MASS TRANSFER IN LIQUID FLOW
THROUGH A RECTANGULAR CHANNEL
WITH AND WITHOUT THERMAL GRADIENTS

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MASS TRANSFER IN CHANNEL FLOW WITH THERMAL GRADIENTS
Mass Transfer in Liquid Flow through a Rectangular Channel with and without Thermal Gradients

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Dr. L.W. Shemilt

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To Ellen and Michelle
ABSTRACT

The limiting current method was used to investigate the mass transfer to a wall of a rectangular Plexiglas™ channel operated as a flow reactor for Reynolds numbers ranging from 200 to 32000. Cathode and anode blocks with smooth nickel surfaces were mounted in opposite walls of the channel, flush with the interior surface. Isothermal and non-isothermal mass transfer experiments were performed with the entire horizontal flow channel

i. rotated in 30° increments so that the cathode position varied from facing up to facing down (angle $\alpha$)

ii. tilted in 30° increments so that the channel position varied from horizontal to vertical (angle $\beta$)

at various bulk solution and cathode surface temperatures.

Isothermal experiments (3277 in total) were performed in a flow channel with an internal rectangular cross-section of 2 cm x 1.5 cm (width x height), the total length being 229 cm between inlet and outlet. The electrolyte bulk solution, comprised of 2 mol/l NaOH, 0.025 mol/l K$_3$Fe(CN)$_6$ and 0.025 mol/l K$_4$Fe(CN)$_6$, was varied in temperature from 25°C to 55°C.

While the bulk solution was maintained at 25°C and the cathode surface temperature varied from 35°C to 55°C, non-isothermal experiments (2394 in total) were conducted in the same apparatus as described above.

Examination of the data revealed that the mass transfer in the laminar flow regime, at $Re < 1900$, seems to be independent of the Reynolds number. For laminar flow conditions an equation of the form

$$Sh = 0.116 (1 + 0.336 (\sin \alpha)^{2.766} + 0.427 (\sin \beta)^{0.829}) (GR_m Sc)^{0.320}$$

gives an adequate prediction for mass transfer rates at $0^\circ \leq \alpha \leq 180^\circ$ and $0^\circ \leq \beta \leq 60^\circ$, where $GR_m$ is the
combined Grashof number for heat and mass transfer. Mass transfer rates for the channel in a vertical position ($\theta = 90^\circ$) can be predicted by:

$$Sh = 0.165 \, (GR_m \, Sc)^{0.42}$$

For Reynolds numbers greater than 1900, mass transfer was found to be controlled by free and forced convection, with the forced convection dominant. For the turbulent flow region the data for $0^\circ \leq \alpha \leq 180^\circ$ and $0^\circ \leq \beta \leq 60^\circ$ can be predicted by:

$$Sh = 0.283 \, (1 + 0.389 \, (\sin \alpha)^{1.03} + 0.318 \, (\sin \beta)^{1.79}) \, Re^{0.122} \, (GR_m \, Sc)^{0.04}$$

Mass transfer rates for channel slopes of $90^\circ$ were best correlated by:

$$Sh = 0.372 \, Re^{0.18} \, (GR_m \, Sc)^{0.08}$$

The isothermal laminar and turbulent experimental mass transfer coefficients were found to be within $\pm 10\%$ of correlations presented by Rouar et al. (1971) and Pickett (1977), respectively. For non-isothermal mass transfer in a horizontal channel under laminar and turbulent flow conditions the correlations of Ajersch (1990) agree well with the experimental data. Non-isothermal operation was found to increase mass transfer approximately four times over the isothermal case under the optimum enhancement conditions.

Since the system under investigation is a coupled one, no analytical solution of the governing equations is possible. The finite element solver FIDAP™ was used to obtain numerically the local mass transfer coefficients, local Reynolds numbers, local densities, stream functions and concentration and temperature profiles for isothermal and non-isothermal case studies. The numerically obtained average mass transfer coefficients agreed within 15% of the experimental values for both the isothermal and non-isothermal case.
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CHAPTER ONE
INTRODUCTION

Design, development and improved efficiency of such industrial electrochemical processes as the electrowinning, electrorefining, and electroplating of metals and the electrolysis of alkali solutions are closely concerned with the development and applications of electrochemical mass transfer phenomena, Faraday's laws, and the principles of electrolytic dissociation, conduction and polarization also are all prerequisites for the solution of problems encountered in the development and the operation of industrial electrochemical processes. One special application of polarization at an operating electrode is based on the constancy of current over a range of applied potential which corresponds to a limiting rate of a particular electrode reaction. This is known as the limiting current phenomena.

Engineering interest in operations involving reactions at liquid-solid interfaces has utilized the limiting current concept quite widely as a method of measurement of the rate of diffusion-controlled reactions. This method is known to be suitable to measure not only the space-time-averaged value and the local-smoothed value but also the local fluctuating value of mass transfer rates.

Measurements of mass transfer rates by the limiting current technique are being employed with increasing frequency. A number of reviews concerning the theoretical considerations, the experimental technique and the several hundreds of correlations for different operating conditions (i.e. free, forced and combined convection) have been presented (e.g. Fahidy, 1985; Pickett, 1977; Selman and Tobias, 1978; Mizushina, 1977). The theoretical principles and the experimental technique for the limiting current method are described in greater detail in Chapters 2 and 4, respectively.

The electrochemical method cannot be applied directly to heat transfer measurements. Nevertheless, many investigators of heat transfer have adopted the electrochemical technique since the knowledge of mass transfer obtained by this method can be correlated with heat transfer by the analogy
between both transport phenomena. Hence, the problem of mass transfer in electrolytic cells is closely related to problems of heat and mass transfer encountered in general chemical and mechanical engineering.

The electrochemical limiting current technique has been applied to a large number of systems including those for flow in pipes, channels or on walls. Systems in which the combined buoyancy forces of heat and mass transfer have significant influences on the momentum, heat and mass transfer, are often encountered. However only a small amount of analytical and experimental research on combined heat and mass transfer at liquid-solid interfaces has been published to date. Isothermal mass transfer correlations for channel flow are readily available in the literature, but none allow for the effects of heat transfer.

The purpose of this project is to examine mixed convection in laminar and turbulent boundary-layer flow in a rectangular flow reactor. Attention is focused on the study of the effects of the coupled thermal and mass diffusion along a solid surface rotated at different angles (α) and elevated from the horizontal at different slopes (β).

The relevant literature for both the experimental aspects and known correlations is presented in Chapter 3. In Chapter 4 and 5 the experimental details and results, respectively, of the present work are described. A theoretical analysis of the problem is provided in Chapter 6, with the conclusions from this project summarized in Chapter 7.
CHAPTER TWO

THEORETICAL PRINCIPLES

2.1 THE LIMITING CURRENT TECHNIQUE

A number of experimental techniques are available for the evaluation of mass transfer rates at solid surfaces. The main disadvantages of most of the methods is the interference by the measurement with the actual transport process. With the electrochemical limited current technique such interference is kept to a minimum. The limiting current method involves the measurement of the current flowing in an electrochemical cell operating under diffusion controlled conditions, with the measured current thus being directly proportional to the mass transfer rate.

Until the application of the limiting current technique, most mass transfer studies were performed by measuring the rate of thickness decrease of a surface dissolving in a fluid. In recent decades, the electrochemical technique of mass transfer measurement has become popular, partially because of its capability for rapid, precise measurement of transfer rates, but particularly because the transfer process causes no appreciable change in the surface. The basic principles of the technique are now well-known. Essentially, the method involves the measurement of the current flowing in an electrochemical cell, operating under such conditions that the reaction rate is controlled by the diffusion toward the electrode surface of a particular ion of known concentration. The advantages it holds over such a common method of measuring mass transfer rates as the dissolution of a coating on the wall of a duct are:

i. Precision: The dissolution method involves the measurement of very small changes in coating thickness, whereas the electrochemical method is a direct measure of the mass transfer rate, determinable with a precision ammeter.

ii. Speed: The cell current can be measured in a very short time, while the dissolution
method requires a new coating for each new measurement, and several minutes of operation for sufficient decreases in coating thickness.

iii. Versatility: Mass transfer rates corresponding to more complex geometries can be determined using the limiting current technique. Vogtlander and Bakker (1963) claim that the preparation of experiments for dissolution mass transfer experiments to be much more cumbersome than ones employing the limiting current technique (i.e. mass transfer to wires).

iv. Constant Surface Conditions: The dissolution of a coated surface causes changes in shape and roughness which will affect mass transfer to a greater or lesser degree. Limiting current conditions can be chosen so that the electrode surface will not undergo such changes.

The choice of the electrochemical system used for mass-transport studies is dependent on the following criteria:

i. Chemical stability.

ii. High solubility.

iii. Electrode potential sufficiently different from that of hydrogen or oxygen to give pronounced well-defined limiting current plateaus.

iv. Low cost.

The limiting current technique is based on the concept of a diffusion controlled electrochemical reaction of an ion or a molecule at an electrode / electrolyte interface. For the interpretation of the limiting current, physical properties of the electrolyte must be known accurately over a large range of compositions. Among the systems used, two in particular have earned a well-deserved popularity:

i. Deposition of copper from acidified copper sulphate solution

ii. Reduction of ferri- to ferrocyanide from solutions containing a large excess of NaOH or KOH.

For forced convection studies, the cathodic reaction of copper deposition has been largely
supplanted by the cathodic reduction of ferricyanide at a nickel or platinum surface (e.g. Grassmann et al., 1961; Reiss and Hanratty, 1962 and 1963; Reiss et al., 1963; Vogtlander and Bakker, 1963; Schütz, 1964; Dawson, 1972). Lin et al. (1951) and Ranz (1958), in studies of various possible electrolytes, had also concluded that the reaction

\[
\text{Fe(CN)}_6^{3-} + e^- \rightarrow \text{Fe(CN)}_6^{4+} \tag{2.1.1}
\]

in the presence of sodium hydroxide is one of the most suitable systems for mass transfer studies. An alkaline-supported equimolar mixture of ferri- and ferrocyanide is normally used. If the anolyte and catholyte are not separated by a diaphragm, oxidation of ferrocyanide at the anode compensates for the cathodic depletion of ferricyanide. Nickel, and to a lesser degree platinum, are the most commonly employed electrode materials in ferricyanide reduction. Both are sensitive to the presence of the cyanide ion, and exhibit 'poisoning' effects. The poisoning of the nickel surface is manifested in a sharp increase of electrode potential relative to the value in the active state. When ferrocyanide is exposed to light, it slowly hydrolyses:

\[
\text{Fe(CN)}_6^{4+} + \text{H}_2\text{O} \rightarrow \text{Fe(CN)}_5\text{H}_2\text{O}^{+} + \text{CN}^- \tag{2.1.2}
\]

\[
\text{CN}^- + \text{H}_2\text{O} \rightarrow \text{HCN} + \text{OH}^- \tag{2.1.3}
\]

This type of behaviour leads to errors, the observed limiting current being lower than if the entire surface were in the active state. Upon progressive poisoning under ostensibly identical conditions, the limiting current measurements from the same solution show a progressive decline. Because of the above reaction it has been strongly recommended that the electrolyte solution be protected from light (Selman and Tobias, 1978).
2.2 THE LIMITING CURRENT PLATEAU AND MASS TRANSFER

Electric current flow between an anode and cathode placed in an electrolyte is the combined result of electrode processes, charge transport and mass transport brought about by the imposition of an electric field across the electrodes. The relative amount of current due to the transport of each individual component depends on the strength of the electric field and the concentration of each electrolyte component. In Figure 2.2.1, a typical current voltage relationship is shown. For instance, when ferricyanide ions in an aqueous caustic soda solution are reduced at a nickel electrode by applying to the latter a negative potential, it is found that, upon increasing the applied potential slowly, the current first increases rapidly and then reaches a constant value, the so-called limiting current plateau. Following a further increase in potential the current rises sharply. This corresponds to the domain where the electrode reaction is no longer exclusively the reduction of the ferricyanide ion and hydrogen ions are also being discharged, resulting in the evolution of hydrogen gas.

The plateau or limiting current condition shown in Figure 2.2.1 is the point at which the reaction rate at the electrode is so fast that the concentration of the reacting ions on the surface is essentially zero. The magnitude of the limiting current, is then determined by the rate of arrival of the reacting ions at the electrode surface. Thus the mechanism involved in the solute ion transport within the electrolytic solution can be directly linked to the limiting current. For an electrochemical system such as the one described above, the ionic flux, \( N_i \), can be described in terms of three contributing mechanisms; diffusion down the concentration gradient, \( \nabla c_i \); ionic migration under the electrical field, \( \nabla \Phi \); and transport by convection (Shemilt, 1989; Fahidy and Mohanta, 1980):

\[
N_i = -D_i \nabla c_i - z_i u_i F c_i \nabla \Phi + c_i U \tag{2.2.1}
\]

The above flux equation (2.2.1) defines the diffusion coefficient, \( D_i \), and the mobility, \( u_i \).

The current is related to the flux through the charge per ion, \( z_i \), and Faraday's constant, \( F \):
FIGURE 22.1 Polarization Curves (25/25/0/0)

- $Sc = 2300$
- $T_B = 25^\circ C$

$\circ Re = 1130$
$\bullet Re = 4480$
$\cdot Re = 18200$
Because of electroneutrality, the convective term in equation (2.2.1) does not contribute to the current. Convection affects the current only indirectly through its influence on the boundary layer thickness and consequently on the concentration gradient. Furthermore, in most practical applications, non-reactive electrolytes are added to reduce the migration current of the reacting ion species by increasing the solution conductivity and thus making the contribution of migration negligible (Appendix I).

Consequently, the last two terms in equation (2.2.1) are dropped, establishing diffusion as the principle mechanism for transport of ions near the electrode:

\[ N_i = -D \left[ \frac{dc}{dy} \right]_{y=0} \]  \hspace{1cm} (2.2.3)

The negative sign indicates cathodic current, and \( y \) is the coordinate perpendicular to the electrode surface. Since electrode reactions involving only one species are considered, the subscript \( i \) can be omitted.

\[ N = \frac{i}{zF} = -D \left[ \frac{dc}{dy} \right]_{y=0} \]  \hspace{1cm} (2.2.4)

The passage of any current is associated with the corresponding concentration gradient - the higher the current, the steeper the gradient. Concentration profiles in systems involving convection are not linear, and the concentration tapers off gradually to its bulk value, \( C_0 \). It is convenient to define an equivalent, Nernst type stagnant diffusion layer of thickness \( \delta_e \) with a linear concentration profile such that the concentration gradient in equation (2.2.4) can be replaced by a linear difference (Fahidy, 1985):

\[ i = -zF D \left[ \frac{dc}{dy} \right] = -zF D \frac{C_e - C_0}{\delta_e} \]  \hspace{1cm} (2.2.5)

As the potential is further increased, ions are consumed faster by the electrode reaction, resulting
in a lower concentration \( C_a \) at the electrode surface. Since \( \delta_e \), the boundary layer thickness, is determined by the flow, and the bulk concentration is fixed, this decrease in \( C_a \) brings about a higher current. Because the concentration at the electrode cannot be less than zero, there is a limit which the current cannot exceed. This mass transport limiting current is determined by setting \( C_o = 0 \) in the above equation (2.2.5).

\[
i_L = -\frac{z_i F D C_b}{\delta_e}
\]  

(2.2.6)

The limiting current is an important design parameter since it presents the maximum rate at which an electrode reaction can proceed. Moreover, it provides a convenient technique for transport rate studies, since mass transport coefficients can thus be readily and accurately calculated from the experimentally obtained current plateau. The mass transport coefficient \( K \) is defined by:

\[
N_r = K(C_b - C_a)
\]  

(2.2.7)

Representing the flux in terms of the current (Fahidy, 1985; Wragg, 1977):

\[
K = \frac{i}{z_i F (C_b - C_a)}
\]  

(2.2.8)

The only parameter in the above equation which can not be readily measured is \( C_o \). However, at the limiting current condition \( C_o \) is essentially zero \( (C_o = 0) \). Hence (Pickett, 1977):

\[
K = \frac{i_L}{z_i F C_b}
\]  

(2.2.9)

Mass transport coefficients are often represented as dimensionless Sherwood or Stanton numbers. These can be expressed in electrochemical systems in terms of the limiting current, respectively:
\[ Sh = \frac{Kd_e}{D} = \frac{i_L}{zFDC_B} \]  
(2.2.10)

\[ St = \frac{Sh}{ReSc} = \frac{i_L}{zFUC_B} \]  
(2.2.11)

where

\[ Re = \frac{ Ud_e }{ v } \]  
(2.2.12)

and

\[ Sc = \frac{ v }{ D } \]  
(2.2.13)

2.3 LIMITING CURRENT AND POLARIZATION

An electrode immersed in an electrolyte solution develops an equilibrium electrode potential through the tendency of metal, M, to ionize and go into solution and vice versa (King, 1981).

\[ M \leftrightarrow M^\nu + ze^- \]  
(2.3.1)

This process results in the formation of a layer of ions adjacent to the electrode, and of opposite charge to the electrode. This is the so-called double layer model, first proposed by Helmholtz. It has undergone developments to include solvent effects. In order to measure electrode potentials, a second electrode of defined electrode potential, known as a reference electrode, is required. Descriptions of reference electrodes and their function are widely available (e.g. Pickett, 1977; Mizushina, 1971).

A perfect thermodynamic reversible electrode process assumes that the electrode potential would
not deviate from its equilibrium value while a current is passed through an electrochemical reactor. These 'ideal' electrodes are referred to as 'non-polarisable'. Electrodes are said to be polarised, when there is a deviation from the equilibrium potential. The magnitude of polarization depends on the local current density, the nature of the electrode process, and the composition of the electrolyte.

The chemical reactions which take place in an electrolytic cell by way of electron transfer exhibit energy changes via current flow in an external circuit. The maximum work available from a cell under thermodynamic equilibrium is given by:

\[ \Delta G = -z_e E_r F \]  \hspace{1cm} (2.3.2)

where \( \Delta G \) is the free energy change, \( z_e \) the number of electrons in the cell reaction, \( E_r \) is the reversible equilibrium cell voltage and \( F \) the Faraday constant. Using the van't Hoff isotherm and equation (2.3.2), the well known Nernst equation is obtained (King, 1981):

\[ E_r = E_r^* + \frac{R T}{z_e F} \ln \left( \frac{a_+}{a_-} \right) \]  \hspace{1cm} (2.3.3)

where \( E_r^* \) is the electrode potential under standard conditions, \( R \) is the gas constant, \( T \) the absolute temperature and \( a_+ \) and \( a_- \) are the activities of the oxidized and reduced species respectively. The subscripts - and + will be used for cathodic and anodic process terms respectively. Equation (2.3.3) describes the electrode reaction under equilibrium conditions with no net reaction. In actual processes with reactions proceeding and current flowing, irreversibilities are inherent. The overall potential required is thus greater than the equilibrium (or thermodynamic) potential.

To drive an electrode reaction either anodically or cathodically, a voltage \( E \) is applied to \( E_r \) of equation (2.3.3), the difference between the two being known as the overpotential, \( \eta \).

\[ \eta = E - E_r \]  \hspace{1cm} (2.3.4)
This overpotential in an electrochemical cell involves the concentration overpotential, \( \eta_c \), the ohmic overpotential, \( \eta_o \), and the activation overpotential \( \eta_a \). The net overpotential is therefore described by:

\[
\eta = \eta_c + \eta_o + \eta_a
\]  
(2.3.5)

2.3.1 The Concentration Overpotential

Concentration overpotential is the overpotential caused by the difference between the bulk concentration and the electrode surface concentration of the active ion. In a cathodic process, e.g.,

\[
\eta_c = \frac{R \cdot T}{z \cdot F} \ln \left( \frac{C_o}{C_s} \right)
\]  
(2.3.1.1)

This overpotential is usually described as the 'Nernst potential'. Combining the above equation (2.3.1.1) with equations (2.2.5) and (2.2.6) gives the logarithmic expression for the concentration overpotential and the limiting current:

\[
\eta_c = \frac{R \cdot T}{z \cdot F} \ln \left( \frac{1-i}{i_L} \right)
\]  
(2.3.1.2)

It is evident from equation (2.3.1.2) that as the current increases, the cathodic overpotential becomes more negative. As the limiting current is reached, the polarization tends towards \(-\infty\), and the limiting current plateau appears. Theoretically, this current would remain constant were it not for other electrode reductions taking place at more negative potentials, e.g., the reduction of hydrogen ions to hydrogen.

Electrode potentials and overpotentials for working electrodes are commonly measured relative to the solution. This explains the convention of defining the concentration overpotential at the working electrode as the potential of a reference electrode near its surface relative to the bulk solution (after interruption of the current). Consequently, the overpotential at an anode will generally be positive, and
that at a cathode will be negative. In processes operated under non-limiting current conditions, the concentration will vary along the electrode surface, and as a result, the concentration overpotential will also depend on the position along the surface.

2.3.2 The Ohmic Overpotential

In practical electrochemical reactors the ohmic drop $IR_e$ may present a significant portion of the overall voltage drop, especially if a large amount of Joule heat is needed to maintain the electrolyte temperature within a specified range (Fahidy, 1985). Membranes and separators in electrochemical cells also contribute to the overall ohmic drop. The ohmic potential is closely associated with the $IR_e$ drop. It depends on the resistivity of the electrolyte and the distance between the working electrode and reference electrode. The ohmic overpotential does not affect the shape of the limiting current plateau; it merely shifts it to more negative values. In practical applications with a large amount of supporting electrolyte the ohmic overpotential can be assumed to be negligible (Landau, 1981). Reliable theoretical estimations of the ohmic overpotential are essentially restricted to very dilute solutions. For single electrolyte of concentrations greater than 0.01 mol/L, the ohmic overpotential is best determined experimentally and its dependence on composition and temperature expressed in terms of appropriate statistical regression equations (Fahidy, 1985).

