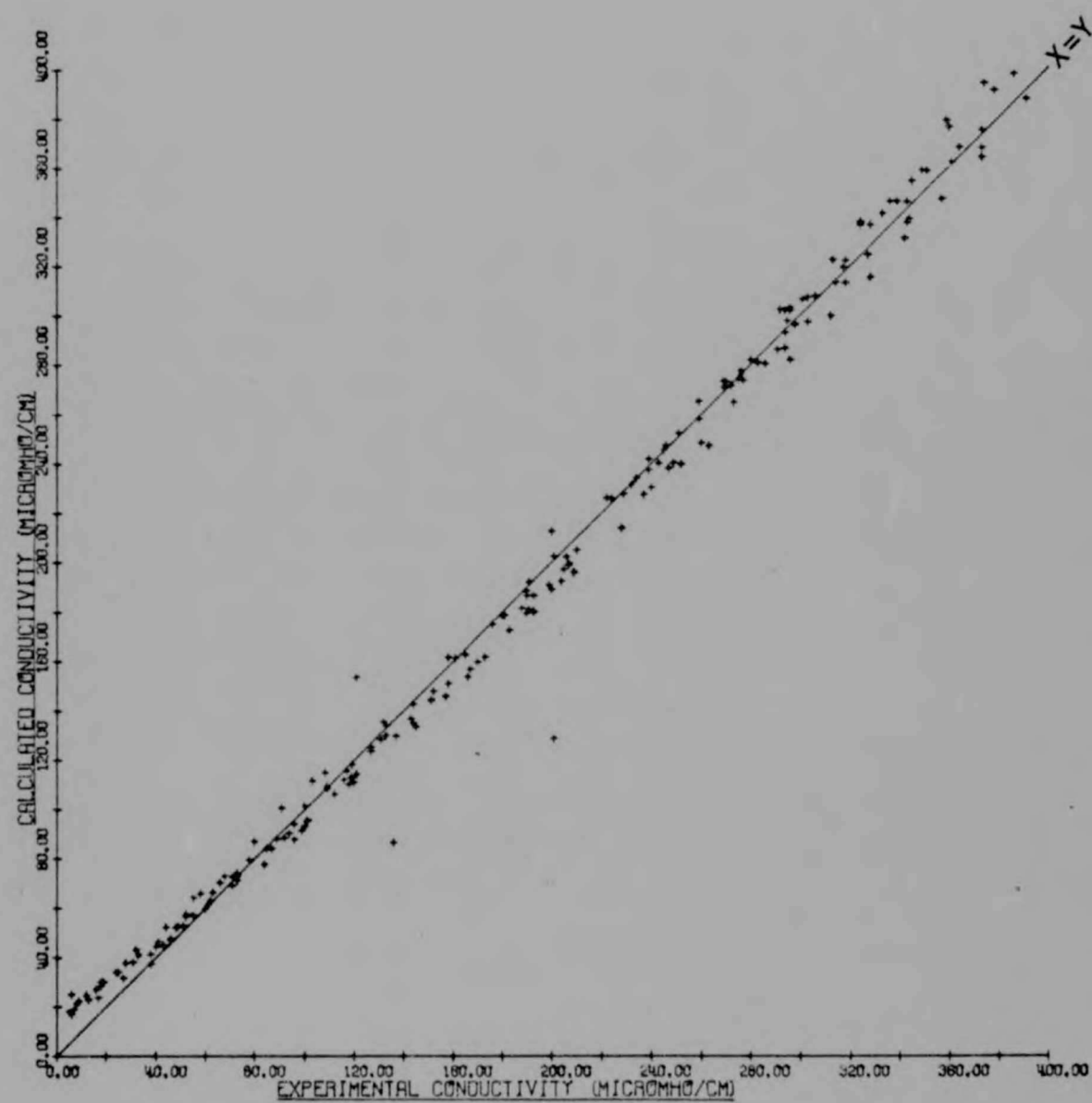


FIGURE 4.2. COMPARISON OF EXPERIMENTAL AND
CALCULATED SPECIFIC CONDUCTIVITIES.

Ionic mobilities are not generally reported in the literature; instead values of ionic equivalent conductances are given. These are related to ionic mobilities by

$$\lambda_i = |z_i| F^2 u_i \quad [4.3]$$

Where λ_i is the ionic equivalent conductance ($\text{mho-cm}^2/\text{eq}$), and the specific conductivity is therefore

$$\kappa = \sum_i |z_i| \lambda_i c_i \quad [4.4]$$

Ionic equivalent conductance values are dependent on concentration and are therefore usually reported at infinite dilution. Values for the ionic species present in the electrolyte used in the experiments are given in Table 4.2 (63).