2.3.3 The Activation Overpotential

An additional contribution to the overall cell potential is the driving force required to make the electrode reactions proceed at appreciable rates. The surface, or activation overpotential involves the passage of ions at the electrode across the electric double layer, their discharge, and changes in surface conditions. The rate of the electrode reactions is measured by the current density at the electrode. By convention, the current density is positive when it flows from the electrode into the solution. Thus, current densities are positive at anodes and negative at cathodes. The current density depends on the driving force and thus is related to the surface overpotential and the composition of the solution at the interface, as well
as the temperature.

It has been found experimentally that, for a great many reactions taking place with an appreciable current density, the cathodic overpotential can than be expressed by an empirical equation:

$$\eta_c = a_c + b_c \log(i) \quad (2.3.3.1)$$

which is called the Tafel equation, $a_c$ and $b_c$ being known as Tafel 'constants'. A Tafel equation is equally appropriate for data from anodic reactions including oxygen evolution. The same form as equation (2.3.3.1) is adopted but quoted values of $a_c$ and $b_c$ have opposite signs to those for cathodic reactions. However, this convention refers to a reversible reaction at a single electrode and is irrelevant for anodic reaction in an electrochemical reactor (Pickett, 1977). In principle the Tafel 'constants' can be determined from a plot of overpotential versus the logarithm of the current density.

The overpotential depends mostly on the kinetic aspects of the electrode reaction. At the limiting current region, although $\eta_c$ dominates, $\eta_e$ increases with current as well. If both are of the same order of magnitude at limiting current conditions, then the limiting current may be obscured. Large surface overpotentials tend to narrow the limiting current plateau region. This effect was shown by Selman and Tobias (1978) to be more dominant in electrochemical system involving copper deposition reactions than for the ferri-ferrocyanide redox system. Surface polarizations may also pose a hazard when the counter electrode (anode) is also used as the reference electrode. The common method to overcome this problem as adopted in this work with an electrolytic system with equimolar amounts of potassium ferri and ferrocyanide is to increase the surface area of the counter electrode (anode) with respect to the working electrode (cathode). Usually the ratio of the anode surface area to the cathode surface area will be kept greater than or equal to two.
2.4 DIMENSIONLESS VARIABLES

The similarity principle is very important as far as sciences of theoretical and experimental fluid mechanics and heat and mass transfer are concerned. First the dimensionless coefficients are independent of the system units. Secondly, the use of the similarity principle can lead to a considerable simplification in the extent of experimental work. The dimensionless groups are also used in heat and mass transfer studies for equipment scale-up and design. It is useful to think of the dimensionless groups as ratios of various forces or effects in the system. Four types of similarities are most important in engineering studies, namely:

i. Geometric similarity (dimensional proportionality)

ii. Mechanical similarity
   a. Static similarity (deformation proportionality)
   b. Kinematic similarity (time proportionality)
   c. Dynamic similarity (force proportionality)

iii. Thermal similarity (temperature proportionality)

iv. Chemical similarity (Concentration proportionality)

The selection of dimensions and operating conditions so as to satisfy geometric, thermal, and chemical similarity requirements requires familiarity with commonly used dimensionless groups and their significance (Fluid Dynamics International, 1990). Following the traditional practice in heat and mass transport the use of dimensionless groups will be followed throughout this work. The principal dimensionless parameters that appear in the problem under investigation are outlined below.

The Reynolds number, Re, (equation 2.4.1) is the ratio of inertia forces to viscous forces. Hence a low Reynolds number means that viscous forces are large in comparison with the inertial forces. The Reynolds number measures transition from laminar to turbulent flow; for internal flows the transition Reynolds number is in the order \(O\) of \(10^5\), while for external flows it is \(O(10^6)\). The Reynolds number is also used in order to describe boundary layer thickness; a typical wall boundary layer thickness
\[ \delta_e \propto \text{Re}^{-0.5}d_e, \text{ where } d_e \text{ is a characteristic length scale.} \]

\[ \text{Re} = \frac{Ud_e}{v} \quad (2.4.1) \]

The Prandtl number, Pr, (equation 2.4.2) is the ratio of diffusivity of momentum to diffusivity of heat. For gases and liquids such as water, Pr has values in the range of 0.1 to 1; for liquid metals, Pr is typically 0.001 to 0.01, while for oils Pr is large, in the range form 1 to 1000 and may be even larger for plastic melts. When Pr < 1, the thermal boundary layer is thicker than the hydrodynamic boundary layer, and develops faster; and conversely, when Pr > 1.

\[ Pr = \frac{v}{\alpha_i} \quad (2.4.2) \]

The Schmidt number, Sc, (equation 2.4.3) is the ratio of diffusivity of momentum to diffusivity of mass. For most common liquids Sc is two or more orders of magnitude greater than Pr; for gases however, Pr and Sc are typically of the same order of magnitude.

\[ Sc = \frac{v}{\alpha_c} \quad (2.4.3) \]

The Peclet number, Pe, is the analog of the Reynolds number for convection of heat (equation 2.4.4) and mass (equation 2.4.5). It is the ratio of heat (mass) transfer by convection to heat (mass) transfer by conduction (diffusion). It is important in forced convection problems and measures the thickness of the thermal (mass) boundary layer (\( \delta_e \propto \text{Pe}^{-0.5}d_e \)).

\[ Pe = \text{RePr} = \frac{Ud_e}{\alpha_i} \quad (2.4.4) \]
\[ Pe_c = Re Sc = \frac{U d_e}{\alpha_e} \]  
(2.4.5)

The Rayleigh number, \( Ra_c \), is the ratio of the energy generated by buoyancy forces to the energy dissipated by diffusion and viscosity. It is important in natural convection problems. A typical natural convection boundary layer has a thickness, \( \delta \), which is proportional to \( Ra^{-0.25} d_e \). The buoyancy effects are measured by the thermal (equation 2.4.6) and solutal (equation 2.4.7) Rayleigh numbers:

\[ Ra_c = Gr_c Pr = \frac{g \beta c \Delta T L^3}{\nu \alpha_c} \]  
(2.4.6)

\[ Ra_e = Gr_e Sc = \frac{g \beta e \Delta C L^3}{\nu \alpha e} \]  
(2.4.7)

The Grashof number is the ratio of buoyancy forces to viscous forces. In natural convection driven flow, transition from laminar to turbulent flows occurs when \( Gr \) is \( O(10^4) \). The Grashof numbers for temperature (\( Gr_t \)) and concentration (\( Gr_c \)) are defined by:

\[ Gr_t = \frac{Ra_t}{Pr} = \frac{g \beta c \Delta T L^3}{\nu^2} \]  
(2.4.8)

\[ Gr_c = \frac{Ra_c}{Sc} = \frac{g \beta e \Delta C L^3}{\nu^2} \]  
(2.4.9)

respectively.

The Sherwood number, \( Sh \), is the mass transfer analog to the Nusselt number of heat transfer. In order to express the relationship for the limiting current density in a form more suitable for scale-up
and design, the Sherwood number can be written as:

\[ Sh = \frac{t_c d_r}{z_F D C_a} \]  \hspace{1cm} (2.4.10)

The Lewis number, \( Le \), (equation 2.4.11) is the ratio of mass diffusivity to thermal diffusivity.

It is typically of order \( 10^2 \) for more common liquids and in the order of one for gases.

\[ Le = \frac{\alpha_c}{\alpha_t} \] \hspace{1cm} (2.4.11)
CHAPTER THREE
LITERATURE REVIEW

In view of the desirability of increasing the speed of production by using higher current densities, mass transfer under forced convection in electrolytic processes is of considerable potential interest. In most practical systems, vertical electrodes are used. Mass transfer due to free convection also occurs, making the total flow pattern very complicated. This brief literature review is thus organized in five sections. The first topic, laminar channel flow, contains a summary of relevant literature in the field of laminar mass transport phenomena. The issue of turbulent mass transport will be reviewed in the second section. The effects of buoyancy forces in forced and combined free and forced convective flow over planar electrodes have been studied extensively in the past. A brief review concerning these phenomena will be presented in the third part. Very little literature exists concerning the effects of combined heat- and mass transfer in comparable systems, but some publications have been located which will be reviewed in the fourth part. In the last section of the literature review, a number of publications involving estimation of the electrolyte solution properties will be discussed.

3.1 EXPERIMENTAL AND THEORETICAL ANALYSIS OF LAMINAR CHANNEL FLOW

In an ideal channel flow reactor, the two parallel electrodes comprising the two opposite walls of the channel are infinitely wide, but are spaced a finite distance apart from each other. For such a system, it is possible to derive an analytical expression for mass transfer under laminar flow conditions. In his work on heat transfer coefficients for laminar flow past a flat plate, Lévêque (1928) presented a theoretical solution where the local velocity profile near the electrode surface was assumed to be linear. This well-known "Lévêque approximation" has also been applied to solving mass transfer equations for
various geometries. The Lévêque solution for a rectangular duct was first presented by Norris and Streid (1940) in their study of laminar flow average heat transfer coefficients. Often referred to as the "Lévêque equation", it is presented in terms of mass transfer as follows:

\[
Sh = 1.849 \left( \frac{Re \cdot Sc \cdot d_e}{L} \right)^{1/6}
\]  

(3.1.1)

This equation is based on the assumption that the concentration of the ionic species is maintained constant away from the vicinity of the channel wall. This agrees well with the results of Tobias and Hickman (1965) who studied experimentally the effects of buoyancy forces on ionic mass transfer at horizontal planar electrodes with both laminar and turbulent bulk flow. Copper electrodes and an acidified copper sulphate electrolyte were used in a rectangular channel with an aspect ratio, \( \gamma \) (\( \gamma = \text{height/width} \)), of 10-15 to obtain local and average mass transfer data by the limiting current technique in the ranges of parameters: \( 10^3 < \text{Gr}_e < 10^{12} \), \( 75 < \text{Re} < 7000 \), \( 600 < \text{Sc} < 12000 \) and \( 0.05 < \frac{(d_e)}{L} < 20 \).

For a smaller aspect ratio, the coefficient in the above equation should be multiplied by a factor \( \chi \):

\[
Sh = 1.849 \chi \left( \frac{Re \cdot Sc \cdot d_e}{L} \right)^{1/6}
\]  

(3.1.2)

Furthermore it was concluded that when the bulk flow is turbulent (\( Re > 4000 \)), the effect of free convection on mass transport appears to be negligible within the experimental conditions studied.

Roušar et al. (1971) derived equations for calculating the local and average limiting current densities for an electrode located on the wall of a rectangular channel. The validity of the equation for the average limiting current density was verified experimentally by the deposition of Hg from an alkaline Hg\(^{2+}\) electrolyte solution, whereas the equation expressing the local limiting current density as a function of the length and width of the electrode was confirmed by the deposition of Ag. For mass transfer in a rectangular channel with different aspect ratios, where the electrodes occupy only a part of the rectangular channel width, the values of \( \chi \) were presented in tabular form as a function of the aspect ratio. In a similar
approach. Ong (1972) has obtained data with electrodes of various lengths (2 mm to 149 mm) in a small rectangular electrochemical flow reactor having \( x_n = 95 \) mm, \( d_e = 11.9 \) mm and \( \gamma = 0.75 \). The electrolyte consisted of 0.01 mol/l to 0.47 mol/l m-nitrobenzene sulphonic acid (sodium salt), 1 mol/l sulphuric acid and distilled water. Ong found that the overall mass transfer coefficient could be closely approximated by the relationship:

\[
Sh = 0.807(f(\text{height, width})\text{Re Sc} \frac{d_e}{L})^{1/3} \tag{3.1.3}
\]

A more rudimentary approximation for a finite reactor has been provided by Pickett and Stanmore (1972). Experimental investigations, at electrolyte temperatures varying from 17°C to 45°C, have been made for ionic mass transfer in a parallel plate electrochemical cell operated with an acidified copper sulphate system, under both laminar and turbulent flow conditions. The results obtained in the vertically mounted flow cell, with an integral rectangular cross-section of 0.5 cm x 3.00 cm and an entry length of 78 cm, operated under laminar flow conditions were found to be well represented by the modified Lévêque equation. This was obtained by introducing the cell aspect ratio as an additional parameter into the Lévêque equation. The influence of decreased mass transfer at the longitudinal edges of the electrodes due to the changes in the velocity profile was found to be small. Therefore the investigators ignored the variation of velocity across the electrode in the z-direction and merely redefined the parameters in the above equation (3.1.3) to give:

\[
Sh = 1.467 \ (\text{Re Sc} \frac{d_e}{L})^{1/3} \ (2/(1+\gamma))^{1/3} \tag{3.1.4}
\]

Experimental investigations have been made of ionic mass transfer in a vertical parallel plate electrochemical cell under laminar and turbulent flow conditions (Pickett and Stanmore, 1972). The acidified copper sulphate bulk solution temperature was varied (17°C and 45°C) in order to investigate the influence of the Schmidt number on the mass transfer coefficient. Overall, the data in the laminar flow
range were correlated by:

\[ Sh = 2.54 \text{Re}^{10} \text{Sc}^{0.29} (d_e / L)^{1/3} \]  \hspace{1cm} (3.1.5)

There are experimental data available for verifying the validity of the above equations (Pickett and Stanmore, 1972; Pickett and Ong, 1974). Pickett (1977) re-examined available experimental data and concluded that there appears to be a trend away from the one-third power on the (Re Sc d_e / L) group. An empirical correlation of the data led to:

\[ Sh = 2.54 (\text{Re Sc d_e} / L)^{0.3} \]  \hspace{1cm} (3.1.6)

Mass transfer data by Wranglén and Nilsson (1962) obtained under the limiting current condition in a horizontal flow reactor with \( \gamma = 0.75 \) and using an acidified copper sulphate solution also show the proportionality between \( Sh \) and the Reynolds number to the \( 1/3 \) power:

\[ Sh = 0.331 \text{Re}^{1/3} \text{Sc}^{1/3} \]  \hspace{1cm} (3.1.7)

Results from the major investigations on mass transfer to planar electrodes are summarized in Table 3.1.1.

**Table 3.1.1 Mass Transfer Correlations for the Laminar Flow Region**

<table>
<thead>
<tr>
<th>Correlation</th>
<th>Range</th>
<th>Comments and References</th>
<th>Equation</th>
</tr>
</thead>
<tbody>
<tr>
<td>( Sh = 1.849(\text{Re Sc} d_e / L)^{1/3} )</td>
<td>Laminar flow</td>
<td>&quot;Lévêque equation&quot; Norris and Streid (1940)</td>
<td>3.1.1</td>
</tr>
<tr>
<td>Correlation</td>
<td>Range</td>
<td>Comments and References</td>
<td>Equation</td>
</tr>
<tr>
<td>-------------------------------------</td>
<td>--------------------------------</td>
<td>---------------------------------------------</td>
<td>----------</td>
</tr>
<tr>
<td>Sh = 1.849(\text{Re Sc } d_e / L)^{1.9}\</td>
<td>Laminar flow: (\text{Re Sc } Ld_e &gt; 10^4)</td>
<td>(\chi) depends on the (\gamma); theoretical; exp. verification; Roudar et al. (1971)</td>
<td>3.1.2</td>
</tr>
<tr>
<td>Sh = 0.807(f(\text{height, width}))(\text{Re Sc } d_e / L)^{1.9}\</td>
<td>Laminar flow</td>
<td>Width &gt; Height; Ong (1972)</td>
<td>3.1.3</td>
</tr>
<tr>
<td>Sh = 1.467(\text{Re Sc } d_e / L)^{1.9}(2(1+\gamma/2)^{1.9}\</td>
<td>Laminar flow</td>
<td>Modified Lévêque equation; theoretical; Pickett and Stanmore (1972)</td>
<td>3.1.4</td>
</tr>
<tr>
<td>Sh = 2.54 (\text{Re}^{1.9} \text{Sc}^{0.29} (d_e / L)^{1.9}\</td>
<td>210 &lt; Re &lt; 2000 (960 &lt; Sc &lt; 3300) (0.25 &lt; (d_e / L) &lt; 1.0) (\gamma = 0.167)</td>
<td>Electrode width to duct width was approx. 0.9; experimental</td>
<td>3.1.5</td>
</tr>
<tr>
<td>Sh = 2.54(\text{Re Sc } d_e / L)^{1.9}\</td>
<td>55 &lt; Re &lt; 2000 (2850 &lt; Sc &lt; 5140) (0.08 &lt; (d_e / L) &lt; 5.95) (\gamma = 0.175)</td>
<td>re-examined available experimental data; Pickett (1977)</td>
<td>3.1.6</td>
</tr>
<tr>
<td>Sh = 0.331 (\text{Re}^{1.2} \text{Sc}^{1.3}\</td>
<td>Laminar flow, (\gamma = 0.75)</td>
<td>experimental</td>
<td>3.1.7</td>
</tr>
</tbody>
</table>

### 3.2 EXPERIMENTAL AND THEORETICAL ANALYSIS OF TURBULENT CHANNEL FLOW

When the Reynolds number of a solution flowing through a parallel plate reactor exceeds about 2000, the flow becomes turbulent. Turbulent flow is characterized by random time-dependent velocity fluctuations superimposed on time independent mean velocities. The application of the Navier-Stokes equation to such a flow results in additional terms involving these fluctuations which can only be accounted for by empirical means. For planar electrodes in fully developed flow, Deissler (1954) has developed the semi-theoretical relationship:
Sh = 0.0222 Re_{0.475} Sc^{0.25} \hspace{1cm} (3.2.1)

For fully developed turbulent flow past planar electrodes Lin et al. (1953) proposed the following theoretical relationship for obtaining the convective mass transfer coefficient, $K$:

$$K \frac{d_e}{D} = 0.0113 \text{ Re}_{0.475} \text{ Sc}^{1/3} \hspace{1cm} (3.2.2)$$

The left hand side of the above equation could be written as the average Sherwood number because the mass transfer coefficient is in fact independent of the electrode length in the fully developed turbulent flow regime. Despite the different Schmidt number powers in the above equations, the calculated values of $K$ for a given practical electrochemical system are not much different in value.

A more empirical mass transfer relationship is provided by the Chilton-Colburn analogy (Chilton and Colburn, 1934) and has been advocated by both Hubbard and Lightfoot (1966) and Pickett and Ong (1974). This can be written:

$$Sh = 0.023 \text{ Re}_{5.8} \text{ Sc}^{1/3} \hspace{1cm} (3.2.3)$$

Hubbard and Lightfoot studied turbulent mass transfer in a horizontal rectangular duct via diffusion-controlled reduction of potassium ferriyanide in excess caustic. Observations were made at Schmidt numbers from 1700 to 30000 and Reynolds numbers from 7000 to 60000 by variation of temperature (1°C to 25°C), caustic concentration (1 mol/l to 4 mol/l) and flow rate. The electrolyte used was 0.005 mol/l with respect to both K$_2$Fe(CN)$_6$ and K$_4$Fe(CN)$_6$. Their results agreed more closely with the Chilton-Colburn analogy than with available semi-empirical expressions. This tends to cast doubt on the latter.

On the evidence of Pickett and Stanmore (1972) and Pickett and Ong (1974), all of the above equations can be used when the concentration profile is fully developed ($L/d_e > 12.5$). Ong (1972)
concluded that fully developed turbulent flow and fully developed mass transfer are obtained after about twelve equivalent diameters along the electrode. For electrodes shorter than twelve equivalent diameters in length, fully developed concentration profiles are not obtained and the mass transfer coefficient exhibits length dependence.

Reiss et al. (1963) developed a semi-theoretical relationship for fully developed turbulent flow, which enters a section of a pipe where mass (or heat) is exchanged between the fluid and the wall. They reported on experiments performed to determine the effect of the length of the mass transfer section on the average mass transfer rate. The electrolytic technique was the same as that described by Wilke et al. (1955). Ten mass transfer sections, with length of from 0.5 mm to 110 mm were employed. The Reynolds number was varied from 1000 to 75000, and the Schmidt number was held constant at about 2400. An originally flat concentration (or temperature) profile changed to one exhibiting large gradients of concentration (or temperature) near the wall. The shape of the profile in the fluid depended on the length of the mass transfer section until, for sufficiently long sections, a fully developed condition was reached. As a consequence of the development of the concentration profile, the local value of the transfer coefficient decreased from a very large value at the inlet to a minimum value downstream. Reiss et al. (1963) proposed a relationship which assumed a constant surface concentration condition in the laminar sublayer:

$$Sh = 0.276 \ Re^{0.58} \ Sc^{1/3} \ (d_e/L)^{1/3}$$  \hspace{1cm} (3.2.4)

Schütz (1964) conducted local mass transfer experiments in a tubular reactor with a fully developed hydrodynamic boundary layer. The tubular reactor with a diameter of 40 mm, was operated at flow rates resulting in Reynolds numbers varying between 5000 and 50000. Based on the evidence of the data of Schütz, and of Ross and Wragg (1965), the above equation (3.2.4) appears to describe mass transfer entrance effects in fully developed pipe flow quite well. Reiss et al. (1963) were using the limiting current technique to determine the average mass transfer coefficient for the entrance region of a tubular
reactor, which does not agree very well with the evidence presented by Schütz. Stanmore (1970), using a rectangular flow reactor, however, reported considerable disparity between their data and the above equation (3.2.4), particularly regarding the one-third power on the \((d_r/L)\) group. Pickett (1977) examined the existing data for parallel plate reactors and proposed:

\[
Sh = 0.145 \text{Re}^{2/3} \text{Sc}^{1/3} (d_r/L)^{1/3}
\]  

(3.2.5)

for short electrodes with \((L/d_r) < 7.5\).

The process chosen by Mizushina et al. (1971) was the reduction of ferricyanide at nickel electrodes in the presence of a large excess of sodium hydroxide. The experiments were performed in a horizontal rectangular channel, 5 mm x 50 mm cross section and 2 m long. Three nickel cathodes of 398 mm, 201 mm and 52 mm were mounted in series, flush with the upper surface in the centre of the horizontal duct. Measurements of mass transfer rates were made on the 201 mm cathode located downstream of the 398 mm cathode in order to assure a developed concentration profile. In order to validate the experimental results a theoretical analysis was performed. For a steady, fully developed rectilinear flow between parallel plates, the time smoothed governing equations were developed. It was assumed that the convection term in the governing concentration equation can be neglected; in other words, the mass flux was assumed to be constant at any distance above the mass transfer surface. It was found, for this rectangular flow reactor with \(3000 < \text{Re} < 80000\) and \(800 < \text{Sc} < 15000\), that both the theoretical predicted and the experimental results agree very well. Furthermore the results show that the Sherwood number varies with the Reynolds number to the 0.9th power, and the Schmidt number to the one-third power.

A review of mass transport phenomena in horizontal flow channel reactors was presented by Landau (1981). Data in the turbulent flow region, measured locally at the downstream edge of the mass transport section and expressed in terms of the dimensionless Sherwood number, are best described by two separate correlations (Landau, 1976): For the turbulent transition region, \(2800 < \text{Re} < 12000\),
$$Sh = 0.0113 \ Re^{0.92} \ Sc^{0.35}$$  \hspace{1cm} (3.2.6)$$

and for fully developed turbulent flow, $12000 < Re < 125000$.

$$Sh = 0.0100 \ Re^{0.92} \ Sc^{0.30}$$  \hspace{1cm} (3.2.7)$$

Experimental investigations have been made of ionic mass transfer in a vertical parallel plate electrochemical cell under laminar and turbulent flow conditions (Pickett and Stanmore, 1972). The acidified copper sulphate bulk solution temperature was varied (17°C and 45°C) in order to investigate the influence of the Schmidt number on the mass transfer coefficient. Pickett and Stanmore (1972) correlated their data for $3000 < Re < 80000$ best by:

$$Sh = 0.0278 \ Re^{0.975} \ Sc^{0.21}$$  \hspace{1cm} (3.2.8)$$

The correlations discussed in this section are summarized in Table 3.2.1.

### Table 3.2.1 Mass Transfer Correlations for the Turbulent Flow Region

<table>
<thead>
<tr>
<th>Correlation</th>
<th>Range</th>
<th>Comments and References</th>
<th>Equation</th>
</tr>
</thead>
<tbody>
<tr>
<td>$Sh = 0.0222 \ Re^{0.975} \ Sc^{0.25}$</td>
<td>Fully developed turbulent flow \ $y' = 26$</td>
<td>theoretical \ $y' = y u / v$ \ $y = \text{distance from surface}$ \ Deissler (1954)</td>
<td>3.2.1</td>
</tr>
<tr>
<td>$Sh = 0.0113 \ Re^{0.97} \ Sc^{0.35}$</td>
<td>Fully developed turbulent flow</td>
<td>theoretical: \ Lin et al. (1953)</td>
<td>3.2.2</td>
</tr>
<tr>
<td>Correlation</td>
<td>Range</td>
<td>Comments and References</td>
<td>Equation</td>
</tr>
<tr>
<td>-------------</td>
<td>-------</td>
<td>-------------------------</td>
<td>----------</td>
</tr>
<tr>
<td>$Sh = 0.023 \ Re^{0.4} \ Sc^{0.3}$</td>
<td></td>
<td>Chilton-Colburn analogy</td>
<td>3.2.3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Chilton and Colburn (1934)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Pickett and Ong (1974)</td>
<td></td>
</tr>
<tr>
<td>$Sh = 0.276 \ Re^{0.84} \ Sc^{0.5} \ (d_e/L_e)^{0.7}$</td>
<td>Fully developed turbulent flow; 1000 &lt; Re &lt; 75000, Sc = 2400 Short electrodes</td>
<td>Average mass transfer coeff. derived from local values Reiss et al. (1963)</td>
<td>3.2.4</td>
</tr>
<tr>
<td>$Sh = 0.145 \ Re^{0.57} \ Sc^{0.8} \ (d_e/L_e)^{0.14}$</td>
<td>3000 &lt; Re &lt; 120000 400 &lt; Se &lt; 4600 $(L/L_e) &lt; 7.5$</td>
<td>Square channel; Fully developed turb. flow Stanmore (1970)</td>
<td>3.2.5</td>
</tr>
<tr>
<td>$Sh = 0.0113 \ Re^{0.87} \ Sc^{0.25}$</td>
<td>2800 &lt; Re &lt; 12000 800 &lt; Se &lt; 15000</td>
<td>Turbulent transition region Landau (1981)</td>
<td>3.2.6</td>
</tr>
<tr>
<td>$Sh = 0.0100 \ Re^{0.91} \ Sc^{0.24}$</td>
<td>12000 &lt; Re &lt; 125000 800 &lt; Se &lt; 15000</td>
<td>Fully developed turb. flow Landau (1981)</td>
<td>3.2.7</td>
</tr>
<tr>
<td>$Sh = 0.0271 \ Re^{0.873} \ Sc^{0.21}$</td>
<td>3000 &lt; Re &lt; 80000 960 &lt; Se &lt; 3300 0.025 &lt; $(d_e/L_e) &lt; 1.0$ $\gamma = 0.167$</td>
<td>Electrode width to duct width was approx. 0.9; experimental Pickett and Stanmore (1972)</td>
<td>3.2.8</td>
</tr>
</tbody>
</table>

### 3.3 FREE AND COMBINED CONVECTION

Due to the significant effect which buoyancy has on hydrodynamic and thermal conditions, mixed convection and free convection has been widely considered for analysis of heat transfer phenomena under laminar and turbulent flow conditions. Numerous experimental and theoretical studies for horizontal tubes and channels have revealed the nature of buoyancy-induced flow, and shown its effect on enhancing heat transfer, reducing the thermal entry length and promoting early transition to turbulence.

For pure natural convection, a theoretical model described by Schmidt and Beckmann (1930) can
be applied, using similarity of mass transfer to heat transfer. Free convection mass transfer at vertical electrodes can then be written in the form:

\[ \text{Sh} = c_1 (\text{Gr}_e \text{ Sc})^{0.25} \]  

(3.3.1)

As the Schmidt number approaches infinity, the constant, \( c_1 \), approaches 0.67. This has been verified by Wilke et al. (1953).

For horizontal upwards-facing circular surfaces, Wragg and Loomba (1970) found the Sherwood number to vary to the 0.25 power of \((\text{Gr}_e \text{ Sc})\) for laminar free convection \((3 \cdot 10^3 < (\text{Gr}_e \text{ Sc}) < 3 \cdot 10^7)\), and to the 0.33 power of \((\text{Gr}_e \text{ Sc})\) for turbulent forced convection \((3 \cdot 10^7 < (\text{Gr}_e \text{ Sc}) < 10^{12})\). The flow patterns were photographed (shadowgraph). It was postulated that for solid-liquid mass transfer in spatially unrestricted free convection at upward facing surfaces, involving a density decrease at the mass transfer surface, various types of flow pattern occur:

i. Laminar boundary layer and upward single column flow, at \((\text{Gr}_e \text{ Sc}) < 3 \cdot 10^6\)

ii. Transitional ‘mushroom’ type flow, at \(3 \cdot 10^6 < (\text{Gr}_e \text{ Sc}) < 2 \cdot 10^9\)

iii. Multi-columned upward flow, at \((\text{Gr}_e \text{ Sc}) > 2 \cdot 10^7\)

iv. Turbulent single column flow, at \((\text{Gr}_e \text{ Sc}) > 2 \cdot 10^9\).

These free convective mass transfer results compared favourably to the earlier data obtained by Fenech and Tobias (1960). Fenech and Tobias had applied the limiting current technique to determine the mass transfer at upwards facing horizontal electrodes in an unstirred reactor. The acidified copper sulphate electrolyte composition ranged from 0.01 to 0.7 mol/l. For electrode width larger than 20 mm the data were best represented by the general correlation:

\[ \text{Sh} = 0.19 (\text{Gr}_e \text{ Sc})^{1.0} \]  

(3.3.2)

The experimental range used in their correlation included \(10^8 < (\text{Gr}_e \text{ Sc}) < 1.4 \cdot 10^{12}\).
The effects of buoyancy forces on ionic mass transfer at horizontal planar electrodes were also studied experimentally with both laminar and turbulent bulk flow by Tobias and Hickman (1965). Copper electrodes and an acidified copper sulphate electrolyte were used to obtain mass transfer data by the limiting current technique in the ranges of parameters: \(10^3 < Gr_e < 10^{12}, 75 < Re < 7000, 600 < Sc < 12000\) and \(0.05 < (d_e/L) < 20\). With turbulent bulk flow, buoyancy forces were found to have a negligible effect on the mass transfer process in the range of variables covered. With laminar bulk flow, buoyancy forces induced free convective secondary flows near the electrode interface. For \(\log(Gr_e) > [2.96 + \log(Re)]\), it was assumed that the process was dominated by free convection, and the equation of Fenech and Tobias (1960) (eq. 3.3.2) could be used to predict the average mass transfer coefficients. Otherwise, forced convection dominates, and the Lévêque equation for a rectangular duct presented by Norris and Streid (1940),

\[
Sh = 1.849 \left(Re \ Sc \ \frac{d_e}{L}\right) \frac{1}{a},
\]

(3.3.3)

yielded acceptable correlation of the experimental data. A similar approach was also taken by Palade de Iribarne et al. (1970) for ionic mass transfer at channel electrodes involving inert zones. The electrolysis cell was both vertically and horizontally placed, in the latter case the working electrodes facing either upwards or downwards. The average dimensionless rate equation became:

\[
Sh = 1.16 \left(Re \ Sc \ \frac{d_e}{L}\right) \frac{1}{a} \left(\frac{d_e}{b}\right) \frac{1}{a}
\]

(3.3.4)

where \(b\) is the one half of the rectangular flow channel height. The experimental numerical coefficient was 10% lower than the one theoretically derived.

In solving the problem of superposed streamline free and forced heat transfer, Martinelli and Boelter (1942) proposed the following relationship for short vertical tubes:
\[ \text{Nu} = 1.75 \left( \text{Gr} + 0.0722 \ (\text{Gr} \text{ Pr} \text{ d}_l / \text{L})^{0.25} \right)^{0.13} \]  

(3.3.5)

Written in mass transfer terms the above equation becomes:

\[ \text{Sh} = 1.75 \left\{ \left( \pi / 4 \ \text{Re} \text{ Sc} \text{ d}_l / \text{L} \right) + 0.0722 \ (\text{Gr} \text{ Sc} \text{ d}_l / \text{L})^{0.25} \right\}^{0.33} \]  

(3.3.6)

The same model for correlating experimental data was adopted by Wragg and Ross (1967). Rates of mass transfer in conditions of transport control and in the mass transfer entry region were determined by measuring limiting currents for the deposition of copper on copper cathodes of different length from acidified solutions of copper sulphate. The cathodes in the experimental set-up of Wragg and Ross formed part of the inner wall of an vertical annular flow cell and the conditions were such that both forced and free streamline convective mechanisms were significant in determining the overall mass transfer rate. The experimental data were correlated by the equation

\[ \text{Sh} = \left\{ (1.96 \text{ Re} \text{ Sc} \text{ d}_l / \text{L}) + 0.04 \ (\text{Gr} \text{ Sc} \text{ d}_l / \text{L})^{0.25} \right\}^{0.39} \]  

(3.3.7)

for an annulus ratio of 0.5.

Wragg and Ross's results agreed very well with the equation

\[ \text{Sh} = 1.614 \left\{ (\text{Re} \text{ Sc} \text{ d}_l / \text{L}) + 0.092 \ (\text{Gr} \text{ Sc} \text{ d}_l / \text{L})^{0.25} \right\}^{0.33} \]  

(3.3.8)

which was obtained through Stanmore's work by using a vertical parallel plate reactor (after Pickett, 1977). Equation (3.3.7) applies to circular tubes and for application to a cell of annular geometry and with a annulus ratio of 0.5. To date, no other work on combined convective mass transfer in channel flow appears to be published.

The correlations for mass transfer under free and forced convection discussed in this section are
summarized in Table 3.3.1.

<table>
<thead>
<tr>
<th>Correlation</th>
<th>Range</th>
<th>Comments and References</th>
<th>Equation</th>
</tr>
</thead>
</table>
| $Sh = 0.67 \ (Gr, \ Sc)^{0.28}$ | $Sc \rightarrow \infty$ | Schmidt and Beckmann (1930)
Wilke et al. (1953) | 3.3.1 |
| $Sh = 0.19 \ (Gr, \ Sc)^{0.9}$ | $10^6 < Gr, \ Sc < 1.4 \times 10^{12}$ | Fenech and Tobias (1960) | 3.3.2 |
| $Sh = 0.19 \ (Gr, \ Sc)^{0.9}$ | $10^5 < Gr < 10^{15}$; $75 < Re < 7000$; $600 < Sc < 12000$; $0.05 < (d_u/L) < 20$ | log($Gr_c$) $> [2.96 + \log(Re)]$ free convection; Tobias and Hickman (1965) | 3.3.2 |
| $Sh = 1.849 \ (Re \ Sc \ d_u/L)^{0.9}$ | $10^5 < Gr < 10^{15}$; $75 < Re < 7000$; $600 < Sc < 12000$; $0.05 < (d_u/L) < 20$ | log($Gr_c$) $< [2.96 + \log(Re)]$ forced convection Norris and Streid (1940) Tobias and Hickman (1965) | 3.3.3 |
| $Sh = 1.16 \ (Re \ Sc \ d_u/L)^{0.9} (d_u/b)^{0.33}$ | Laminar flow | Electrode with inert zones: $b = 0.5$ channel height Palade de Iribane et al. (1970) | 3.3.4 |
| $Nu = 1.75 \ (Grz + 0.0722 \ (Gr, \ Pr \ d_u/L)^{0.25})^{0.33}$ | | Short vertical tubes Martinelli and Boelter (1942) | 3.3.5 |
| $Sh = 1.75 \ ((w/4 \ Re \ Sc \ d_u/L) + 0.0722 \ (Grz, \ Sc \ d_u/L)^{0.33})^{0.33}$ | | Eq. 3.3.5 in mass transfer terms; Martinelli and Boelter (1942) | 3.3.6 |
| $Sh = ((1.96 \ Re \ Sc \ d_u/L) + 0.04 \ (Grz, \ Sc \ d_u/L)^{0.33})^{0.33}$ | $10 < Re < 500$ | Annulus ratio = 0.5 Wragg and Ross (1967) | 3.3.7 |
3.4 ANALYSIS OF COMBINED HEAT AND MASS TRANSFER

Mass transfer operations are often accompanied by the transfer of heat. In the past, these have been largely studied as independent processes. In natural convection the two processes have a definite and predictable effect on each other and hence need not be considered separately.

Relatively little experimental work in combined heat and mass transfer has been published for liquid-solid systems such as in an electrochemical reactor. Fewer papers are published dealing specifically with electrochemical processes, and only one has been found for channel flow. The scientific literature appears to be devoid of studies aimed at the clarification of the combined effects of applied thermal and concentration gradients on mass transfer rates.

The effects of the combined transport phenomena can be significant, as observed in the work of Mathers et al. (1957) which dealt with transfer of naphthalene from coated spheres to air. A force balance, total material balance, and a material balance on the volatile component was used to derive the governing partial differential equations. A steady-state numerical solution for a free convection solid-gas system was obtained by the use of an analog computer. They introduced combined Grashof numbers $\text{GR}_h$ and $\text{GR}_c$, which took into account the buoyancy effects of both concentration and temperature gradients:

$$\text{GR}_h = \text{Gr}_h + (\text{Pr}/\text{Sc})^{1/2} \text{Gr}_c$$

$$\text{GR}_c = \text{Gr}_c + (\text{Sc}/\text{Pr})^{1/2} \text{Gr}_h$$
After experimental verification (evaporation of naphthalene sphere) the following expressions were proposed:

\[ \text{Nu} = 0.670 \ (GR, \ Pr)^{1/4} \quad (3.4.3) \]
\[ \text{Sh} = 0.670 \ (GR, \ Sc)^{1/4} \quad (3.4.4) \]

The above equations agree remarkably well with the more exact calculations obtained by Ostrach (1952) on a card-programmed digital computer for the simple heat transfer problem.

Wilke et al. (1953), using their previously published results for isothermal heat transfer from a vertical plate to a liquid, although with a different \((Gr, \ Sc)\) range, found good agreement with the correlations proposed by Mathers et al (1957). Thus the Mathers approach and equations appear to have applicability to solid-liquid stems.

De Leeuw den Bouten et al. (1968) studied simultaneous heat and mass transfer in laminar free convection in the liquid phase at a vertical plate. The De Leeuw den Bouten et al. (1968) experimental studies of simultaneous heat and mass transfer in laminar free convection appear to be the earliest ones to apply the work of Mathers et al. (1957) to a solid-liquid system. They used the limiting current technique with an acidified copper sulphate electrolyte solution, obtaining results which confirmed the use of the theoretically derived composite Grashof number. Two groups of experiments were performed: i. mass transfer alone, and ii. simultaneous heat and mass transfer, with cathode temperatures of 14°C and 30°C and the bulk solution temperature varying from 15°C to 29°C. The results for high Schmidt and Prandtl numbers showed that heat transfer affected mass transfer more than mass transfer affected heat transfer. The data were best correlated by the general equation:

\[ \text{Sh} = c_2 \ (GR, \ Sc)^a \quad (3.4.5) \]

The statistical treatment of results gave the values:
\[ n = 0.243 \pm 0.007 \]  
(3.4.6)

\[ c_x = 0.79 \pm 0.12 \]  
(3.4.7)

These experimental results did not confirm Mathers et al.'s solution rigorously since only two different 
\( (GR_x, Pr) \), \( (GR_x, Sc) \) values were available.

Marchiano and Arvía (1968) derived a theoretical solution for laminar free convection under 
simultaneous concentration and thermal gradients at an ideal vertical plate electrode. Their rate equation 
is also valid for the limiting case of isothermal free convection and presents a relationship between the 
maximum average flux and the temperature gradient. The same researchers (Marchiano and Arvía, 1969) 
conducted experiments for testing their developed relationships. In dimensionless form, the equations were 
presented as:

\[ Sh = 0.637 \ (GR_x, Sc)^{1/4} \]  
(3.4.8)

where the combined Grashof number was redefined in the following form:

\[ GR_x = [Gr_x^{1/4} \pm Gr_x \ (Sc/Pr)^{1/4}]^{1/4} \]  
(3.4.9)

Wragg and Nasiruddin (1973) criticized Marchiano and Arvía's approach for testing their model, 
because the experimental conditions were not the same as the assumptions in the theoretical model (i.e. 
no control of the bulk solution temperature and instead, the counter electrode temperature was maintained 
constant). Wragg and Nasiruddin investigated non-isothermal free convection with an upward facing 
horizontal disc electrode of different sizes and a wider range of copper sulphate concentrations and 
temperatures than Marchiano and Arvía. The data were correlated as follows:

\[ Sh = 1.75 \ (GR_x, Sc)^{0.277} \]  
(3.4.10)
\[ Sh = 0.163 \left( \text{Gr, Sc} \right)^{1/9} \]  

(3.4.11)

for the ranges of \(10^5 < (\text{Gr, Sc}) < 5 \cdot 10^9\) and \(5 \cdot 10^9 > (\text{Gr, Sc})\), respectively.

The most recent experimental work was performed by Ajersch (1990). The limiting current technique was used to investigate combined heat and mass transfer in a horizontal parallel-plate electrochemical flow reactor, for Reynolds numbers ranging from 200 to 4400. The bulk solution was maintained at 25°C, and the cathode surface temperature varied from 30°C to 50°C. For these combined heat and mass transfer experiments, mass transfer was found to be controlled by free convection in the laminar region, whereas in the transition and turbulent regime, mass transfer was found to be controlled by free and forced convection. The results for the laminar and transition/turbulent regions respectively were correlated by:

\[ Sh = 0.000469 \left( \text{Gr, Sc} \right)^{0.33} \]  

(3.4.12)

and

\[ Sh = 0.0732 \times 10^{0.37} \left( \text{Gr, Sc} \right)^{0.15} \]  

(3.4.13)

There are only a few publications concerning the theoretical treatment of combined heat and mass transfer. A steady state laminar boundary layer analysis of combined heat and mass transfer characteristics has been carried out by Hasan and Mujumdar (1985). The problem of combined heat and mass transfer in laminar free convection along a vertically rotating plate subjected to uniform heat flux and/or a uniform mass flux was studied. They considered gases or liquid vapours to diffuse into a main fluid. Numerical results of the local wall shear stress, the local Nusselt number, and the local Sherwood number were presented in tabular form. It was found that along the rotating plate the buoyancy force decreases causing the local wall shear stress, the local Nusselt number and the local Sherwood number to decrease along the plate. For a fixed location along the rotating plate, the local Nusselt number and the local wall shear stress are found to enhance and diminish when the buoyancy force from species diffusion reinforces and
counteracts, respectively, the thermal buoyancy force. The local Nusselt number and the local wall shear stress enhances with the decrease of Schmidt number while local Sherwood number increases with the increase of the Schmidt number. In addition, the species diffusion becomes more effective with the increase of the thermal buoyancy force.

Chen and Strobel (1980) studied the heat and mass transfer characteristics of mixed convection flow along vertical and inclined flat plates under combined buoyancy effects of thermal and mass diffusion. In Chen's previous studies (1979) it was shown that the effects of thermal buoyancy forces on the wall shear and surface heat transfer rate diminished as the angle of inclination from the vertical increased, for both assisting and opposing flows.

The effects of the buoyancy-induced pressure gradient, the Prandtl number, and the ratio of wall velocity to free-stream velocity on the laminar forced-convective flow and heat and mass transfer over a horizontal plane were studied analytically by Yeh et al. (1987). Numerical results were presented for Prandtl numbers of 0.7 and 7.0 and a Schmidt number of 0.6. It was shown that the local friction factor, local Nusselt number and local Sherwood number increased with increasing buoyancy force.

Yan and Lin (1988) investigated the role of vaporization or condensation of the water vapour on the wetted vertical channel walls in laminar mixed convection flows under simultaneous influences of combined buoyancy effects of thermal and mass diffusion. Results for an air-water system under various conditions were presented. The effects of wetted wall temperatures and the Reynolds number of the flow were examined in great detail. It was shown that the presence of the mass diffusion effect caused pronounced heat transfer enhancement.