Table 4.2 Values of limiting ionic equivalent conductances at infinite dilution in water at 25°C.

Ion	z_i	λ_i° ($\text{mho-cm}^2/\text{eq}$)
H^+	1	349,8
Ni^{++}	2	53
SO_4^{2-}	-2	80

Application of equation [4.4], without any correction for the concentration dependence of the ionic conductances, to a solution containing A moles per litre of NiSO_4 and B moles per litre of H_2SO_4 yields.

$$\kappa = 0,266A + 0,860B \quad [4.5]$$

which may be expressed in terms of the units of equation [4.1] as

$$\kappa = (4,5[\text{Ni}^{2+}] + 4,30 \times 10^5 [\text{H}^+]) \times 10^{-6} \quad [4.6]$$

and this is in fair agreement with equation [4.1].

4.1.4 Cell Voltage

Experimental results for the range of cell voltage are plotted in Figure 4.3. The lower curve applies to the start of experimental runs (when catholyte conductivities were relatively high) and the upper curve to the cell voltages measured towards the end of runs.

4.1.5 Diffusion of Hydrogen ions through the Anion Permeable Membrane

The results of batch diffusion experiments conducted along the lines indicated in Appendix E are plotted in Figure 4.4. The hydrogen ion diffusion coefficients have been calculated from the slopes of the straight line sections of the curves. The mean value of $3.62 \times 10^{-6} \text{ cm}^2/\text{sec}$ for diffusion through the membrane is approximately 1/25th of the typical value of $9.312 \times 10^{-5} \text{ cm}^2/\text{sec}$ reported in the literature (12,63) for the diffusion coefficient in aqueous solution at infinite dilution.

4.1.6 Investigation of Side Reaction Kinetics at a Rotating Disc Electrode

The results of an extensive series of rotating disc electrode polarisation experiments conducted on a blank electrolyte solution prepared in a similar manner to the solution used in the main investigation are reported and briefly analysed in Appendix D.

Experimental conditions are summarised in Table 4.3 together with the experimentally determined values of Tafel slope (b) and exchange current density $-i_{0S, \text{ ref}}$.

4.2 Investigation of Copper Removal

The results (11) of Young's investigation of copper removal from acidic solutions of copper sulphate are summarised in Figures 4.5 and 4.6. Further details are given in Table 4.4.