Simultaneous heat and mass transfer through laminar boundary layers in combined forced and free convection was studied by Barton and Trass (1969). Heat transfer coefficients were obtained from temperature gradients at the wall and mass transfer rates from thickness decrease measurements of naphthalene coatings. The forced convection data covered a Grashof number range from 100 to $2 \times 10^6$ and the combined flow parameters $(Gr / Re^3)$ and $(Gr / Re^3)$ vary from 0.2 to 100 and 0.02 to 3.25, respectively. The absorption of ultraviolet light by naphthalene vapour was used to measure free
convection concentration profiles. The predictions from the boundary layer solution were in good agreement with the experimental data.

Besides such classical chemical engineering topics as drying and distillation processes, no other theoretical and experimental work for combined heat and mass transfer in any other flow configuration appears to exist. The correlations discussed in this section are summarized in Table 3.4.1.

**Table 3.4.1 Correlations for Combined Heat and Mass Transfer**

<table>
<thead>
<tr>
<th>Correlation</th>
<th>Range</th>
<th>Comments and References</th>
<th>Equation</th>
</tr>
</thead>
<tbody>
<tr>
<td>$Nu = 0.670 (GR_e Pr)^{0.14}$</td>
<td>$GR_e = GR_t + (Pr/Sc)^{0.12} GR_t$</td>
<td>Experimental and theoretical&lt;br&gt;Mathers et al. (1957)</td>
<td>3.4.3</td>
</tr>
<tr>
<td>$Sh = 0.670 (GR_e Sc)^{0.13}$</td>
<td>$GR_e = GR_t + (Sc/Pr)^{0.12} GR_t$</td>
<td>Experimental and theoretical&lt;br&gt;Mathers et al. (1957)</td>
<td>3.4.4</td>
</tr>
<tr>
<td>$Sh = 0.79 (Gr_e Sc)^{0.23}$</td>
<td>$T_{in} = 14^\circ C$ and $30^\circ C$&lt;br&gt;$T_{in} = 15^\circ C$ to $25^\circ C$</td>
<td>High Schmidt numbers&lt;br&gt;Leeuw den Bouter et al. (1968)</td>
<td>3.4.5</td>
</tr>
<tr>
<td>$Sh = 0.637 (Gr_e Sc)^{0.16}$</td>
<td>$GR_e = [Gr_e^{0.44} + Gr_t (Sc/Pr)^{0.13}]^{0.3}$</td>
<td>Experimental and theoretical&lt;br&gt;Marchiano and Arvia (1969)</td>
<td>3.4.8</td>
</tr>
<tr>
<td>$Sh = 1.75 (Gr_e Sc)^{0.27}$</td>
<td>$10^7 &lt; GR_e Sc &lt; 5 \times 10^9$</td>
<td>Data used from:&lt;br&gt;Marchiano and Arvia (1969)&lt;br&gt;Ref.: Wragg and Nasiruddin (1973)</td>
<td>3.4.10</td>
</tr>
<tr>
<td>$Sh = 0.163 (Gr_e Sc)^{0.32}$</td>
<td>$5 \times 10^9 &gt; GR_e Sc$</td>
<td>Data used from:&lt;br&gt;Marchiano and Arvia (1969)&lt;br&gt;Ref.: Wragg and Nasiruddin (1973)</td>
<td>3.4.11</td>
</tr>
<tr>
<td>$Sh = 0.000469 (Gr_e Sc)^{0.23}$</td>
<td>$(Re \ Sc \ d/L) &lt; 7.1 \times 10^3$&lt;br&gt;$T_{in} = 25^\circ C$;&lt;br&gt;$T_{in} = 30$ to $50^\circ C$</td>
<td>Non-isothermal mixed convection:&lt;br&gt;experimental&lt;br&gt;Ajersch (1990)</td>
<td>3.4.12</td>
</tr>
<tr>
<td>Correlation</td>
<td>Range</td>
<td>Comments and References</td>
<td>Equation</td>
</tr>
<tr>
<td>----------------------------</td>
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<td>-------------------------------</td>
<td>----------</td>
</tr>
<tr>
<td>Sh = 0.0732 Re0.85 (Gr, Sc0.1)</td>
<td>(Re Sc d, /L) &gt; 7.1×10^4;</td>
<td>Non-isothermal mixed convection;</td>
<td>3.4.13</td>
</tr>
<tr>
<td></td>
<td>Tmkn = 25°C;</td>
<td>experimental</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Tcalor = 30 to 50°C</td>
<td>Ajersch (1990)</td>
<td></td>
</tr>
</tbody>
</table>

The correlations presented in Tables 3.1.1, 3.2.1, 3.3.1 and 3.4.1 are an extract of a larger spreadsheet containing more correlations and a more detailed analysis of the conditions reported in the publications found in the literature (see Lotus File: GBHMT1.WK3).

### 3.5 SOLUTION PROPERTIES

The choice of electrochemical systems used for limiting current mass transport studies is determined by several factors. The main factors are a low cost system which provides a well defined long limiting current plateau. Different electrochemical systems are described by Selman and Tobias (1978). Of the systems reported so far in the literature, however, the deposition of copper from an acidified copper sulphate solution and the reduction of ferri- to ferrocyanide from solutions containing a large excess of NaOH or KOH are most widely used. The latter system was chosen for this project. Since most mass transfer correlations are presented in terms of the Sherwood-, Reynolds-, Schmidt- and Grashof numbers, the most important electrolyte solution properties to consider are the density, viscosity and diffusivity. Correlations found in the literature for these physical properties are discussed below.

#### 3.5.1 Diffusivity Data

For studies of the mass transfer rates in electrochemical systems, a solution containing potassium ferro- and ferricyanide and sodium hydroxide in excess has very often been used, because the redox reaction involved can be studied in both directions on activated nickel or platinum electrodes. The effective diffusion coefficient of ferro- and ferricyanide ions in aqueous sodium hydroxide solutions, is
required to interpret ionic mass transfer data.

Studies of the diffusion of ferro- and ferricyanide ions in aqueous sodium hydroxide solutions showed that the Stokes-Einstein ratio involving the effective diffusion coefficient, the macroscopic viscosity and the absolute temperature is satisfactorily constant. Data reported in the literature (Eisenberg et al., 1954 and 1956) about the diffusion coefficients of potassium ferro- and ferricyanide are for solutions where the concentration of the supporting electrolyte was not appreciably changed. Bazán and Arvía (1965) evaluated the diffusion coefficients within a large range of concentration using a rotating disk electrode operated under limiting current conditions. To calculate the experimental diffusion coefficients, the Levich equation (Levich, 1962) for limiting current density under convective-diffusion control for the rotating disk electrode was used:

\[ i_L = 0.621 \times F \cdot c \cdot D_i^{2/3} \cdot v^{1/6} \cdot \omega^{1/2} \]  

The determination of the dependence of \( i_L \) on \( \omega \) thus allows the calculation of the diffusion coefficient. Aqueous solutions of potassium ferro- and ferricyanide were used; the concentration of these salts were varied between 0.0001 mol/l and 0.1 mol/l. The caustic soda concentration was approx. 0.5 mol/l, 1 mol/l and 2 mol/l. The experimental diffusion coefficients (Bazán and Arvía, 1965) were fitted to an exponential law, in the familiar Stokes-Einstein format, with respect to temperature for a solution temperature range between 24°C and 40°C. The diffusion coefficients for potassium ferricyanide, \( D_{03} \), and potassium ferrocyanide, \( D_{04} \), were thus best estimated by

\[ D_{03} \mu/T = (2.54\pm0.13)\cdot10^{-13} \text{ kg m/(s}^2 \text{ K)} \]  
\[ D_{04} \mu/T = (2.09\pm0.11)\cdot10^{-13} \text{ kg m/(s}^2 \text{ K)} \]

The above equations are valid for supporting electrolyte concentrations used during the course of the present study.
Bourne et al. (1985) also reported diffusion coefficients for potassium ferri- and ferrocyanide in concentrations of 10 and 20 mol-m\(^{-3}\), respectively. The higher ferrocyanide concentration was used together with a larger anode than cathode area to protect the system against anodic oxidation and ensure that the cathodic process becomes rate determining. To suppress mass transfer by ionic migration, 500 to 3000 mol-m\(^{-3}\) was added as indifferent electrolyte. The average Stokes-Einstein product for a temperature range of 15°C to 35°C was best expressed by

\[ D_{03} \mu / T = 2.511 \times 10^{-14} (\pm 1.7\%) \ \text{kg m/(s}^2 \ \text{K}) \]  

This temperature variation coupled with the wide range of NaOH concentrations permitted a range of Schmidt numbers from 873 to 6279 to be utilized.

The results of Gordon et al. (1966) for densities, viscosities and diffusivities were obtained in equimolar ferro-ferricyanide electrolyte solutions (0.01 mol/l to 0.20 mol/l) in the presence of NaOH or KOH (0.0 mol/l to 3.5 mol/l). Again a rotating disk electrode was used for the measurement of the ionic diffusion coefficients. A least square fit of the values obtained with KOH or NaOH agreed within ±4% of the experimental data and resulted in the following relationships and standard errors of estimate:

\[ D_{03} \mu / T = (0.234 + 0.0014 \Gamma) \times 10^{-14} \ \text{kg m/(s}^2 \ \text{K}) \]  
\[ D_{04} \mu / T = (0.187 + 0.0034 \Gamma) \times 10^{-14} \ \text{kg m/(s}^2 \ \text{K}) \]  

Gordon et al. (1966) state that although the temperature was not a variable in their study, the parameter (D \(\mu\)/T should be independent of temperature since the behaviour of the ferro- or ferricyanide ion should be similar in this respect to that of the large tri-iodide ion. As a comparison, the data of Eisenberg et al. (1956) at \(c_{NaOH} = 2 \ \text{mol/l}\) and \(\Gamma = 5.15 \ \text{mol/l},\) gave

\[ D_{03} \mu / T = 0.250 \times 10^{-14} \ \text{kg m/(s}^2 \ \text{K}) \]  

(3.5.1.7)
\[ D_{oa} \mu / T = 0.215 \cdot 10^{-14} \text{ kg m/(s}^2 \text{ K)} \]  (3.5.1.8)

whereas Gordon et al.'s equations gave, at the same ionic strength:

\[ D_{oa} \mu / T = 0.240 \cdot 10^{-14} \text{ kg m/(s}^2 \text{ K)} \]  (3.5.1.9)
\[ D_{oa} \mu / T = 0.202 \cdot 10^{-14} \text{ kg m/(s}^2 \text{ K)} \]  (3.5.1.10)

Gordon et al. (1966) claimed that the effusion capillary technique used by Eisenberg et al. (1956) could be responsible for the difference.

A vertical pipe wall electrode was used in the work of Tonini et al. (1978), and the results of diffusion coefficients for different electrochemical solutions, at temperatures ranging from 17°C to 30°C were presented. For a solution composition of an equimolar amount of 0.01 mol/l potassium ferro- and ferricyanide and 2 mol/l NaOH the diffusivity of the Fe(CN)\(^3\) ion was described by

\[ D_{oa} \mu / T = 2.60 \cdot 10^{-15} \text{ kg m/(s}^2 \text{ K)} \]  (3.5.1.11)

For this study diffusion coefficients could not be determined with equations 3.5.1.2 to 3.5.1.6 since the temperature ranges were not as wide as those required. It was decided to use in addition to the above equations (3.5.1.2 to 3.5.1.6) the data compiled in Table A.1.1. Using multiple regression, the diffusivities were best expressed in terms of the molar concentration of caustic soda, potassium ferrocyanide, potassium ferricyanide and the temperature in degrees Celsius:

\[
D \left[ \frac{cm^2}{s} \right] = 0.01 \left( 5.5207 \cdot 10^{-4} - 1.4448 \cdot 10^{-4} C_{NaOH} - 5.989 \cdot 10^{-4} C_{pm} - 3.367 \cdot 10^{-4} C_{fer} \right. \\
\left. + 6.22 \cdot 10^{-4} T + 7.28 \cdot 10^{-4} C_{pm}^2 \right. \\
\left. + 1.258 \cdot 10^{-3} C_{pm}^2 \right. \\
\left. + 9.26 \cdot 10^{-4} C_{fer}^2 \right. \\
\left. + 1.7 \cdot 10^{-7} T^2 \right)
\]  (3.5.1.11)
3.5.2 Density Data

Bourne et al. (1985) conducted extensive measurements on potassium ferro- and ferricyanide solutions. Measured densities were expressed as functions of temperature according to

\[ \rho = c_3 + c_4 T \]  \hspace{1cm} (3.5.2.1)

giving the values of \( c_3 \) and \( c_4 \) as functions of NaOH concentrations. Concentrations of potassium ferri- and ferrocyanide were 10 and 20 mol·m\(^{-3}\), respectively. The addition of potassium ferro- and ferricyanide at the low concentrations considered here resulted in an increase in density of about 0.7% compared to NaOH solutions.

Gordon et al. (1966) obtained density data for the electrolyte solutions mentioned above by using a pycnometer. All solutions were thermostated to 25°C ± 0.05°C before the measurements. Gordon et al. followed the pycnometer technique described by Willihganx (1934) for measuring the solution densities. A second degree polynomial involving \( C_{\text{redox}} \) and \( C_{\text{NaOH}} \) was fitted to the data by a least-squares technique. The equations and their associated standard errors of estimate are:

\[ \rho [\text{g/cm}^3] = 0.9971 + 0.3996 \cdot C_{\text{redox}} + 0.427 \cdot C_{\text{NaOH}} - 0.00113 \cdot C_{\text{NaOH}}^2 - 0.00657 \cdot C_{\text{redox}} \cdot C_{\text{NaOH}} \]  \hspace{1cm} (3.5.2.2)

The above equation is valid in the range of \( C_{\text{redox}} = 0.01 \text{ mol/l to 0.20 mol/l and } C_{\text{NaOH}} = 0.0 \text{ mol/l to 3.5 mol/l}. \) The standard error of estimate = 0.0011 g/cm\(^3\).

Many authors (Washburn, 1929; Eisenberg et al., 1956; Reiss et al., 1963; Bourne et al., 1965; Mohanta and Fahidy, 1976; Bazán and Arvía, 1964 and 1965; Sutay and Knudsen, 1967; Storck and Coeuret, 1977) reported individual densities for the same electrolyte system with a different composition, but no other density correlations for this system have been found.

Due to the limitations of Bourne et al. (1985) and Gordon et al. (1966) experimental investigations, it was decided to use in addition to their correlations the density data reported by
Eisenberg, 1956; Reiss et al., 1963; Bazán and Arvía, 1964; Bourne et al. 1985; Sutey and Knudsen, 1967; Storck and Coeuret, 1977 (see Appendix A: Table A.1.1). Using multiple regression, the densities were best expressed in terms of the molar concentration of caustic soda, potassium ferrocyanide, potassium ferricyanide and the temperature in degrees Celsius:

\[ \rho \left( \frac{g}{cm^3} \right) = 0.001 \left( 1006 + 41.9 C_{NaOH} + 151.58 C_{fer} + 233.95 C_{fer}^2 \right) \]
\[ \cdots - 0.2677T - 0.691 C_{NaOH}^2 + 9.55 C_{fer}^2 - 82.73 C_{fer} - 0.00229 T^2 \]  

(3.5.2.3)

3.5.3 Viscosity Data

Eisenberg et al. (1956) used an Ostwald viscometer with an efflux volume of 1.5 ml to determine electrolyte viscosities. The viscosity data were presented in form of a table for two different temperatures of 25°C and 30°C. The ferro- and ferricyanide composition varied from 0.01 mol/l to 0.05 mol/l whereas the support electrolyte concentration (2 mol/l NaOH) remained constant.

Willhinganx et al. (1934) had earlier published an analytical procedure for determining viscosities by using a U-tube viscometer. Gordon et al. (1966) followed this technique in their work for viscosity measurement of equimolar ferro- and ferricyanide solution (0.01 mol/l to 0.20 mol/l) in the presence of 0.0 mol/l to 3.5 mol/l NaOH. The authors published an equation for computing viscosities for the system containing potassium ferrocyanide, potassium ferricyanide and sodium hydroxide, where the viscosity was given as a second order polynomial.

\[ \mu [cp] = 0.8937 + 0.6969 C_{redx} + 0.1539 C_{NaOH} + 0.0235 C_{redx}^2 + 0.0450 C_{NaOH}^2 + 0.2527 C_{redx} C_{NaOH} \]  

(3.5.3.1)

The standard error of estimate was given to be equal to 0.014 cp.

Individual viscosity data were given by (Eisenberg, 1956; Reiss et al., 1963; Bazán and Arvía,
1964 and 1965; Bourne et al. 1985; Sutey and Knudsen, 1967; Storck and Coeuret, 1977; Grassmann et al. 1961) but no other correlations were found.

Due to the limitations of Bourne et al. (1985), Eisenberg et al. (1956) and Gordon et al. (1966) experimental investigations, it was decided to use in addition to their correlations the viscosity data reported by Reiss et al., 1963; Bazán and Arvía, 1964; Sutey and Knudsen, 1967; Storck and Coeuret, 1977. These data, which are listed in Appendix A (Table A.1.1), and equation 3.5.3.1 were used to estimate the electrolyte viscosity. Using multiple regression, the viscosities were best expressed in terms of the molar concentration of caustic soda, potassium ferrocyanide, potassium ferricyanide and the temperature in degrees Celsius:

\[
\nu \left[ \frac{cm^2}{s} \right] = 0.02 + 0.00195 C_{\text{NaOH}} + 0.0032 C_{\text{FeCN}} - 0.00267 C_{\text{FeCN}} - 0.0006327 T \\
\ldots + 3.9 \cdot 10^{-3} C_{\text{NaOH}}^2 + 0.0029 C_{\text{FeCN}}^2 + 0.0305 C_{\text{FeCN}}^2 + 7 \cdot 10^{-6} T^2
\]  

(3.5.3.2)

3.5.4 Heat Capacity Data / Thermal Conductivity Data

The heat capacity of an ideal solution is, by definition, the sum of the heat capacities of the various constituents in their pure states at the same temperature and pressure. In any actual solution, this ideal heat capacity will be modified by the intermolecular and electrostatic effects which each constituent exerts upon the heat capacities of the other constituents. The heat capacity data found were based on the investigations of pure aqueous sodium hydroxide solutions (Richards and Hall, 1929).

Thermal conductivities, \( \lambda \), of aqueous solutions of sodium- and potassium hydroxide were measured by Riedel (1950) using the stationary cylinder technique. Based on dissociation in aqueous solutions the thermal conductivity could be predicted by

\[
\lambda = \lambda_w + \Sigma (\alpha_i c_i)
\]  

(3.5.4.1)
Applying this approach to the estimation of the thermal conductivity of aqueous NaOH solutions, Riedel found it in good agreement with his experimental values. Other results on thermal conductivities for sodium hydroxide solutions are reported in the form of tables (Landolt-Börnstein, 1936; Losenicky, 1969).

Since no data or correlations were found in the literature for the heat capacities and the thermal conductivities for the ferro- and ferricyanide, caustic soda electrolyte solution used in this study, it has been assumed that the values of the pure caustic solution will be a reasonable estimate of these values. Performing multiple regression on the data listed in Table A.1.1 (see Appendix A), the heat capacities (equation 3.5.4.3) and the thermal conductivities (equation 3.5.4.3) were best expressed in terms of the molar concentration of caustic soda and the temperature in degrees Celsius:

\[
C_p \left[ \frac{J}{gK} \right] = 4.18 - 0.229C_{NaOH} + 1.425 \times 10^{-4}T + 0.02903C_{NaOH}^2 - 3.748 \times 10^{-13}T^2 \quad (3.5.4.2)
\]

\[
\lambda \left[ \frac{J}{cm s^\circ C} \right] = 0.01 \left( 0.5841 + 0.0063161C_{NaOH} + 0.001541T - 0.000283C_{NaOH}^2 - 2.76 \times 10^{-6}T^2 \right) \quad (3.5.4.3)
\]

A more detailed explanation of the derivation of equations 3.5.1.11, 3.5.2.3, 3.5.3.2, 3.5.4.2 and 3.5.4.3 is summarized in Appendix A.
CHAPTER FOUR
EXPERIMENTAL

4.1 DESIGN OF THE RECTANGULAR CHANNEL FLOW APPARATUS

The cell and flow circuit are shown schematically in Figure 4.1.1. The system forms a closed loop from the centrifugal pump through a rotameter, the test section, and a 55 litre Nalgene storage tank. The tubing consisted of 3/4" and 1" PVC pipes, aluminium foil covered Tygon tubing and PVC valves. A bypass loop is provided to allow a better control of the flow rate. The electrolyte was maintained at the desired temperature with a Brinkman-IC2 Immersion Circulator placed in the storage tank.

The flow cell, which is shown schematically in Figures 4.1.2 and 4.1.3, was made of Plexiglas™ with an internal rectangular cross-section 2 cm x 1.5 cm (width x height), the total length being 229 cm between inlet and outlet. This permitted a hydrodynamic entry length of 127 cm (=74 hydraulic diameters) and an exit length of 87 cm. Both electrodes, manufactured from oxygen-free high purity copper, were plated by Aerospace Ltd. with 20 μm of nickel. The removable anode of 2 cm x 30 cm (width x length) is fitted on to one wall. The cathode (2 cm width x 15 cm length) is also removable and when bolted down, its surface is flush with the opposite channel wall. An O-ring provided the necessary seal. Two holes were drilled to allow 1/16" diameter ungrounded T-type thermocouples, labelled as T₁ and T₃ in Figure 4.1.4, to be located 1 mm from the cathode surface, and Swagelock fittings were used to provide the means of holding the thermocouples in place. The thermocouples, labelled as T₂ and T₄ in Figure 4.1.4, were inserted flush with the inside surface of the Plexiglas™ channel wall and secured in place with Swagelock fittings. In order to measure the bulk solution temperature, the thermocouples were mounted 20 cm and 18 cm from the upstream and downstream edge of the cathode surface, respectively. The electrical contact to the electrodes for the low voltage electrochemical circuit was achieved by a 1/8"
FIGURE 4.1.1 Electrochemical Flow Circuit

- **RECTIFIER/RECORDER:**
  - Voltage
  - Current

- **CATHODE-HEATER:**
  - Heater Voltage
  - Heater Current
  - Temperature

- **ELECTROLYTE:**
  - Temperature
  - Concentration
  - Volumetric Flow Rate
screw assembly. An auto-transformer (Hammond Mfg.) enabling an adjustable 0 to 115 V AC output was connected to the 9.7 mm in diameter, 300 Watt electrical cartridge heater (Chromolox Mfg.) which was inserted in the 10 mm diameter large hole through the cathode block in order to maintain a constant heat flux at the cathode surface. The transformer output voltage was adjusted to the desired value and the surface temperature was deemed to be equal to the reading of the value obtained from thermocouple T2. An isolation transformer was employed to minimize the line voltage noise. The heater voltage and current were measured with Beckman and/or BBC multimeters.