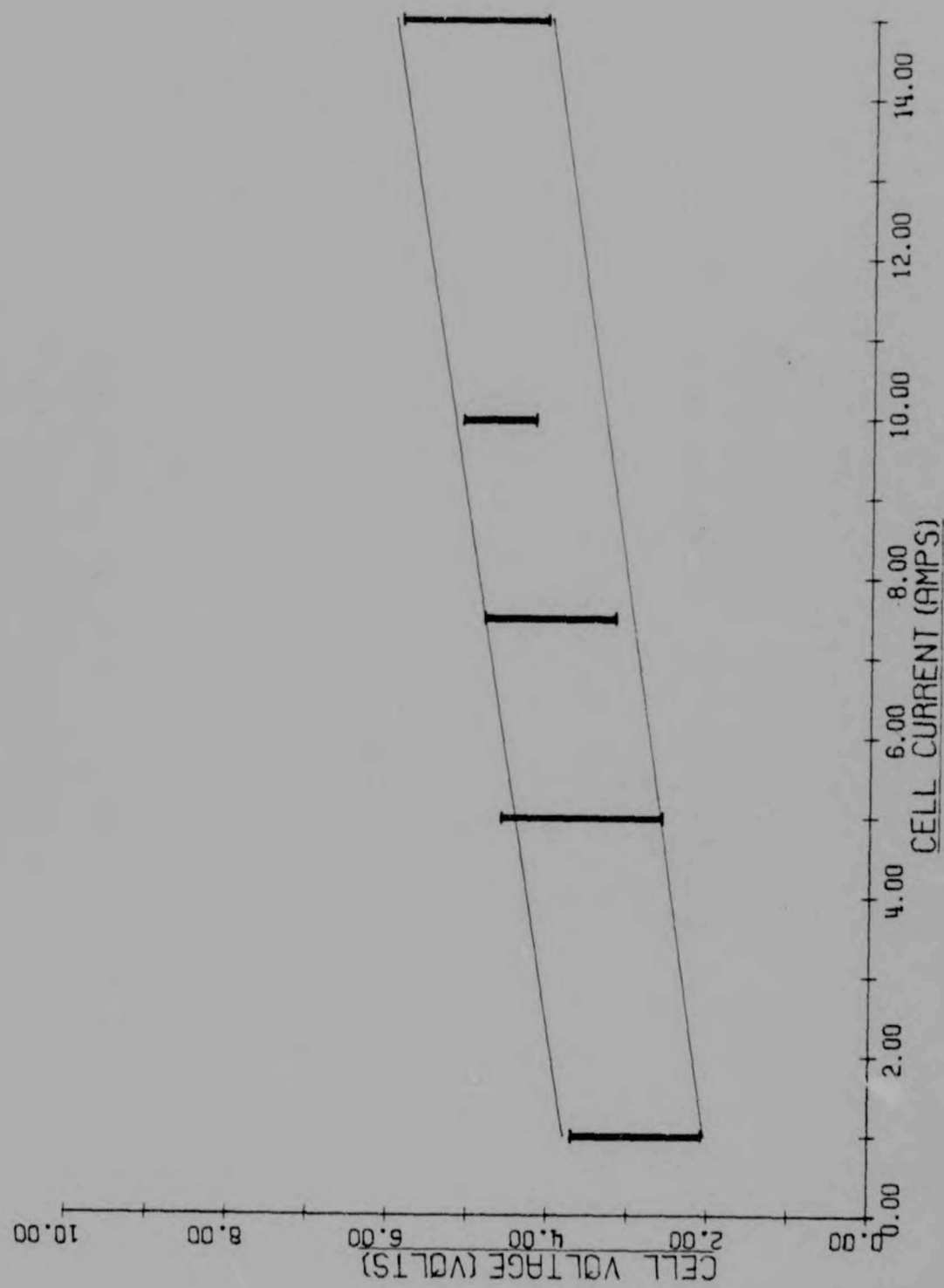


FIGURE 4.3. RANGE OF MEASURED CELL VOLTAGE FOR NICKEL EXPERIMENTS.