The low voltage electrochemical circuit is shown in Figure 4.1.5. Power was supplied to the cell via a Supplyst™ WP706 DC power supply. The input voltage was measured with a Hewlett-Packard 3465A digital multimeter. A 1.130 ohm resistor was placed in series with the electrochemical cell. The total resistance of the complete wiring assembly (resistor + lead wire) was measured as 1.238 ohms. The potential drop across the resistor assembly corresponded to the cell current, which was recorded on a Philips PM8100 Strip Chart Recorder and in addition measured and displayed by a Hewlett-Packard 3465A digital multimeter. The Omega CL-466 digital calibrator, which was calibrated by Thermo Electric Ltd., was used to verify the accuracy of the measuring/recording equipment. The Philips PM8100 Strip Chart Recorder and Hewlett-Packard 3465A digital multimeter (voltmeter) were accurate within and ±1 mV and ±0.1 mV over the range of 1 VDC, respectively. The 'voltage drop' data collection system, consisting of a Samsung SPC 3000 IBM® XT-compatible computer equipped with the D2121 analog-digital converter module (DGH Corporation, Manchester, NH 03108, U.S.A.) was connected in parallel to the resistor assembly. The Borland Turbo Pascal® 3.0 software routine (AVGVOLTS,PAS) enabled the user to read the voltage drop across the resistor assembly directly, without 'data entry errors' caused by the human interaction, into a data file. The data collection system was calibrated and accurate within ±0.1 mV.

The 'temperature' data acquisition system, consisting of a Samsung SPC 3000 IBM® XT-compatible computer, along with the DT-752, DT-756 and the DT-2801 boards from Transduction Ltd. for 8 bit analog-digital conversion, is shown schematically in Figure 4.1.4. The PCLAB software
FIGURE 4.1.4 Temperature Control and Data Collection System
Electrochemical Flow Reactor

DC Power Supply
0 - 5 VDC

Anode

R = 1.130 Ω

Cathode

Strip Chart Recorder
0 - 1 VDC

Computer incl. D2121
0 - 1 VDC

FIGURE 4.1.5 Low Voltage Electrochemical Circuit
subroutines for the thermocouple voltage to temperature conversion was supplied by Data Translation Inc. The data collection software (Appendix G) was written in Borland's Turbo Pascal* 3.0 programming language.

4.2 EXPERIMENTAL

During the course of the experimental work the laboratory temperature (ambient temperature) was frequently measured with an alcohol thermometer. The ambient temperature readings varied between 20°C and 21°C.

4.2.1 Calibration of Thermocouples

The ungrounded T-type 1/16" diameter thermocouples used in the experiments give measurements accurate to within ±0.25°C of the true temperature according to the manufacturer (Thermo Electric Ltd.). Each thermocouple assembly needs to be calibrated individually against a reference temperature, because measurements are very sensitive to factors such as the tolerances in the Transduction Ltd. data collection boards and the analog - digital data conversion.

All the thermocouples were taped together and placed 4 cm deep into a insulated and stirred 10 l bath of water to ensure that uniform temperature conditions existed for each thermocouple. An alcohol thermometer (Indication range: 18°C to 63°C), verified by Quantum Ltd. to be accurate within ±0.05°C over the whole temperature range, was placed close to the tips of the thermocouples. The water was stirred and slowly heated to the desired temperature level. The thermocouples were calibrated at temperatures between 20°C and 60°C in increments of 5°C. During this procedure the temperature was recorded with the data acquisition equipment and displayed on the computer monitor. For each individual thermocouple the difference between the water bath temperature and the one displayed on the computer monitor was minimized by adjusting the multi-turn potentiometers marked as 'zero' and/or 'span' on the Transduction Ltd. board. After numerous repetitions of the calibration procedure all thermocouples agreed within ±0.1°C of the alcohol thermometer over the full range between 20°C and 60°C. A verification of the system
computer ➔ data collection boards ➔ thermocouple wire to the output voltage of the Omega CL-466 digital calibrator, which was calibrated by Thermo Electric Ltd., showed differences of about ±0.06°C over the full range of calibration for each thermocouple channel. The test equipment and procedure used by Thermo Electric Ltd. is listed in Table 4.2.1.1.

**TABLE 4.2.1.1 Calibration Equipment of Thermo Electric Ltd.**

<table>
<thead>
<tr>
<th>EQUIPMENT TYPE</th>
<th>EQUIPMENT MANUFACTURER</th>
<th>SERIAL NUMBER</th>
</tr>
</thead>
<tbody>
<tr>
<td>D.C. Voltage Calibrator</td>
<td>E.D.C.</td>
<td>12308</td>
</tr>
<tr>
<td>Digital Multimeter</td>
<td>Fluke</td>
<td>3105049</td>
</tr>
<tr>
<td>Digital Multimeter</td>
<td>Hewlett-Packard</td>
<td>1211400104</td>
</tr>
</tbody>
</table>

The warm-up period to rated accuracy is one hour for the calibration equipment. The accuracy of the test equipment used for the calibration of the Omega CL-466 is traceable to the National Research Council of Canada and evidence is on file at Thermo Electric Ltd. The above described thermocouple calibration procedure was repeated once every week in order to assure an accurate calibration of the complete temperature recording equipment.

**4.2.2 Calibration of Rotameters**

The basic operation of a rotameter will be explained very briefly. The medium to be measured flows through a tapered circular tube in which a fixed diameter float is located. At each different position along the axis, the float allows a different free cross-sectional area to be accessible for the fluid. At steady state constant volume flow, the gravitational forces acting on the float equal the buoyancy and frictional forces exerted by the fluid on the float. The frictional forces are proportional to the pressure-drop, which results primarily from the velocity increase at the free cross-sectional area. As the volumetric flow increases, the resulting force increases and vice versa. Hence the position of the float is directly
proportional to the volumetric flow of the fluid.

Three rotameters, with overlapping but different volumetric flow ranges, were used in the electrochemical reactor assembly. They were constructed from different glass tubes and stainless steel floats. The calibration was performed by using the "bucket and stopwatch" method with the appropriate safety precautions (full alkaline resistance coverall, rubber boots, rubber gloves, safety goggles and face shield). The flow rate was found to vary linearly with the scale reading in all three measurement ranges. The calibration curves for 25°C and a nominal electrolyte solution (2 mol/l NaOH, 0.025 mol/l K₃[Fe(CN)₆], 0.025 mol/l K₄[Fe(CN)₆]) and the regression equations for the three rotameters (Small, Medium and Large) are shown in Appendix B. The same procedure was employed for volumetric flow measurements at different temperature readings of 30°C to 55°C performed in 5°C increments.

4.2.3 Preparation of the Electrolyte Solution and Quantitative Analysis

The electrolyte solution used in this project was made from reagent grade K₃[Fe(CN)₆], K₄[Fe(CN)₆], and NaOH (crystalline) all from BDH Chemicals, and deionized water. Approximately 65 litres of deionized water was placed in a storage tank, and the required amount of sodium hydroxide, predissolved in a small amount of water, was added. After thorough mixing of the solution, weighted amounts of potassium ferricyanide and ferrocyanide were added, and the solution mixed again. The nominal concentration of the electrolyte solution was:

\[ 2 \text{ mol/l NaOH} \]
\[ 0.025 \text{ mol/l K}_3\text{Fe(CN)}_6 \]
\[ 0.025 \text{ mol/l K}_4\text{Fe(CN)}_6 \]

A 25 ml sample was withdrawn and titrated with standard 1 mol/l hydrochloric acid according to the following equation, using phenolphthalein as an indicator (Jander and Blasius, 1977):

\[ \text{NaOH} + \text{HCl} \rightarrow \text{NaCl} + \text{H}_2\text{O} \quad \text{(4.2.3.1)} \]
The ferricyanide was analyzed iodometrically using the procedures commonly described (Rosin, 1946; Koltzoff and Sandell, 1955). A 50 ml sample was acidified with concentrated HCl and about 3 g potassium iodide, 1 drop of glacial acid, and a solution of 1.5 g zinc sulphate in 10 ml of water were added. The flask was closed, the contents mixed and allowed to react for about 30 minutes. Then the liberated iodine was titrated with standardized 0.1N (0.05 mol/l) sodium thiosulphate, using starch toward the end as an indicator. The blue starch-iodine colour was titrated to a colourless endpoint. The zinc serves to combine with the ferrocyanide as soon as the latter is formed, and by the virtue of the insolubility of zinc ferrocyanide, the Fe(CN)₆³⁻ is removed from reaction and the following reactions are permitted to go to completion. One ml of 0.1N (0.05 mol/l) sodium thiosulphate is equivalent to 0.03292 g K₃Fe(CN)₆.

\[
2\text{K}_3\text{Fe(CN)}_6 + 2\text{KI} \rightarrow 2\text{K}_4\text{Fe(CN)}_6 + 2\text{I} \tag{4.2.3.2}
\]

\[
2\text{I} + \text{Na}_2\text{S}_2\text{O}_3 \rightarrow \text{Na}_2\text{S}_4\text{O}_6 + 2\text{NaI} \tag{4.2.3.3}
\]

Because of the very dilute solution concentration, the titre was only reproducible within 0.05 ml. As it is common practice, at least three titrations were performed, and the average titre was used to calculate the ferricyanide ion concentration.

The ferrocyanide concentration was determined by using permanganometric (Rosin, 1946; Koltzoff and Sandell, 1955). A 50 ml sample was drawn and neutralized with concentrated hydrochloric acid. After adding 10 ml of concentrated H₂SO₄ the well mixed solution was titrated with standardized 0.1N (0.1 mol/l) potassium permanganate to slight reddish colour. One ml of permanganate is equivalent to 0.03683 g K₃Fe(CN)₆. The reaction equation for this titration process can be written as follows:

\[
5\text{K}_3\text{Fe(CN)}_6 + \text{KMnO}_4 + 4\text{H}_2\text{SO}_4 \rightarrow 5\text{K}_4\text{Fe(CN)}_6 + \text{MnSO}_4 + 3\text{K}_2\text{SO}_4 \tag{4.2.3.4}
\]

Again each titration was repeated at least 3 times and the average taken to calculate the ferrocyanide concentration.
After about every seven mass transfer experiments, a full set of analyses was carried out immediately in order to determine the concentrations of individual electrolyte components (NaOH, K₃Fe(CN)₆ and K₄Fe(CN)₆). The concentrations are given in the 'Data Summary' (see Appendix L).

During the course of the experiments electrolyte solution had to be replaced due to losses from evaporation and the taking of analysis samples. The make-up solution was stored in a dark closed container and was added as required. After some solution was added, and before commencing the experiments, the electrolyte solution was mixed by circulating it in the apparatus for about 10 minutes, and then analyzed as described above.

4.2.4 Electrode Preparation Procedure

In systems such as the parallel-plate electrochemical flow reactor in which the mass transfer rates are high, proper preparation of the electrode surface is necessary to ensure that the surface reaction rate will not be the controlling factor. Reiss and co-workers (1962, 1963) claimed that a satisfactory cleaning procedure for nickel electrodes consisted of buffing the surface followed by cathodic treatment in an electrolysis cell. Since with higher mass transfer rates, the surface conditions become much more critical, Dawson (1972) found this method to be inadequate for rough nickel surfaces. After some experimentation, he found that rinsing the surface for a few seconds with 1N (0.05 mol/l) sulphuric acid solution after buffing with commercial cleanser, followed by a 30 minute cathodic treatment in freshly prepared 1 mol/l sodium hydroxide at a cell potential of 6 volts, resulted in good, reproducible polarization curves. This electrode surface preparation method was used for all experiments reported here.

4.2.5 Preliminary Experiments

For familiarization with the equipment and procedures, a series of preliminary isothermal and non-isothermal experiments was conducted which are marked as IP1 to IP336 and NP1 to NP504 in the 'Data Summary' (see Appendix L), respectively.

A nomenclature was introduced in order to ensure an easy management of the expected amount
of isothermal and non-isothermal data. All data are presented in the following way:

Temperature/Temperature/Angle/Slope

which corresponds to:

Bulk temp., T₁, (°C) / Upstream Cath. surf. temp., T₂, (°C) / Cathode angle, α, (°) / Channel slope, β, (°).

The angle of the channel inclination (β) is defined as the angle of the cathode surface to a hypothetical horizontal surface in direction of flow. This definition is more useful for immediate visualization of the origin of the convective surface rather than the angle formed with the vertical. The angle of cathode rotation (α) is defined as the rotation angle of the cathode to a hypothetical surface perpendicular to the direction of flow (Figure 4.2.5.1).

4.2.5.1 Preliminary Experiments - Use of Polarization Curves

Before a series of experimental runs, the electrodes were treated as described in Chapter 4.2.4 and then installed in the apparatus. After the circulation of electrolyte through the apparatus was started, high purity nitrogen was bubbled through the reservoir for approximately one hour to drive off any dissolved oxygen from the electrolyte. Suthey and Knudsen (1967) reported on experiments where high purity (99.5 Vol.% nitrogen gas bubbled through the liquid with a fritted glass tube in order to drive off any dissolved oxygen in the electrolyte and to form a nitrogen blanket over the electrolyte, and starting 1 hour prior to the experimental runs. They emphasized the importance of eliminating the dissolved oxygen and avoiding light, and the proper cleaning of the electrodes in electrochemical systems containing ferri- and ferrocyanide. Their study has shown that, if the test solution is first saturated with nitrogen, future exposure to air for a short period of time does not greatly affect the mass transfer data. Under their flow conditions (Re < 22000), one could apply the limiting current technique up to 70% O₂ saturation within an operating time of 275 minutes and still obtain coefficients within 5% of the correct values. During this period the electrolyte was thermostated by the means of the immersion heater and the cooling coils at the required bulk solution temperature (T₁) and a quantitative analysis of the electrolyte was performed.
$\alpha = \text{CATHODE ANGLE}$

$\beta = \text{CHANNEL SLOPE}$

FIGURE 4.2.5.1 Cathode Angle and Channel Slope
For the isothermal preliminary experiments IP1 to IP126, polarization curves were obtained to define the range of applied potentials to achieve limiting current conditions in the reactor at different flow rates. Some sample polarization curves are shown in Figure 2.2.1. A potential, negative with respect to the cathode was applied to the cell in successive steps of 10 mV for potentials up to 100 mV. Above 100 mV cell potential the step size was increased to 50 mV until a potential of about 1600 mV was obtained. After each increase in cell potential, the current response was observed by measuring the voltage drop across the standard resistor with the strip chart recorder. For comparison and as a backup to the strip chart recorder, the current was also measured with the Hewlett-Packard 3465A precision voltmeter and the value recorded in a spreadsheet. The steady state current was usually reached within about 10 minutes. The power was disconnected, the voltage increased to the next increment, and the process repeated. This procedure was continued until the limiting current plateau was significantly surpassed, which usually occurred at about 1.5 V. After each limiting current curve was obtained the potential was turned off and the electrolyte flow increased to the maximum to remove any bubbles from the flow channel and electrodes.

4.2.5.2 Preliminary Experiments - Use of the Potential Step Method

Examination of Figure 2.2.1 shows that the limiting current plateau for an isothermal case (Re = 18200) can be observed between 300 mV and 1200 mV electrode potential. It can be concluded, from the experiments described in Section 4.2.5.1, that if a potential of about 800 mV is applied, the electrochemical system will operate at limiting current condition. Except for experiments IP1 to IP126 where complete polarization curves were obtained, all other experiments were performed by using the potential step method at a potential of approximately 800 mV. The advantage of conducting experiments with the potential step method was that the individual limiting current measurements could be obtained without requiring an entire polarization curve. This not only reduced the time to conduct one experiment to about 10 minutes but also enabled a larger number of experiments to be performed without cleaning the electrodes. Before a series of experimental runs, the electrodes were treated as described in Chapter
4.2.4 and then installed in the apparatus. After the circulation of electrolyte through the apparatus was started, high purity nitrogen was bubbled through the reservoir for approximately one hour to drive off any dissolved oxygen from the electrolyte. During this period the electrolyte was thermostated by the means of the immersion heater and the cooling coils at the required bulk solution temperature (T_r) and a quantitative analysis of the electrolyte was performed. A typical datum point was obtained by adjusting the temperatures and the volumetric flow rate to the desired values. After reaching the steady state flow rate and temperatures, the known potential as required to achieve limiting current (potential step to 800 mV) was applied to the electrochemical system. The 'voltage drop' computer data acquisition system (see sections 4.1 and 4.2.7) was activated when the limiting current reached the steady state value (observed on the strip chart recorder). The voltage drop across the resistor assembly was then read once every 0.5 seconds into a temporary data file. After 5 minutes of data collection, the average value of these 600 individual voltage readings was displayed on the computers monitor and automatically entered into the main data file. This average value was then manually compared to the voltage drops obtained from the chart recorder and the voltmeter. The temperatures, the voltage drop across the resistor which corresponds to the limiting current value, and the date were added automatically through the Turbo Pascal® program interface to this main data file. The rotameter setting, the time of cathodization and the bulk solution concentration were manually added to the data file. The potential was turned off, and the volumetric flow rate increased to its maximum in order to remove any gas bubbles from the channel walls. After every five experiments, the bulk solution concentration was determined as described in Section 4.2.3 and the electrodes were cleaned according to the procedure outlined in Section 4.2.4.

4.2.6 Reproducibility of the Experiments

To verify the reproducibility of the limiting current technique, sets of consecutive experiments were performed. In each of the isothermal and non-isothermal cases three different flow rates were utilized, rotation (α = 0°) and elevation (θ = 0°) were set, and the temperature adjusted as:

\[ T_i = 25^\circ C \]
non-isothermal: $T_1 = 25^\circ C$, $T_2 = 35^\circ C$.

A single bulk electrolyte concentration was employed (2 mol/l NaOH, 0.025 mol/l K$_3$Fe(CN)$_6$, 0.025 mol/l K$_4$Fe(CN)$_6$).

At flow rates of 25 cm$^3$/s (small rotameter) and 414 cm$^3$/s (large rotameter) 50 consecutive isothermal experiments were carried out but in the transition flow region (medium rotameter at a flowrate of 99 cm$^3$/s), where the change from the laminar to the turbulent flow was observed during the course of the preliminary experiments, 100 consecutive measurements were made. In each of the experiments (in both the 50 set and the 100 set) both the applied potential and the flow rate were set to the desired values. After each experiment the applied potential was set to zero, and the flow rate to the maximum possible for about 15 minutes and then both reset to the specified value. In each case the limiting current was read after it reached steady state (defined by no observable change in current within 10 minutes). The temperatures were also noted to ±0.01°C and then recorded to the nearest 0.1°C. For a given volumetric flow rate the replicate experiments were performed on at least two different days. After every seven experiments the cathodization process was performed as described in Section 4.2.4. In addition to the sets of isothermal experiments, 3 sets of 50 consecutive non-isothermal experiments at flowrates of 25, 99, and 414 cm$^3$/s were conducted. The individual limiting current sets and their respective averages are shown in Figures 5.2.1 to 5.2.6. Averages and deviation from the averages were calculated for the limiting current for each of the 3 isothermal and 3 non-isothermal sets.

4.2.7 Isothermal Mass Transfer Experiments

A typical datum point was obtained, in the same manner as described in Section 4.2.5.2. While the bulk solution temperature ($T_s$) was maintained at the desired level, a potential step was applied to the system, and the steady state limiting current measured. Two typical potential step limiting current curves for the isothermal experiments 11 and 126 are shown in Figure 4.2.7.1. For low flow rates, the steady state limiting current remained fairly constant, enabling accurate measurements to be made directly with the digital voltmeter. But at higher flow rates, the limiting current fluctuated up to ±4% about the mean value,
Experiment I1

Re = 266
E = 16.33 mV, Ω 1.238 Ohms
I (lim.) = 14.81 mA

Experiment I26

Re = 28570
E = 221.38 mV, Ω 1.238 Ohms
I (lim.) = 178.82 mA

FIGURE 4.2.7.1 Typical Limiting Current Potential Step Curves (Example: Isothermal Experiment I1 and I26)
which made it impossible to use the digital voltmeter for accurate readings. At these flow rates the strip chart recorder could have been used to approximate an average limiting current by drawing a line through the middle of the minimum and maximum values. This method would have been cumbersome, inaccurate and in addition susceptible to data entry errors. Hence, for the isothermal (and non-isothermal) experiments at all bulk flow rates the ‘voltage drop’ data acquisition system was utilized to obtain the average limiting current values (see sections 4.1 and 4.2.5.2). During the early stages of the experimental work after every seven experiments or at least once every day, the bulk solution concentration was determined as described in Section 4.2.3. It was found that the bulk solution concentration did not change over the course of seven experimental runs ('Data Summary' (see Appendix L)). Hence as of experiment 1338 the frequency of bulk solution analysis was decreased to every 21 experiments or at least one every day. The electrodes were cleaned at least after every ten experiments according to the procedure outlined in Section 4.2.4. Experimental conditions for the isothermal mass transfer investigations employed cathode angles (α) of 0, 30, 60, 90, 120, 150 and 180 degrees, channel slopes (β) of 0, 30, 60 and 90 degrees; and flow rates at 21 different settings which corresponded to Reynolds numbers varying between 200 and 32000. The isothermal experiments were performed at four different levels of bulk solution temperatures (T₁) of approximately 25, 35, 45 and 55°C (measured precisely to ±0.05°C).

4.2.8 Non-Isothermal Mass Transfer Experiments

The limiting current for the non-isothermal case was established and measured in the same manner as described in Section 4.2.5.2. For a given flow rate, the cartridge heater voltage was adjusted until the desired steady state cathode surface temperature, T₂, was reached. All non-isothermal experiments were conducted at a bulk solution temperature, T₁, of about 25°C. The temperatures, T₁ to T₄, and the reservoir temperature (for check of immersion heater function) were obtained through the data acquisition system and then displayed on the computer monitor. After reaching steady state temperature at the cathode surface, a known potential (E = 800 mV) as required to achieve limiting current was applied to the electrochemical system and the steady state limiting current value established. All experimental parameters
were added to a data file in the same way described in Section 4.2.5.2. As described in Section 4.2.7 the bulk solution concentration was quite frequently determined as described in Section 4.2.3. After the non-isothermal experiment N189 the frequency of bulk solution analysis was decreased to every 21 experiments or at least one every day. The electrodes were cleaned at least after every ten experiments according to the procedure outlined in Section 4.2.4.

The experimental conditions for the non-isothermal mass transfer covered cathode angles (α) of 0, 30, 60, 90, 120, 150 and 180 degrees, channel slopes (β) of 0, 30, 60 and 90 degrees; and flow rates at 21 different settings which corresponded to Reynolds numbers varying between 200 and 32000. The non-isothermal experiments were performed at a bulk solution temperature (T₁) of approximately 25°C (measured precisely to ±0.05°C) and with the cathode surface temperature, T₂, set to 35, 45 and 55°C (measured precisely to ±0.05°C).
CHAPTER FIVE
EXPERIMENTAL RESULTS AND DISCUSSION

In order to minimize the possibilities of the introduction of data analysis errors all raw data were entered in Lotus-123 spread sheets. The formulas required for the individual quantities and dimensionless numbers involved, were developed for one experiment and then copied for all the others. These quantities and dimensionless groups were derived from the equations in Chapter 2 and Appendix C and then converted into SI units. The isothermal 'Data Summary' includes (see Appendix L):

- Experiment Number:
- Rotameter reading, [size, %]: assigned reading
- Volumetric flow rate, [m³/s]: eq. B.1, B.2, or B.3 (depends on rotameter)
- Bulk velocity, [m/s]: eq. C.15
- Voltage drop across R = 1.238Ω, [mV]: measured
- Limiting current, [mA]: eq. C.4
- Bulk solution temperature, Tᵣ, [°C]: measured
- Caustic soda bulk concentration, [mol/l]: measured
- Potassium ferricyanide bulk conc., [mol/l]: measured
- Potassium ferrocyanide bulk conc., [mol/l]: measured
- Density, [kg/m³]: eq. C.10
- Viscosity, [m²/s]: eq. C.11
- Diffusivity, [m²/s]: eq. C.12
- Cathode angle, [°]: measured
- Channel slope, [°]: measured
- Reynolds number, [-]: eq. C.16
- Schmidt number, [-]: eq. C.17
- Average mass transfer coefficient, [m/s]: eq. 2.3.9
- Sherwood number, [-]: eq. C.19
- Date experiment performed, [DD/MM/YY]: Date
- Exp. when electrodes were cleaned: assigned
- Exp. when electrolyte comp. was determined: assigned

All isothermal results, which will be discussed in the succeeding sections, were obtained using the
information contained in these spread sheets and printed out in the 'Data Summary' (see Appendix L).

Similarly the non-isothermal results and correlations, which will be discussed in the following sections, were compiled in spread sheets and printed out in Tables. The non-isothermal 'Data Summary' includes:

- Experiment Number: assigned
- Rotameter reading, [size, %]: reading
- Volumetric flow rate, [m³/s]: eq. B.1, B.2, or B.3 (depends on rotameter)
- Bulk velocity, [m/s]: eq. C.15
- Voltage drop across R = 1.238Ω, [mV]: measured
- Limiting current, [mA]: eq. C.4
- Temperature, T₁, [°C]: measured
- Temperature, T₂, [°C]: measured
- Temperature, T₃, [°C]: measured
- Temperature, T₄, [°C]: measured
- Surface temperature, [°C]: eq. C.5
- Film temperature, [°C]: eq. C.7
- Cathode heater voltage, [V]: measured
- Cathode heater amperage, [A]: measured
- Caustic soda bulk concentration, [mol/l]: measured
- Potassium ferricyanide bulk conc., [mol/l]: measured
- Potassium ferrocyanide bulk conc., [mol/l]: measured
- Bulk density, [kg/m³]: eq. C.10
- Density at film conditions, [kg/m³]: eq. C.10
- Density at surface conditions, [kg/m³]: eq. C.10
- Viscosity at film conditions, [m²/s]: eq. C.11
- Diffusivity at film conditions, [m²/s]: eq. C.12
- Thermal conductivity, [J/(m·s·K)]: eq. C.13
- Heat capacity, [J/(kg·K)]: eq. C.14
- Cathode angle, [°]: measured
- Channel slope, [°]: measures
- Reynolds number, [-]: eq. C.16
- Schmidt number, [-]: eq. C.17
- Prandtl number, [-]: eq. C.18
- Grashof number for heat transfer, [-]: eq. C.23
- Grashof number for mass transfer, [-]: eq. C.21
- Combined Grashof number for mass transfer, [-]: eq. C.24
- Average mass transfer coefficient, [m/s]: eq. 2.3.9
- Sherwood number, [-]: eq. C.19
- Date experiment performed, [DD/MM/YY]: Date
- Exp. when electrodes were cleaned: assigned
- Exp. when electrolyte comp. was determined: assigned
5.1 PRELIMINARY EXPERIMENTS

The experimental data for the 336 preliminary isothermal experiments are fully tabulated in the 'Data Summary' and denoted there as IP1 to IP336. All derived quantities e.g. volumetric flow rate, Reynolds number, average mass transfer coefficient, and Sherwood number, are also listed.

The overall objective of these experiments was to ensure that desired experimental conditions could be achieved and maintained, and that adequate skills in experimental technique were developed. It was necessary also to determine that reasonable agreement with any previous work under similar conditions, could be attained. All of these objectives were achieved. For comparison to previous work, correlations of the data in terms of Sh as a function of (Re Sc d, /L) were made. These agreed satisfactorily with existing correlations.

It was clear that experimental manipulation and operational skills gradually developed in reliability through the series of runs. However, it was decided not to include this early work in the final results, which were based on the higher precision that practice had made possible. The same observations apply to the 504 preliminary experiments under non-isothermal conditions. In both cases the data are available in the 'Data Summary' (see Appendix L) for the purpose of comparison.

5.2 REPLICATE EXPERIMENTS

The isothermal experimental data for each of the 3 sets (3 different volumetric flow rates) of experiments provide the limiting current in mA. Each experiment had been arranged for independent settings of the applied potential and the flow rate. The experimental data for the replicate isothermal experiments are fully tabulated in the 'Data Summary' (see Appendix L). The individual isothermal limiting current readings and their averages for bulk flowrates of 414 cm³/s, 25 cm³/s, and 99 cm³/s are shown in Figures 5.2.1 to 5.2.3, respectively. Examination of these limiting current data sets indicates that the average deviation from their respective averages for every data set is about 4%. This observation holds also for the set of the 100 isothermal consecutive experiments, which were conducted in the transition flow region. The reasons for the observed deviations are:
FIGURE 5.23 Replicate Exp. (Flowrate = 99 cm$^3$/s)

- Average

Isenthalpic Experiment Number

54 53 52 51 50 49 48

Limine Current [mA]
i. a small effect attributed to the slight variations in bulk solution temperature

ii. the accuracy of the limiting current reading being about ±0.1 mA

iii. the flow rate settings being of limited accuracy and the flow always showing some slight unsteadiness

iv. the electrode potential being set quite precisely, and with the limiting current plateau being fairly wide, the effect of this factor could be neglected.

It is evident that, over the flow range under investigation, there is a high degree of reproducibility with appropriately set random conditions, and for repeats on different days.

Figures 5.2.4 to 5.2.6 depict the individual non-isothermal limiting current readings and their averages for bulk flow rates of 25 cm$^3$/s, 414 cm$^3$/s, and 99 cm$^3$/s, respectively. It is apparent that the average deviation from their respective averages is about 4 to 5%. The reasons for the deviation, in addition to the ones mentioned above, are:

i. the cathode surface temperature control, using an auto-transformer instead of an expensive temperature control circuit, was accurate within approximately ±0.2°C

ii. the actual surface temperature measurement was an indirect one, due to the fact that the thermocouple was located about 1 mm from the actual cathode surface with the 'thermal contact' between the thermocouple tip and the copper achieved by means of heat conducting paste

Again there is a high degree of reproducibility for appropriately set random non-isothermal conditions. The data are available in the 'Data Summary' (see Appendix L) for the purpose of comparison.

5.3 ISOThERMAL EXPERIMENTS

The experimental data for the 3277 isothermal experiments are fully tabulated in the 'Data Summary' (see Appendix L) and denoted there as I1 to IP3277. All derived quantities e.g. volumetric flow rate, Reynolds number, average mass transfer coefficient, and Sherwood number, are also listed.

Conforming to the classical presentation of mass transfer data in terms of dimensionless groups,
FIGURE 5.24 Replicate Exp. (Flowrate = 25 cm³/s)

Limiting current (mA)
FIGURE 5.2.5 Replicate Exp. (Flowrate = 414 cm$^3$/s)
all isothermal experimental data were plotted as Sherwood numbers vs. Reynolds numbers or as Sherwood numbers against the functional grouping (Re Sc d_f /L). As noted by Wranglen and Nilsson (1962) and Pickett (1977), there is a clear change in slope for such plates corresponding to the transition from the laminar to the turbulent flow regime. From the present study for one series of experiments the observed Sherwood numbers are plotted against the Reynolds numbers in Figure 5.3.1.1. These data confirm the observations of Wranglen and Nilsson (1962) and Pickett (1977). The transition region in the present study was found to occur at Re = 1900. The data were therefore separated into groups:

Laminar flow - Re ≤ 1900
Transition/turbulent flow - Re > 1900.

5.3.1 Laminar Flow Results

Experimental data were obtained using a constant flow channel aspect ratio (height/width), γ, (γ = 0.75), (d_f /L) = 0.114, and a temperature imposed change in Schmidt numbers (810 ≤ Sc ≤ 2350). The isothermal data for horizontal (α = 0°, β = 0°) and vertical flow (β = 90°) were used to validate the experimental technique. To illustrate the influence of change in cathode angle (α) on the overall mass transfer 112 typical data points for 0° ≤ α ≤ 180° and β = 0° are given in Table 5.3.1.1.

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Several equations have been proposed for predicting the mass transfer coefficients between a channel wall and laminar flow with a fully developed hydrodynamic profile under isothermal conditions. These have been summarized in section 3.1 above. For the conditions $\alpha = 0^\circ$ and $\beta = 0^\circ$ the overall laminar data were correlated by the general dimensionless Lévêque equation (equation 3.1.1) format:

$$Sh = a_i \left( \frac{Re Sc}{d_x L} \right)^{\gamma_i} \tag{5.3.1.1}$$

For application to other values of the cathode angle ($\alpha$) and the channel slope ($\beta$) it is now proposed that the general dimensionless correlation 5.3.1.1 should be given the form of:

$$Sh = a_i \left( 1 + a_2 (\sin \alpha)^{\gamma_2} + a_4 (\sin \beta)^{\gamma_4} \right) \left( \frac{Re Sc}{d_x L} \right)^{\gamma_i} \tag{5.3.1.2}$$

This has been applied to all isothermal data except those for $\beta = 90^\circ$ (the vertical flow position). The confidence intervals of the regression coefficients and the standard error estimates were calculated using the non-linear regression unit of the SYSTAT 4.0 by SYSTAT Inc., Evanston, IL., U.S.A. If the aspect ratio ($\gamma = 0.75 \rightarrow$ after Roušar (1971) $\rightarrow \chi = 0.8487$) of the channel is taken into account, the 95% confidence intervals of the regression coefficients are $a_i = 1.387 \pm 0.044$, $a_2 = 0.595 \pm 0.027$, $a_3 = 2.231 \pm 0.0538$, $a_4 = 0.342 \pm 0.013$, $a_5 = 0.942 \pm 0.029$, and $a_6 = 0.347 \pm 0.017$. Hence the isothermal laminar results ($Re \leq 1900$) for $0^\circ \leq \alpha \leq 180^\circ$ and $0^\circ \leq \beta \leq 60^\circ$ were best correlated by:

$$Sh = 1.39 (1 + 0.60 (\sin \alpha)^{2.23} + 0.34 (\sin \beta)^{0.94}) (Re Sc d_x / L)^{0.33} \tag{5.3.1.3}$$
FIGURE 5.3.1.1 Comparison of Isothermal Experimental Results to known Correlations
When $\beta = 90^\circ$ the flow channel is vertical and hence the angle of rotation ($\alpha$) does not have any effect. Therefore the data were correlated in terms of the traditional parameters for mass transfer at a vertical plate, with the best fit as given in equation (5.3.1.4):

$$Sh = (1.862 \pm 0.024)(Re \ Sc \ d_e / L)^{0.44 \pm 0.021}$$  \hspace{1cm} (5.3.1.4a)

For practical purposes, the equation 5.3.1.4a can be given as:

$$Sh = 1.86(Re \ Sc \ d_e / L)^{0.35}$$  \hspace{1cm} (5.3.1.4b)

A comparison to the Roušar (1971) mass transfer correlation (equation 3.1.2) for laminar flow is shown in Figure 5.3.1.1. This modified Lévêque equation is described by Pickett (1977) to be a good design equation for isothermal laminar mass transfer in a rectangular flow channel. Two sample cases with $\alpha = 0^\circ$, $\beta = 0^\circ$, $Sc = 1800$, $(d_e / L) = 0.1142$, and $\chi = 0.8487$ are shown in Table 5.3.1.2. The predicted Sherwood numbers using equation 5.3.1.3 agree with the predicted Sherwood numbers of the modified Lévêque equation 3.1.2 to within 6%. Considering the possible errors involved in the estimation of the physical property data and the general limitations of the experimental technique (see Appendix J for the estimation of all the experimental errors), the agreement is considered satisfactory, and the experimental technique used in this study to be valid.

Figure 5.3.1.2 shows the effect of the change in cathode angle ($\alpha$) on the mass transfer in a horizontal flow channel ($\beta = 0^\circ$). A 60% increase in mass transfer is shown when the flow channel is reconfigured from the 25/25/0/0 position to the 25/25/90/0 one.

Normalized Sherwood numbers as a function of the cathode angle ($\alpha$) were plotted in Figure 5.3.1.3. To normalize the Sherwood numbers for isothermal laminar mass transfer, equation 5.3.1.3 was divided by equation 5.3.1.3 for $\alpha = 0^\circ$, $\beta = 0^\circ$. The resulting function thus became independent of the Reynolds number, Schmidt number and the dimensionless length $(d_e / L)$ with the only dependent variable
being the angle $\alpha$. Figure 5.3.1.3 is valid for isothermal laminar mass transfer at $0^\circ \leq \alpha \leq 180^\circ$, $\beta = 0^\circ$, $200 \leq \text{Re} \leq 1900$, $\text{Sc} = 2000$, and $(d_i/L) = 0.114$ and is illustrative of the overall effect of rotation of a mass transfer surface in a horizontal plane.

| Table 5.3.1.2 Comparison of isothermal laminar Results to Roušar's equation (3.1.2) |
|---------------------------------|---------------------------------|---------------------------------|---------------------------------|
| $\text{Re}$ | $\text{Sh predicted by}$ (equation 3.1.2) | $\text{Sh predicted by}$ (equation 5.3.1.3) | $\text{Sh - deviation [%]}$ vs. equation 3.2.1 |
| $\phantom{0}$ | $\text{Sh predicted by}$ Roušar ($\chi = 0.8487$) | $\phantom{0}$ |  |
| $\phantom{0}$ | 54.2 | 55.4 | $+2.2$ |
| $\phantom{0}$ | 110.5 | 116.3 | $+5.4$ |

Examination of Figures 5.3.1.2 and 5.3.1.3 indicates no observable differences in mass transfer for $\alpha = 0^\circ$, $\beta = 0^\circ$ (cathode facing upwards) and $\alpha = 180^\circ$, $\beta = 0^\circ$ (cathode facing downwards). It was expected to see a difference in mass transfer due to the density difference between the bulk solution and the "cathode surface" solution. These should have caused some instabilities in the boundary layer while the cathode was operated in the downward facing position ($\alpha = 180^\circ$, $\beta = 0^\circ$), with a consequent enhancement in the rate of mass transfer. The data of the present study are not sufficiently definitive to provide an answer to the question of instabilities. To answer this question flow visualizations will have to be conducted in future studies.

The influence of the angle of elevation is shown in Figures 5.3.1.4 and 5.3.1.5. Figure 5.3.1.4 shows experimental data for the isothermal laminar mass transfer at a constant cathode angle ($\alpha = 0^\circ$) at different channel slopes. There is an 34% increase in the mass transfer as the channel is elevated from the horizontal ($25/25/0/0$) to the vertical ($25/25/0/90$) position. Normalized Sherwood numbers were calculated by dividing equations 5.3.1.3 and 5.3.1.4 by equations 5.3.1.3 ($\alpha = 0^\circ$, $\beta = 0^\circ$) and 5.3.1.4 ($\alpha = 0^\circ$, $\beta = 0^\circ$), respectively. The influence of the channel slope ($\beta$) on the isothermal laminar mass
FIGURE 5.3.1.2 Isothermal Mass Transfer in Horizontal Flow with Rotation of M.T. Surface
FIGURE 5.3.1.3 Isothermal Mass Transfer with Rotation of the M.T. Surface (Eq 5.3.1.3)
FIGURE 5.3.1.4 Isothermal Mass Transfer with Elevation of the M.T. Surface
$200 \leq \text{Re} \leq 1900$

$\text{Sc} \approx 2000$

$\alpha = 0^\circ$

$d_e/L = 0.114$

**FIGURE 5.3.15** Isothermal Mass Transfer with Elevation of the M.T. Surface (Eq 5.3.1.3, 5.3.1.4)
FIGURE 5.3.16 Isothermal Laminar M.T. (Eq. 5.3.1.3)
Compared to M.T. at (a=0°, β=0°)
transfer at $\alpha = 0^\circ$, $0^\circ \leq \beta \leq 90^\circ$, $200 \leq \text{Re} \leq 1900$, $\text{Sc} = 2000$, and $(d, L) = 0.114$ is shown in Figure 5.3.1.5. Examination of Figure 5.3.1.5 shows that the Sherwood number increases approximately 1.3 times when the channel is elevated from the horizontal (cathode facing upward position) to a slope of $60^\circ$. However only a very small increase in mass transfer (7%) was found when the channel was elevated from the $60^\circ$ slope to the vertical position.

Following a similar procedure to the one given above, normalized Sherwood numbers were calculated by dividing equation 5.3.1.3 by equation 5.3.1.3 ($\alpha = 0^\circ$, $\beta = 0^\circ$). The influence of the cathode angle ($0^\circ \leq \alpha \leq 180^\circ$) and the channel slope ($0^\circ \leq \beta \leq 60^\circ$) on the isothermal laminar mass transfer is shown in Figure 5.3.1.6. A 90% increase of mass transfer over the horizontal case ($\alpha = 0^\circ$, $\beta = 0^\circ$) was observed at $\alpha = 90^\circ$ and $\beta = 60^\circ$ (see Figure 5.3.1.6), whereas only a 34% increase was found when the channel was re-positioned from the horizontal ($\alpha = 0^\circ$, $\beta = 0^\circ$) to the vertical ($\beta = 90^\circ$) position (see Figure 5.3.1.5). The incremental increase in cathode angle ($0^\circ$, $30^\circ$, $60^\circ$, $90^\circ$, $120^\circ$, $150^\circ$ and $180^\circ$) and channel slope ($0^\circ$, $30^\circ$, $60^\circ$ and $90^\circ$) limited the amount of information available to predict mass transfer coefficients at different cathode angles ($\alpha$) and channel slopes ($\beta$). Therefore no predictions in the development of the isothermal laminar mass transfer for $0^\circ \leq \alpha \leq 180^\circ$ and $60^\circ > \beta < 90^\circ$ can be made.

The laminar and turbulent mass transfer data for the 3277 isothermal experiments (Sherwood number vs. Reynolds number) are presented in Figures E.1 to E.21 in Appendix E.

5.3.2 Transition - Turbulent Flow Results

Following similar procedures to those given in the above section, the experimental data for the transition / turbulent flow regime were fitted to the equation:

$$Sh = a_1 \left(1 + a_2 \left(\sin \alpha \right)^{a_3} + a_4 \left(\sin \beta \right)^{a_5}\right) \text{Re}^{a_6} \cdot \text{Sc}^{a_7} \left(\frac{d}{L}\right)^{a_8}$$

(5.3.2.1)

As noted in section 3.2, Pickett and Stanmore (1972) examined the existing isothermal turbulent mass transfer data for parallel plate reactors and proposed a deviation from the conventional mass transfer
correlation format which included the term \((d_v/L)^{1/4}\). They suggested that isothermal turbulent mass transfer correlations should include in the dimensionless group \((d_v/L)\) to the 1/4 power (see equation 3.2.5). Following their recommendation, the functional group \((d_v/L)^{1/4}\) has been introduced in the isothermal turbulent mass transfer correlations obtained in this study (i.e. equation 5.3.2.2, 5.3.2.3a and 5.3.2.3b).