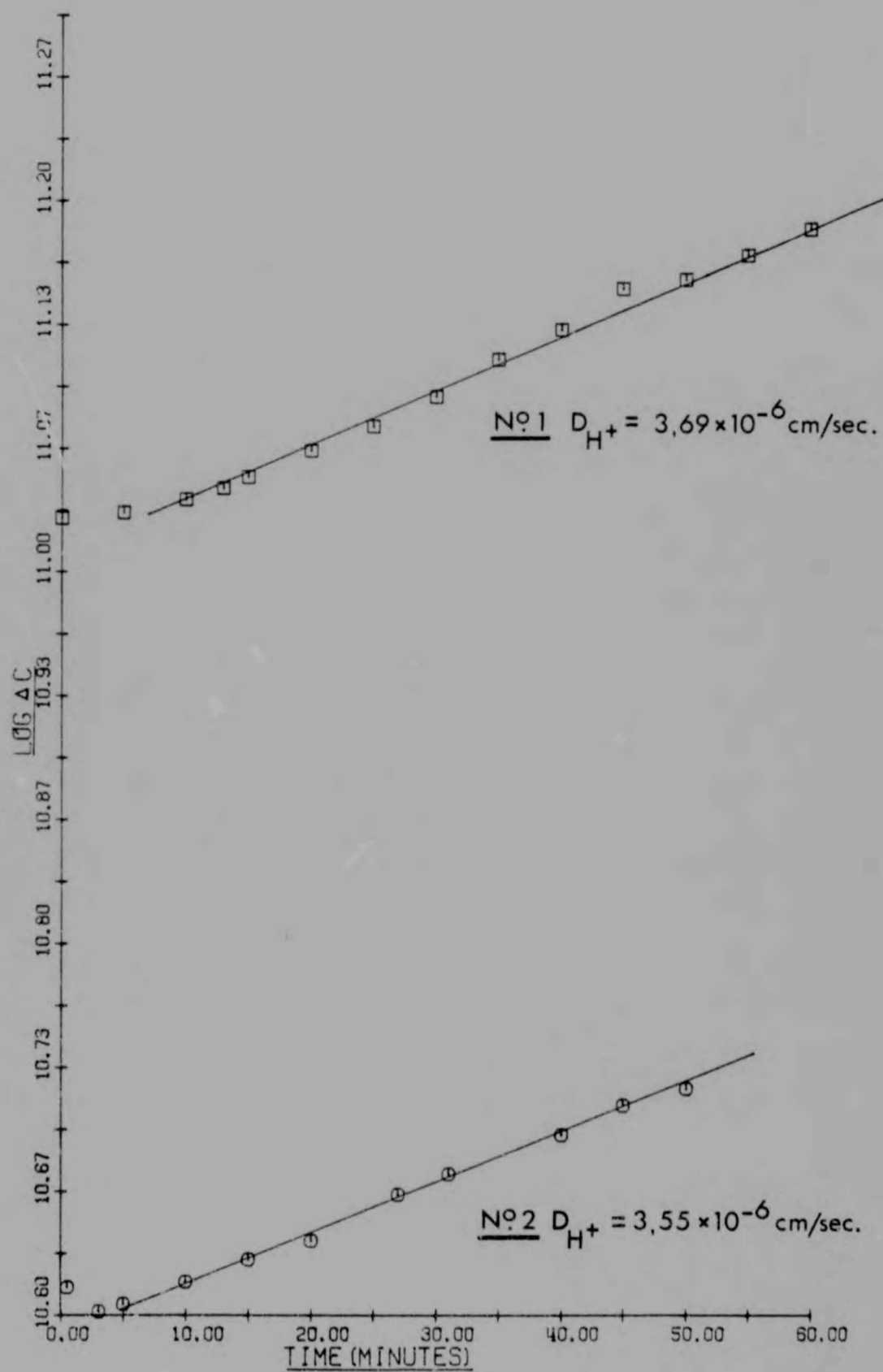


FIGURE 4.4. RESULTS OF BATCH DIFFUSION EXPERIMENTS.

Table 4.3 Summary of rotating disc electrode experimental conditions and experimentally determined electrochemical kinetic parameters

Run	pH	Speed (rpm)	Sweep rate (mV/S)	b (mV)	$-i_{o5,ref}$ (A/cm ²)	Potential Range (mV _{SCE})	Remarks
A1	3,0	500	10	545	$-1,46 \times 10^{-4}$	500-2000	
A2	3,0	2000	50	700	$-5,59 \times 10^{-4}$		
A3	3,0	4000	50	615	$-3,55 \times 10^{-4}$		
A4	3,0	500	50	670	$-4,02 \times 10^{-4}$		
A5	3,0	2000	20	530	$-1,46 \times 10^{-4}$		
B1	4,0	2000	50	335	$-5,17 \times 10^{-6}$	1000-2600	
B2	5,7	500	50	320	$-3,28 \times 10^{-6}$		
B3	5,8	500	100	320	$-3,28 \times 10^{-6}$		
B4	6,0	2000	100	320	$-3,37 \times 10^{-6}$		
B5	6,7	2000	100	320	$-3,37 \times 10^{-6}$		
B6	7,4	2000	100	320	$-3,28 \times 10^{-6}$		
B7	8,3	2000	1000	325	$-3,66 \times 10^{-6}$		
B8	8,7	2000	100	330	$-4,08 \times 10^{-6}$		
B9	3,7	2000	50	400	$-2,13 \times 10^{-5}$	500-2600	Effects of varying aeration
B10	3,7	2000	50	960	$-6,25 \times 10^{-4}$		
B11	3,7	500	50	1030	$-10,0 \times 10^{-4}$		
B12	3,7	2000	50	820	$-5,43 \times 10^{-4}$		
C1	4,1	2000	20	470	$-2,70 \times 10^{-5}$	500-2600	Electrolyte treated with activated C
C2	4,1	2000	20	370	$-6,20 \times 10^{-6}$	1000-2600	
C3	4,1	4000	20	380	$-7,88 \times 10^{-6}$	1000-2600	
D1	4,1	4000	20	545	$-7,31 \times 10^{-5}$	500-2600	Dull Ni