The confidence intervals of the regression coefficients and the standard error estimates were calculated using the non-linear regression unit of the SYSTAT™ 4.0. The 95% confidence intervals of the regression coefficients are \(a_1 = 0.102\pm 0.004\), \(a_2 = 0.519\pm 0.015\), \(a_3 = 3.182\pm 0.060\), \(a_4 = 0.471\pm 0.012\), \(a_5 = 0.807\pm 0.027\), \(a_6 = 0.683\pm 0.023\), and \(a_7 = 0.321\pm 0.011\). In conventional form the results for the transition / turbulent isothermal results \((Re > 1900)\) can be written for \(0^\circ \leq \alpha \leq 180^\circ\) and \(0^\circ \leq \beta \leq 60^\circ\) as:

\[
Sh = 0.102 \left(1 + 0.52 \left(\sin\alpha\right)^{0.18} + 0.47 \left(\sin\beta\right)^{0.34}\right) Re^{0.65} Sc^{0.32} \left(d_v/L\right)^{1/4} \tag{5.3.2.2}
\]

For vertical flows \((\beta = 90^\circ)\), the experimental data were best correlated by:

\[
Sh = (0.152\pm 0.006) Re^{0.671\pm 0.003} Sc^{0.32\pm 0.015} \left(d_v/L\right)^{1/4} \tag{5.3.2.3a}
\]

For practical purposes, the above equation (5.3.2.3a) can be given as:

\[
Sh = 0.15 Re^{0.67} Sc^{0.33} \left(d_v/L\right)^{1/4} \tag{5.3.2.3b}
\]

The exponents of the Reynolds and Schmidt numbers' respectively are almost exactly 2/3 and 1/3. These are the values commonly found in the literature for isothermal turbulent forced convective mass transfer. For comparison the correlation (equation 3.2.5) given by Pickett (1977) for turbulent forced convective mass transfer in vertical channels is included in Figure 5.3.1.1. Results reported here are
slightly higher (averaging 8%). This discrepancy could result from the fact that Pickett (1977) examined all available data from a variety of investigations with different experimental parameters, whereas the correlation presented in this study is the result of a single system under investigation. Considering the fact that the experimental conditions were not exactly the same (e.g. different electrolyte, aspect ratio, and electrode length), a rigorous comparison to equation 3.2.5 cannot be made. Nonetheless the correlation equation has a similar form, and considering the above factors, the agreement is reasonable.

The influence of the Reynolds number on the turbulent mass transfer is shown in Figure 5.3.1.2. With increasing bulk velocities the Sherwood number also increases. Under isothermal turbulent flow conditions in a horizontal channel the mass transfer was increased by a factor of 1.5 when the cathode was re-positioned from the horizontal upwards facing arrangement \((\alpha = 0^\circ, \beta = 90^\circ)\) to the \(\alpha = 0^\circ, \beta = 90^\circ\) position. Again following the approach of normalization (equation 5.3.2.2), Figure 5.3.2.1 shows the dependence of the Sherwood number on the cathode angle for the range of \(0^\circ \leq \alpha \leq 180^\circ, \beta = 0^\circ, 1900 < \text{Re} \leq 32000, \text{Sc} = 2000, \) and \((d_e/L) = 0.114\). The maximum mass transfer under isothermal turbulent conditions \((\beta = 0^\circ)\) occurs at a cathode rotational angle of 90°.

With increasing channel slope, \(\beta\), mass transfer increases up to a maximum at \(\beta = 90^\circ\) (Figure 5.3.1.4). Figure 5.3.2.2 illustrates the influence of the channel elevation on the turbulent isothermal mass transfer for \(\alpha = 0^\circ, 0^\circ \leq \beta \leq 90^\circ, 1900 < \text{Re} \leq 32000, \text{Sc} = 2000, \) and \((d_e/L) = 0.114\). The maximum increase in mass transfer (≈ 42%) will be observed if the channel slope \(\beta\) is varied from the horizontal upward facing position \((\alpha = 0^\circ, \beta = 0^\circ)\) to the vertical one \((\alpha = 0^\circ, \beta = 90^\circ)\).

Upon examination of all isothermal experimental data (Appendix E; Figures E.1 to E.21), it can be concluded that under the experimental conditions (cathode angle and channel slope incremented in 30° steps) of this study the largest increase in isothermal (laminar and turbulent) mass transfer over the horizontal case \((\alpha = 0^\circ, \beta = 0^\circ)\) can be achieved by positioning the flow channel at an angle of \(\alpha = 90^\circ, \beta = 60^\circ\).

For the limited arrangement of horizontal laminar flow and vertical turbulent flow the experimental data substantiate the correlations presented respectively by Roušar (1971) and Pickett (1977).
1900 < Re ≤ 35000
Sc ≈ 2000
β = 0°
d_{c}/L = 0.114

FIGURE 5.3.2.1 Isothermal Mass Transfer with Rotation of the M.T. Surface (Eq. 5.3.2.2)
FIGURE 5.3.2.2 Isothermal Mass Transfer with Elevation of the M.T. Surface (Eq 5.3.2.2, 5.3.2.3)
as has been noted before (Figure 5.3.1.1). The Reynolds and Schmidt numbers exponents in the
correlations for turbulent flow conditions (equations 5.3.2.2 and 5.3.2.3) have essentially the values 2/3
and 1/3, respectively. No other data or correlations have been found in the literature to compare to the
isothermal correlations at various cathode angles and channel slopes, as obtained in this study. From the
extensive work reported here, covering full rotation of the flow channel and the full inclination range from
horizontal to vertical, the general equations advanced for laminar flow (equations 5.3.1.3 and 5.3.1.4) and
for turbulent flow (equations 5.3.2.2 and 5.3.2.3) will be appropriate design equations for electrochemical
systems or any solid-liquid mass transfer process with comparable configuration.

Following similar procedures to those given in the above section, normalized Sherwood numbers
were calculated by dividing equation 5.3.2.2 by equation 5.3.2.2 (\(\alpha = 0^\circ, \beta = 0^\circ\)). The influence of the
cathode angle (\(0^\circ \leq \alpha \leq 180^\circ\)) and the channel slope (\(0^\circ \leq \beta \leq 60^\circ\)) on the isothermal turbulent mass
transfer is shown in Figure 5.3.2.3. Again the maximum increase in mass transfer (= 93%) over the
isothermal turbulent case (\(\alpha = 0^\circ, \beta = 0^\circ\)) can be expected at a flow channel position of \(\alpha = 90^\circ\) and
\(\beta = 60^\circ\). As stated in section 5.3.1, no predictions in the development of the isothermal turbulent mass
transfer for \(0^\circ \leq \alpha \leq 180^\circ\) and \(60^\circ > \beta < 90^\circ\) can be made.

Finally, to show the adequacy of these isothermal laminar and turbulent mass transfer correlations
(equation 5.3.1.3, 5.3.1.4, 5.3.2.2 and 5.3.2.3), the percent deviations of the predicted Sherwood numbers
from the experimental values are plotted in Figure 5.3.2.4 for all 3277 laminar and turbulent experiments.
The percent deviation of the experimental Sherwood number from the predicted Sherwood number
(equation 5.3.1.3, 5.3.1.4, 5.3.2.2 and 5.3.2.3) is approximately ±4%. Not unexpectedly the developing
experimental technique, as in the last 2000 or so measurements, yields a similar average deviation of less
than 3%.
FIGURE 5.3.2.3 Isothermal Turbulent M.T. (Eq. 5.3.2.2)
Compared to M.T. at (\(\alpha=0^\circ\), \(\beta=0^\circ\))
FIGURE 5.3.2.4 Goodness of Isothermal Data Fit
5.4 NON-ISOTHERMAL EXPERIMENTS

5.4.1 Thermal Boundary Layers

When fluids flow past immersed bodies, such as plates, heat or matter is often transferred at the boundary between the solid and the fluid. This transfer must take place through the hydrodynamic boundary layer which forms on the immersed object. For heat transfer to take place, the temperature of the immersed body must be different from that of the flowing fluid. In such a case, as the fluid flows past the immersed solid, a thermal boundary layer, as well as a hydrodynamic boundary layer, forms. In the thermal boundary layer the fluid temperature at the solid surface equals that of the surface. On moving away from the surface, the fluid temperature changes and becomes equal to the temperature of the undisturbed stream at the edge of the thermal boundary layer. The thickness of the thermal boundary layer may be different from that of the hydrodynamical boundary layer, and it starts at the point where the heat transfer starts (Figure 5.4.1.1). For the case of mass transfer, the concentration boundary layer will have similar attributes but is not identical.

The case of heat transfer from an isothermal flat plate was early studied analytically by Pohlhausen (1921), who solved the energy equation to obtain the temperature distribution in the boundary layer as well as the local heat transfer coefficient on the plate. The momentum and continuity equations had been solved by Blasius (1908), and Pohlhausen used Blasius' solution to solve the energy equation.

The general boundary layer relationships, including concentration boundary layers have been reviewed by Fahidy (1985). The hydrodynamic local boundary layer thickness, \( \delta_{\text{h}} \), derived by the boundary layer theory for laminar forced convective flow past a flat plate electrode can be given (Fahidy, 1985) as:

\[
\delta_{\text{h}} = 5.2 \left( \frac{v}{U} \right)^{1/2} \sqrt{x}
\]  

(5.4.1.1)

Let the velocity distribution in the hydrodynamic boundary layer be \( U_i(x,y) \) and \( U_j(x,y) \). The concentration distribution in the laminar concentration boundary layer is then given by Fick's second law:
\[ U \frac{\partial c}{\partial x} + U \frac{\partial c}{\partial y} = D \frac{\partial^2 c}{\partial y^2} \]  

(5.4.1.2)

The above equation may be solved by a rather elaborate technique based on similarity variables. This solution will lead to the local concentration boundary layer thickness, \( \delta_c \):

\[ \delta_c = 2.95 \left( \frac{D}{V} \right)^{0.33} \left( \frac{V}{U} \right)^{0.5} \sqrt{\chi} \]  

(5.4.1.3)

In a similar way the thermal boundary layer thickness can be derived which will lead to:

\[ \delta_t = 2.95 \left( \frac{\alpha_t}{V} \right)^{0.33} \left( \frac{V}{U} \right)^{0.5} \sqrt{\chi} \]  

(5.4.1.4)

Thus, the boundary layer thicknesses are parabolic functions of the distance. The above equation can be used to estimate the thermal boundary layer thickness. For a bulk solution velocity of 2 cm/s, a kinematic viscosity of 1.005 \cdot 10^{-3} \text{ cm}^2/\text{s}, and a thermal diffusivity of 1.57 \cdot 10^{-3} \text{ cm}^2/\text{s} equation 5.4.1.4 predicts, according to Figure 5.4.1.1, the thermal boundary layer thickness for the non-isothermal experiment, N117, to be less than 0.5 cm or about 30% of the electrochemical reactor height. As the bulk flow velocity increases the boundary layer thickness will decrease. Following the same procedure, it can be shown that the concentration boundary layer for the same case is approximately twice as thick.

The average values of the thermal and concentration boundary layer can be obtained by integrating the local values over the entire length under investigation. Finally it can be shown that the relative magnitude of the concentration boundary layer in comparison to the hydrodynamic boundary layer is independent of the flow velocity and the electrode geometry, and depends entirely on the physical characteristics of the electrolyte (equation 5.4.1.5).

\[ \frac{\bar{\delta}_c}{\delta_c} \equiv 0.6 \left( \frac{D}{V} \right)^{0.33} \]  

(5.4.1.5)
The non-isothermal experimental conditions involve:

i. a fully developed hydrodynamic boundary layer, due to the flow entrance length of 127 cm (= 74 equivalent diameters)

ii. a developing thermal boundary layer, starting from zero at the leading edge of the cathode and increasing over the length of the cathode to about 0.5 cm

iii. a developing concentration boundary layer, starting from zero at the leading edge of the cathode and increasing over the length of the cathode to about 1 cm

According to equations 5.4.1.3 and 5.4.1.4 with increasing bulk solution velocity the boundary layer thickness will decrease. The thermal and concentration boundary layers will not reach the opposite wall of the flow channel under the experimental conditions of this study. Hence, it can be assumed that the transport phenomena observed are not being influenced by any boundary layer deformation.

5.4.2 Laminar Flow Results

The experimental raw data and their derived quantities for all of the 2394 non-isothermal laminar and turbulent experiments are fully tabulated in the 'Data Summary', and denoted there as N1 to N2394.

The Sherwood numbers calculated for the +10°C, +20°C and +30°C temperature difference, between the cathode surface and bulk solution, are shown in Figure 5.4.2.1. For low Reynolds numbers (Re < ~1900) the Sherwood numbers appear to be independent of the flow conditions, in contrast to the isothermal measurements, and thus data measured at Re ≤ 1900 can be considered laminar. Turbulent flow conditions were said to occur at Reynolds numbers larger than 1900. For comparison two additional data sets are included in Figure 5.4.2.1:

i. the isothermal laminar and turbulent results for the 25/25/0/0 case obtained in this study

ii. Ajersch's (1990) non-isothermal laminar and turbulent results for the 25/35/0/0 case.

Upon closer examination of Figure 5.4.2.1, a few conclusions can be drawn. Since the Sherwood numbers remain constant for a given temperature gradient, at Reynolds numbers less then 1900 the flow conditions seem to have no influence on the transfer of mass. With increasing temperature gradients the
FIGURE 5.4.2.1 M.T. with and without Temperature Gradient at the Horizontal M.T. Surface
Sherwood numbers became significantly larger. Hence, the imposed temperature gradients caused free convection 'mixing' effects which increased the mass transfer. At larger Reynolds numbers (Re > 1900), forced convection became dominant, and mass transfer started to follow the isothermal forced convection behaviour, although at higher levels. Ajersch's (1990) experimental data were adjusted to account for the differences in the aspect ratio and electrode width using the aspect ratio factor estimation presented by Roušar (1977). The aspect ratio factors were for Ajersch (χ = 0.9463) and in this study (χ = 0.8487). Hence Ajersch's results were multiplied by the factor of (0.8487/0.9463) to compensate for the differences in aspect ratios. The Sherwood numbers obtained for the 25/35/0/0 case compare well with the experimental data of Ajersch's work (1990). The differences between the non-isothermal laminar results of Ajersch (1990) and the results obtained in this study are about 10%, whereas the differences for the turbulent regime are not significant (=3 to 5%). The differences in the non-isothermal laminar flow regime can be attributed to:

i. different electrolytes being employed in these studies, with correspondingly different buoyancy forces

ii. different estimation of the cathode surface temperature (temperature measurement)

iii. differences in the apparatus design: smoothness of the transition between the Plexiglas™ flow channel and the cathode can cause unwanted turbulences and influence the boundary layer development

As discussed above, the experimental data revealed that the mass transfer in the laminar flow regime, at Re ≤ 1900, seems to be independent of the Reynolds number. Hence it was decided to correlate the data for the cathode angle range of 0° ≤ α ≤ 180° and the channel slope range of 0° ≤ B ≤ 60° in terms of free convection alone, yielding for non-isothermal laminar flow conditions, an equation of the form:

\[
Sh = (0.116 \pm 0.011)(1 + (0.336 \pm 0.019)(\sin \alpha)^{0.266 \pm 0.018} + (0.427 \pm 0.027)(\sin B)^{0.226 \pm 0.012})(GR_{\infty} Se)^{0.324 \pm 0.011}
\]

(5.4.2.1a)
For practical purposes equation 5.4.2.1a can be written as:

$$\text{Sh} = 0.116(1 + 0.34(\sin \alpha)^{2.77} + 0.43(\sin \beta)^{0.83})(GR_m \text{ Sc})^{0.32}$$ \hspace{1cm} (5.4.2.1b)

When $\beta = 90^\circ$ the flow channel is vertical and hence the angle of rotation ($\alpha$) does not have any effect. Hence the data were correlated in terms of the combined Grashof number ($GR_m$) and the Schmidt number, and the best fit is given in equation (5.4.2.2):

$$\text{Sh} = (0.165 \pm 0.014)(GR_m \text{ Sc})^{0.30 \pm 0.01}$$ \hspace{1cm} (5.4.2.2a)

For practical purposes equation 5.4.2.2a can be written as:

$$\text{Sh} = 0.17(GR_m \text{ Sc})^{0.30}$$ \hspace{1cm} (5.4.2.2b)

Examination of Figure 5.4.2.2 indicates no observable differences in non-isothermal laminar mass transfer for $\alpha = 0^\circ$, $\beta = 0^\circ$ (cathode facing upwards) and $\alpha = 180^\circ$, $\beta = 0^\circ$ (cathode facing downwards). As discussed in section 5.3.1, it was expected to see a difference in mass transfer due to the density difference between the bulk solution and the 'cathode surface' solution. However the density difference is quite small, and, within experimental error, no mass transfer difference was observable.

Normalized Sherwood numbers (equation 5.4.2.1 divided by equation 5.4.2.1 for $\alpha = 0^\circ$, $\beta = 0^\circ$) as a function of the cathode angle ($\alpha$) were plotted in Figure 5.4.2.3. The normalized Sherwood numbers were independent of the Grashof number and Schmidt number; the only dependent variable was the cathode angle $\alpha$. Figure 5.4.2.3 is valid for non-isothermal laminar mass transfer at $0^\circ \leq \alpha \leq 180^\circ$, $\beta = 0^\circ$, $200 \leq \text{Re} \leq 1900$, $\text{Sc} = 2000$, and $(d_c/L) = 0.114$. A 34% increase in non-isothermal laminar mass transfer in a horizontal channel was observed at a cathode angle of $90^\circ$.

Figure 5.4.2.4 shows experimentally obtained Sherwood numbers for non-isothermal laminar and
FIGURE 5.4.2.2 Non-Isothermal Mass Transfer with Rotation of the M.T. Surface
FIGURE 5.4.2.3 Non-Isothermal Mass Transfer with Rotation of M.T. Surface (Eq 5.4.2.1)
FIGURE 5.4.2.4 Non-Isothermal Mass Transfer with Elevation of the M.T. Surface
turbulent mass transfer at various flow channel elevations \((\alpha = 0^\circ \text{ and } \beta = 0^\circ, 30^\circ, 60^\circ, 90^\circ)\). The influence of the channel slope \((\beta)\) on the non-isothermal laminar mass transfer at \(\alpha = 0^\circ\), \(0^\circ \leq \beta \leq 90^\circ\), \(200 \leq \text{Re} \leq 1900\), \(\text{Sc} = 2000\), and \((d_1/L) = 0.114\) is shown in Figure 5.4.2.5. There is a 43% increase in the mass transfer as the channel is elevated from the horizontal \((25/25/0/0)\) to the vertical \((25/25/0/90)\) position. Examination of Figure 5.4.2.5 shows that the Sherwood number increases approximately 1.4 times when the channel is elevated from the horizontal (cathode facing upward position) to a slope of 60°. But only a very small increase in mass transfer \((= 5\%)\) was found while the channel was elevated from a slope of 60° to the vertical position.

Figure 5.4.2.6 shows the influence of the cathode angle \((0^\circ \leq \alpha \leq 180^\circ)\) and the channel slope \((0^\circ \leq \beta \leq 60^\circ)\) on the non-isothermal laminar mass transfer. A 74% increase in mass transfer over the non-isothermal laminar case \((\alpha = 0^\circ, \beta = 0^\circ)\) can be expected at a flow channel position of \(\alpha = 90^\circ\) and \(\beta = 60^\circ\). As stated in section 5.3.1, no predictions in the development of the non-isothermal laminar mass transfer for \(0^\circ \leq \alpha \leq 180^\circ\) and \(60^\circ > \beta < 90^\circ\) can be made.

5.4.3 Transition - Turbulent Flow Results

The Sherwood numbers calculated for the +10°C, +20°C and +30°C temperature difference, between the cathode surface and bulk solution, are shown in Figure 5.4.2.1. Since there is an increase in the non-isothermal turbulent mass transfer of about 20% over the isothermal turbulent case \((25/25/0/0)\), it suggests that there is an influence of free and forced convection on the overall mass transfer in the turbulent flow region. Hence, both free and forced convection should be taken into consideration when correlating the experimental data. The non-isothermal turbulent regime \((\text{Re} > 1900)\) data for the cathode angle range of \(0^\circ \leq \alpha \leq 180^\circ\) and the channel slope range of \(0^\circ \leq \beta \leq 60^\circ\) were best correlated by:

\[
\text{Sh} = (0.283 \pm 0.009)(1 + (0.389 \pm 0.008)(\sin \alpha)^{0.031 \pm 0.022} + (0.318 \pm 0.017)(\sin \beta)^{0.931 \pm 0.035})...
\]

\[
... \text{Re}^{0.66 \pm 0.09} (\text{GR}_m \text{ Sc})^{0.664 \pm 0.014}
\]  

(5.4.3.1a)
200 \leq Re \leq 1900
Sc = 2000
\alpha = 0^\circ
\frac{d_e}{L} = 0.114

FIGURE 5.4.2.5  Non-Isothermal M.T. with Elevation of the M.T. Surface (Eq 5.4.2.1, 5.4.2.2)
FIGURE 5.4.2.6 Non-Isoth. Laminar M.T. (Eq. 5.4.2.1)
Compared to M.T. at ($\alpha=0^\circ$, $\beta=0^\circ$)
For practical purposes equation 5.4.3.1a can be written as:

\[
Sh = 0.283(1 + 0.39\sin\alpha)^{0.66} + 0.32(\sin\beta)^{0.41})\ Re^{0.66} (Gr_{\infty}\ Sc)^{0.084}
\]  
(5.4.3.1b)

For vertical flows (\(\beta = 90^\circ\)), the experimental data were best correlated by:

\[
Sh = (0.372\pm 0.029)\ Re^{0.68\pm 0.034} (Gr_{\infty}\ Sc)^{0.08\pm 0.006}
\]  
(5.4.3.2a)

For practical purposes equation 5.4.3.2a can be written as:

\[
Sh = 0.372\ Re^{0.66} (Gr_{\infty}\ Sc)^{0.090}
\]  
(5.4.3.2b)

Figure 5.4.3.1 depicts the normalized Sherwood numbers as a function of the cathode angle, while Figure 5.4.3.2 shows the influence of the channel slope on the mass transfer. The same conclusions can be drawn as in the previous sections:

i. the maximum mass transfer occurred at an a cathode angle of 90° in the horizontal apparatus configuration.

ii. at a constant cathode angle of 0°, the mass transfer increased approximately 32% when the channel was elevated from the horizontal to the vertical position.

Figure 5.4.3.3 presents the effect of flowrate (Reynolds number) on the mass transfer (Sherwood number) for the optimum arrangement (25/55/90/60) of the non-isothermal operation compared to the isothermal case (25/25/0/0). Under laminar flow conditions at a Reynolds number of about 300, there is an approximate fourfold increase in mass transfer. This enhancement is less under turbulent conditions but is still at least twofold.