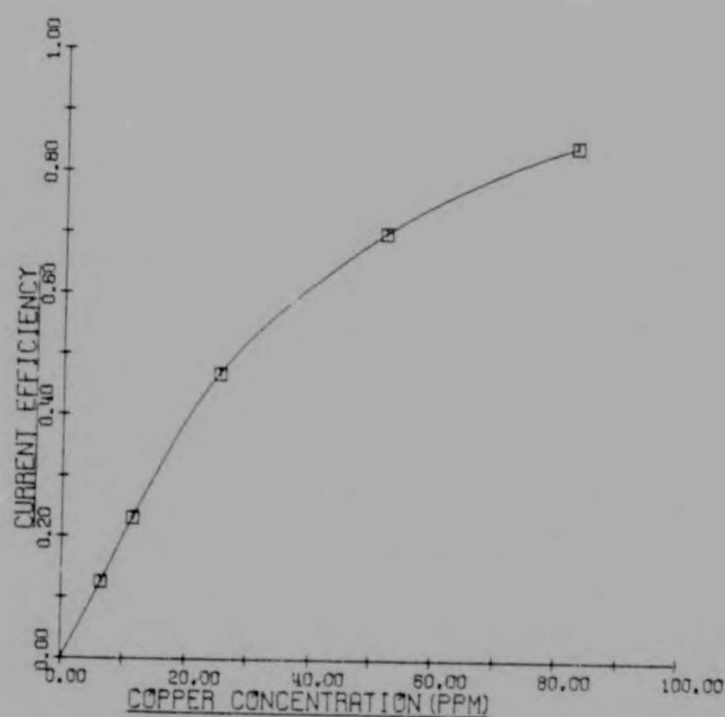


FIGURE 4.5. EFFECT OF CONCENTRATION ON COPPER REMOVAL EFFICIENCY.

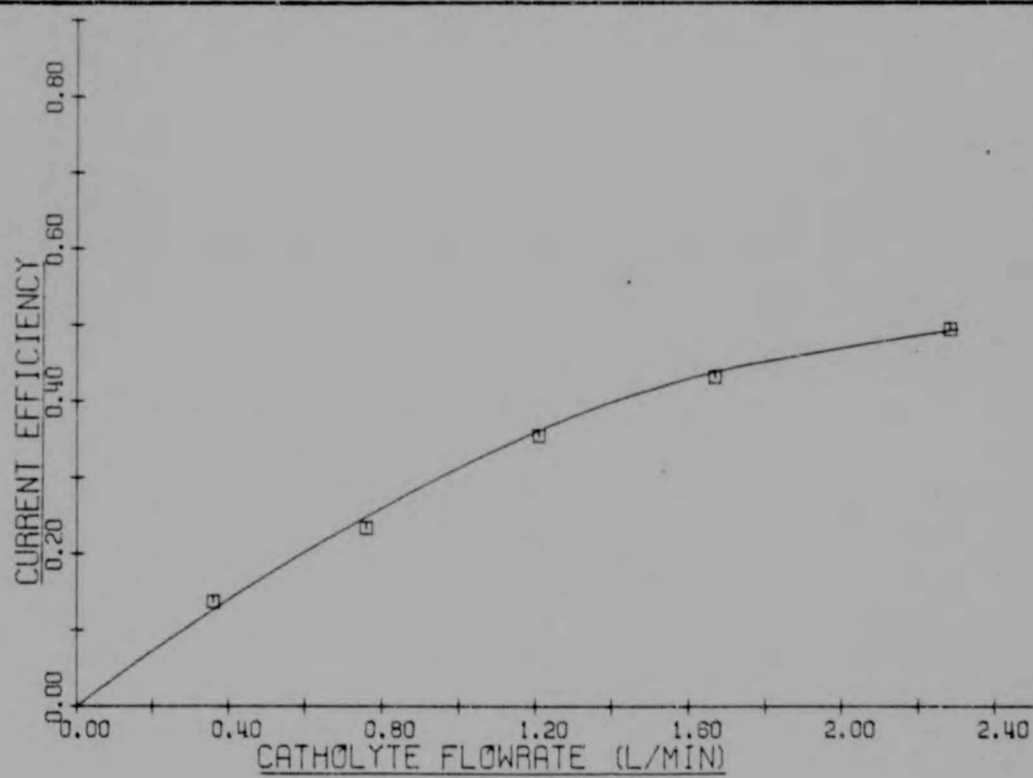


FIGURE 4.6. EFFECT OF FLOWRATE ON COPPER REMOVAL EFFICIENCY.

Author Dardis K A

Name of thesis Deposition of Copper and Nickel from dilute solutions in packed bed electrolytic cells 1981

PUBLISHER:

University of the Witwatersrand, Johannesburg

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