Figure 5.4.3.4 shows the influence of the cathode angle (0° ≤ \(\alpha\) ≤ 180°) and the channel slope (0° ≤ \(\beta\) ≤ 60°) on the non-isothermal turbulent mass transfer. A 68% increase in mass transfer over the
non-isothermal turbulent case \((\alpha = 0^\circ, \beta = 0^\circ)\) can be expected at a flow channel position of \(\alpha = 90^\circ\) and \(\beta = 60^\circ\). No predictions in the development of the non-isothermal turbulent mass transfer for \(0^\circ \leq \alpha \leq 180^\circ\) and \(60^\circ > \beta < 90^\circ\) can be made (see section 5.3.1).

Finally, to show the adequacy of these non-isothermal laminar and turbulent mass transfer correlations (equation 5.4.2.1, 5.4.2.2, 5.4.3.2 and 5.4.3.3), the percent deviations of the predicted Sherwood numbers from the experimental values are plotted in Figure 5.4.3.5 for all 2394 experiments. The percent deviation of the experimental Sherwood number from the predicted Sherwood number (equation 5.4.2.1, 5.4.2.2, 5.4.3.2 and 5.4.3.3) is approximately \(\pm 3\%\).
FIGURE 5.4.3.1 Non-Isothermal M.T. with Rotation of the M.T. Surface (Eq 5.4.3.1)

- $1900 < Re \leq 35000$
- $Sc = 2000$
- $\beta = 0^\circ$
- $d_c/L = 0.114$
FIGURE 5.4.3.2 Non-Isothermal M.T. with Elevation of the M.T. Surface (Eq 5.4.3.1, 5.4.3.2)
FIGURE 5.4.3.4 Non-Isoth. Turbulent M.T. (Eq. 5.4.3.1)
Compared to M.T. at \((\alpha=0^\circ, \beta=0^\circ)\)
CHAPTER SIX

COMPUTER SIMULATION AND DISCUSSION

6.1 MATHEMATICAL DESCRIPTION OF THE LAMINAR FLOW DOMAIN

Combined buoyancy forces of heat and mass transfer, resulting from the simultaneous presence of differences in temperature and concentration, may be present in flow systems which can thus exhibit both free and forced convection. Despite their importance in engineering applications, the combined buoyancy effects of thermal and mass diffusion on forced convection channel flow have not received much attention. Here we present an analysis of combined buoyancy effects of thermal and mass diffusion on laminar forced convection flow as related to the present study. The geometry under investigation has been for mixed convection flow in a horizontal rectangular channel flow reactor. The isothermal experiments involved only mass transfer effects. For the non-isothermal experiments, one wall (the upward facing cathode) was kept at uniform but higher temperature level from the bulk solution thus introducing heat transfer effects in addition.

In an initial effort to analyze the mass diffusion effects in mixed convection flows, the problem was thought of as a 3-dimensional flow system. The primary objective was to determine the effects of the buoyancy forces, resulting from both a temperature and a concentration gradient at the cathode surface - which was positioned in a 3-dimensional space - , in each direction (x, y, z). The resulting 3-dimensional equations required for their solution computer resources which exceeded by far those of the computer system available for the numerical analysis. Hence the problem was reduced to a 2-dimensional one (see Chapter 6.6 for further explanation). Nevertheless all the governing equations and computer source codes were developed for both the 2-and 3-dimensional case.

The equations governing the flow of a viscous fluid, which may be either a homogenous (single
component) fluid or a (multi-component) mixture containing chemical species, are mathematical
representations of the principles of conservation of mass, momentum and energy. In this section these
equations are presented for the case of laminar flow. The modifications necessary when the flow is
turbulent are explained in Section 6.2.

The equation of continuity describes the rate of change of density at a fixed point resulting in
the changes in the mass velocity vector \( \mathbf{v} \). This equation in any form is simply a statement of
conservation of mass. Mass conservation also applies to the individual components of a mixture. The rate
of change of mass concentration of a species can be due to advection, diffusion and chemical reaction but
this case involved only advection and diffusion.

Application of the principle of conservation of linear momentum to a fluid element leads to the
well known Navier-Stokes equations (\( \nu = \text{constant} \)). When all three components of the momentum
equation are added together vectorially, it will result in:

\[
\rho \frac{Dv}{Dt} = -\nabla p + \nabla \cdot (\tau) + \rho g
\]  \hspace{1cm} (6.1.1)

In this form, the equation of motion states that a small volume element moving with the fluid is
accelerated because of the forces acting upon it. In other words, this is a statement of Newton’s second
law in the form mass \( x \) acceleration = sum of forces. It can be seen that the momentum balance is
completely equivalent to Newton’s second law of motion. The equation of motion is also valid for non-
isothermal flow. In using it, of course, one must consider \( \rho \) and \( \mu \) to be functions of the temperature as
well as pressure.

When the flow is non-isothermal, the temperature of the fluid is a dependent variable which is
a function of the space (x, y, z direction) and the time. Just as the continuity equation is a mathematical
expression of the law of conservation of mass and gives the velocity distribution in space, the energy
equation is a mathematical expression for the law of conservation of energy and gives the temperature
distribution in space.
To complete the specification of the problem, it is necessary to add an additional equation, called the equation of state, which typically relates density, temperature, pressure and species concentration. Since the electrolyte under investigation is to be assumed incompressible, the pressure is not present in the equation of state. There are, however, two distinct equations of state available to relate density, temperature and species concentration (Fluid Dynamics International, 1990; Bird, Stewart and Lightfoot, 1960). One of these is the so-called Boussinesq approximation and the other is a restricted variable density formulation.

The equation of state for the Boussinesq approximation, simply takes the form that the density is constant.

\[ \rho = \rho_0 \]  \hspace{1cm} (6.1.2)

except that in the presence of a gravitational field, a buoyancy force exists due to density variations. This force has the representation

\[ - \rho_0 \left[ \beta_1 (T - T_0) + \beta_{c_1} (c_{1a} - c_{10}) + \beta_{c_2} (c_{2a} - c_{20}) \right] g \]  \hspace{1cm} (6.1.3)

where \( \beta_1, \beta_{c_1}, \beta_{c_2} \) are volumetric expansion coefficients associated with variations in temperature, species 1 concentration and species 2 concentration respectively, \( T_0, c_{10}, c_{20} \) are constant reference values, and \( g \) is the acceleration due to gravity.

A more general equation of state (Variable-Density Model) expresses the density as a function of temperature and species concentration in the form

\[ \rho = \rho_0 \left[ 1 - \beta_1 (T - T_0) \right] \left[ 1 - \beta_{c_1} (c_{1a} - c_{10}) \right] \left[ 1 - \beta_{c_2} (c_{2a} - c_{20}) \right] \]  \hspace{1cm} (6.1.4)

This representation is quite general, since the coefficients \( \beta_1, \beta_{c_1}, \beta_{c_2} \) may be arbitrary functions of \( T, \)
c_{1v} and c_{2v}.

Without accounting for the changes in density, the governing equations for laminar flow conditions can be written as follows:

**Continuity equation**

\[
\frac{\partial \rho}{\partial t} = - \left( \frac{\partial}{\partial x} \rho u_x + \frac{\partial}{\partial y} \rho u_y + \frac{\partial}{\partial z} \rho u_z \right)
\]  \(6.1.5\)

**Momentum equation**

**x-component**

\[
\rho \left( \frac{\partial u_x}{\partial t} + u_x \frac{\partial u_x}{\partial x} + u_y \frac{\partial u_x}{\partial y} + u_z \frac{\partial u_x}{\partial z} \right) = - \frac{\partial P}{\partial x} + \mu \left( \frac{\partial^2 u_x}{\partial x^2} + \frac{\partial^2 u_x}{\partial y^2} + \frac{\partial^2 u_x}{\partial z^2} \right) + \rho g_x
\]  \(6.1.6\)

**y-component**

\[
\rho \left( \frac{\partial u_y}{\partial t} + u_x \frac{\partial u_y}{\partial x} + u_y \frac{\partial u_y}{\partial y} + u_z \frac{\partial u_y}{\partial z} \right) = - \frac{\partial P}{\partial y} + \mu \left( \frac{\partial^2 u_y}{\partial x^2} + \frac{\partial^2 u_y}{\partial y^2} + \frac{\partial^2 u_y}{\partial z^2} \right) + \rho g_y
\]  \(6.1.7\)

**z-component**

\[
\rho \left( \frac{\partial u_z}{\partial t} + u_x \frac{\partial u_z}{\partial x} + u_y \frac{\partial u_z}{\partial y} + u_z \frac{\partial u_z}{\partial z} \right) = - \frac{\partial P}{\partial z} + \mu \left( \frac{\partial^2 u_z}{\partial x^2} + \frac{\partial^2 u_z}{\partial y^2} + \frac{\partial^2 u_z}{\partial z^2} \right) + \rho g_z
\]  \(6.1.8\)
Energy equation

\[ \rho c_p \left( \frac{\partial T}{\partial t} + u_x \frac{\partial T}{\partial x} + u_y \frac{\partial T}{\partial y} + u_z \frac{\partial T}{\partial z} \right) = \lambda \left( \frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2} \right) - \]

\[ ... + 2\mu \left[ \left( \frac{\partial u_x}{\partial x} \right)^2 + \left( \frac{\partial u_y}{\partial y} \right)^2 + \left( \frac{\partial u_z}{\partial z} \right)^2 \right] + \mu \left[ \left( \frac{\partial u_x}{\partial y} + \frac{\partial u_y}{\partial x} \right)^2 + \left( \frac{\partial u_x}{\partial z} + \frac{\partial u_z}{\partial x} \right)^2 + \left( \frac{\partial u_y}{\partial z} + \frac{\partial u_z}{\partial y} \right)^2 \right] \]

(6.1.9)

The terms in brackets, [...], represent the viscous dissipation, which is an irreversible rate of internal energy increase per unit volume due to viscous effects. Thus equation (6.1.9) states (on a per-unit-volume basis) that the temperature of the moving fluid changes due to heat conduction, viscous dissipation, and expansion effects. The viscous dissipation usually has a negligible effect on the fluid temperature and may be omitted except for highly viscous fluids or problems having velocities near the sonic velocity. The viscous dissipation terms will be neglected in the analysis of this problem.

Fick's second law

\[ \frac{\partial c_A}{\partial t} + u_x \frac{\partial c_A}{\partial x} + u_y \frac{\partial c_A}{\partial y} + u_z \frac{\partial c_A}{\partial z} = D \left( \frac{\partial^2 c_A}{\partial x^2} + \frac{\partial^2 c_A}{\partial y^2} + \frac{\partial^2 c_A}{\partial z^2} \right) \]

(6.1.10)

In order to account for the variations in density, due to existing temperature and concentration gradients, the bodyforce term in equations (6.1.6) to (6.1.8) will have to be replaced with the Boussinesq approximation equation (6.1.3). Without writing them out, the resulting new equations will be called (6.1.11) to (6.1.16) respectively. An important simplification of the above equation is, that for a steady state analysis, all derivatives with respect to the time are equal to zero.

For the two dimensional case (x,y direction) the continuity, momentum, energy and concentration equations can be written as equation (6.1.17) to (6.1.21), respectively.
\[ \frac{\partial \rho}{\partial t} = - \left( \frac{\partial}{\partial x} \rho u_x + \frac{\partial}{\partial y} \rho u_y \right) \] (6.1.17)

\[ \rho \left( \frac{\partial u_x}{\partial t} + u_x \frac{\partial u_x}{\partial x} + u_y \frac{\partial u_x}{\partial y} \right) = - \frac{\partial P}{\partial x} + \mu \left( \frac{\partial^2 u_x}{\partial x^2} + \frac{\partial^2 u_x}{\partial y^2} \right) + \rho g_x \] (6.1.18)

\[ \rho \left( \frac{\partial u_y}{\partial t} + u_x \frac{\partial u_y}{\partial x} + u_y \frac{\partial u_y}{\partial y} \right) = - \frac{\partial P}{\partial y} + \mu \left( \frac{\partial^2 u_y}{\partial x^2} + \frac{\partial^2 u_y}{\partial y^2} \right) + \rho g_y \] (6.1.19)

\[ \rho c_p \left( \frac{\partial T}{\partial t} + u_x \frac{\partial T}{\partial x} + u_y \frac{\partial T}{\partial y} \right) = \lambda \left( \frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} \right) \] (6.1.20)

\[ \frac{\partial c_A}{\partial t} + u_x \frac{\partial c_A}{\partial x} + u_y \frac{\partial c_A}{\partial y} = D \left( \frac{\partial^2 c_A}{\partial x^2} + \frac{\partial^2 c_A}{\partial y^2} \right) \] (6.1.21)

6.2. MATHEMATICAL DESCRIPTION OF THE TURBULENT FLOW DOMAIN

"Turbulence is one of the unsolved problems in the area of physical sciences. In many areas of fluid mechanics, the flows of practical and industrial relevance are almost always turbulent; this means that the fluid motion is highly random, unsteady and three dimensional. Due to these complexities, the turbulent motion and the heat and mass transfer phenomena associated with it are extremely difficult to describe and thus to predict theoretically. It is believed that the solution of the time-dependent three dimensional governing equations 6.1.5 to 6.1.9 can describe turbulent flows completely. However, these equations cannot be solved analytically and even today's supercomputers are not fast enough nor do they have the storage capacity to solve these equations directly for the required range of length and time scales, even for simple flows. The reason is that turbulent motion contains scales which are much smaller than the extent of the flow domain, typically of the order of 1000 times smaller. To solve the motion on these scales in a numerical procedure would require a mesh discretization far beyond the capabilities of today's computers. Hence, it is of practical importance to describe turbulent motion in terms of the time averaged quantities rather than instantaneous ones" (Fluid Dynamics International, 1990).
This approach is based on the conservation laws of mass, momentum and energy. These basic conservation laws are expressed by the exact equations referenced above, which describe all the details of the fluid motion. Since there was and still is hope of solving these equations, a statistical approach, first suggested by Osborne Reynolds, is taken and the equations are averaged over the time scale which is long compared with that of the turbulent motion. The resulting equations describe the distribution of mean velocity, pressure and temperature and thus the quantities of prime interest to the engineer. The derivation of these time-averaged turbulent equations have been commonly described (Fluid Dynamics International, 1990; Vlachopoulos, 1985; Bird, Stewart and Lightfoot, 1960; Hinze, 1959). The following material in sections 6.2, 6.3, 6.5 and 6.7 draws from the work of Fluid Dynamics International (1990), Vlachopoulos (1985), Schlichting (1978) and Hinze (1959).

The resulting turbulent governing equations are identical to those for the laminar case (equations 6.1.5 to 6.1.10) with the velocity, pressure, temperature and concentration replaced by their time averages and the viscosity replaced by the effective viscosity. The effective viscosity (eddy-viscosity concept) can be described as the sum of the laminar ($\mu_\alpha$) and the eddy viscosities ($\mu_T$) (equation 6.2.1).

$$\mu = \mu_\alpha + \mu_T$$  \hspace{1cm} (6.2.1)

In fact, the determination of the eddy viscosity ($\mu_T$), which is a function of many parameters (e.g. velocity, flow configuration, size, position in the flow field), presents the main problem in calculating turbulent flows. Because of all those factors involved, a general solution to the problem of turbulent flow cannot be presented (Vlachopoulos, 1985). The eddy-viscosity concept shifts the problem of turbulence modelling to the determination of $\mu_T$. In the literature of fluid mechanics two models are widely used to estimate $\mu_T$:

i. the zero-equation model (or Prandtl's mixing length model) and ii. the two equation model (or k-epsilon model). Other approaches to determine $\mu_T$ are e.g. the similarity hypothesis introduced by Von Karmán (1930) and an empirical formula advocated by Deissler (1955).
6.2.1 Zero Equation Model (or Prandtl’s Mixing Length Model)

The Prandtl mixing length hypothesis involves a single unknown parameter, the mixing length \( l_x \). The mixing length can be thought of as the mean free path for the collision or mixing of globules of turbulent fluid. The mixing-length hypothesis describes the flow field by means of empirical information. Typically \( \mu_r \) is computed using equation 6.2.1.1.

\[
\mu_r = \rho f_x^2 \left[ \left( \frac{\partial u_i}{\partial x_j} + \frac{\partial u_j}{\partial x_i} \right) \frac{\partial u_i}{\partial x_j} \right]^{\frac{3}{2}}
\]  

(6.2.1.1)

This model is described by Hinze (1959) to be difficult to apply in complex flows because of the problems in specifying the mixing length.

6.2.2 Two Equation Model (or k-\( \varepsilon \) Model)

In the \( k-\varepsilon \) model the turbulence field is characterized in terms of two variables, the turbulent kinetic energy \( k \), and the viscous dissipation \( \varepsilon \). Fluid Dynamics International presents an equation (6.2.2.1) showing that the turbulent viscosity is directly related to the turbulent quantities \( k \) and \( \varepsilon \).

\[
\mu_r \propto \rho u_r l_x \propto \frac{\rho k^2}{\varepsilon}
\]

(6.2.2.1)

"A transport equation for \( k \) can be obtained from the Navier-Stokes equations by a sequence of algebraic manipulations. This transport equation in addition to \( \varepsilon \) contains a number of unknown correlations. A second transport equation for \( \varepsilon \) can also be derived from the Navier-Stokes equations. Simplifications to these two equations will result in the well known equations of turbulent kinetic energy and viscous dissipation of the \( k-\varepsilon \) model" (Fluid Dynamics International, 1990). Then the unknown variables become \( u, T, c, k \) and \( \varepsilon \). The corresponding field equations, as presented in the Fluid Dynamics International Manuals (1990), are:
\[ u_{i,j} = 0 \] (6.2.2.4)

\[
\rho \left( \frac{\partial u_i}{\partial t} + u_i u_{i,j} \right) = -P_i + \left[ \mu \left( u_{i,j} + u_{i,j} \right) \right]_{i,j} - \rho g_i \left( \beta_i (T - T_0) + \beta_e (e - e_0) \right) \] (6.2.2.5)

\[
\rho \varepsilon_p \left( \frac{\partial T}{\partial t} + u_i T_{i,j} \right) = \left( \lambda T_{i,j} \right)_{i,j} + 2 \mu T_{i,j} \varepsilon_{i,j \varepsilon} \] (6.2.2.6)

\[
\rho \left( \frac{\partial \varepsilon}{\partial t} + u_i \varepsilon_{i,j} \right) = \left( D \varepsilon_{i,j} \right)_{i,j} \] (6.2.2.7)

\[
\rho \left( \frac{\partial k}{\partial t} + u_i k_{i,j} \right) = \left( \frac{\mu_r k}{Pr_k} \right)_{i,j} + 2 \mu T_{i,j} \varepsilon_{i,j \varepsilon} + \mu T_{i,j} \left( \frac{\beta_e}{Sc_r} \right)_{j,i} - \rho \varepsilon \] (6.2.2.8)

\[
\rho \left( \frac{\partial e}{\partial t} + u_i e_{i,j} \right) = \left( \frac{\mu_r e}{Pr_e} \right)_{i,j} + 2 c_1 \frac{e}{k} \mu T_{i,j} \varepsilon_{i,j \varepsilon} + c_1 (1 - c_2) \frac{e}{k} \left( \frac{\mu_r}{Pr_r} \right)_{j,i} + \beta_e \left( \frac{T_{i,j}}{Sc_r} \beta_e c_{i,j} \right) - \rho c_2 \frac{e^2}{k} \] (6.2.2.9)

Assuming that the effective quantities (i.e. viscosity, thermal conductivity and diffusivity) are the sum of their respective laminar and eddy quantities, the eddy viscosity can be written as:

\[
\mu_r = \rho c_p \frac{k^2}{\varepsilon} \] (6.2.2.10)

The equations (6.2.2.5) to (6.2.2.9) contain empirical constants \( Pr_r, Sc_r, Pr_e, Pr_v, c_1, c_2 \) and \( c_3 \). For a given flow problem, these constants may be available in the literature, or in the absence of such data, they have to be obtained by 'matching' the experimental results to the theoretical solution using a 'trial and error' approach.

### 6.3 NONDIMENSIONALIZATION OF THE GOVERNING EQUATIONS

The equations outlined in the preceding sections are expressed in dimensional form. These
equations can also be transformed into dimensionless ones. In this case, the input data are dimensionless parameters, such as Reynolds number, and the output results are then dimensionless quantities. Dimensionless formulation of problems may have significant advantages, such as:

i. a reduction of the large differences in orders of magnitude that can occur between terms in an equation

ii. an identification of the relative importance of various terms in the governing equations.

The non-dimensional sets of governing equations ((6.3.1.a) to (6.3.3.a) or (6.3.4.b) to (6.3.6.b) or (6.3.7.c) to (6.3.9.c)) were presented by Fluid Dynamics International (1990) for strongly coupled flows, such as the problem under investigation. For the sake of simplicity, the governing equations are shown with constant material properties, and with heat and mass source terms and reaction rates omitted.

\[ Re \left( \frac{\partial u_i^*}{\partial t^*} + u_j^* u_i^* \right) = -p_i^{**} - g_i \left[ \frac{Ra_{1i} T^*}{Pe_i} + \frac{Ra_c c^*}{Pe_c} \right] + u_{i,uu} \]  

\[ Pe_i \left( \frac{\partial T^*}{\partial t^*} + u_i^* T_{i,j}^* \right) = T_{i,uu} \]  

\[ Pe_c \left( \frac{\partial c^*}{\partial t^*} + u_i^* c_{i,j}^* \right) = c_{i,uu} \]  

\[ \frac{1}{Pr} \left( \frac{\partial u_i^*}{\partial t^*} + u_j^* u_{i,j}^* \right) = -p_j^{**} - g_i \left[ Ra_{1i} T^* + Ra_c Le c^* \right] + u_{i,uu} \]  

\[ \frac{\partial T^*}{\partial t^*} + u_j^* T_{i,j}^* = T_{i,uu} \]  

\[ \frac{1}{Le} \left( \frac{\partial c^*}{\partial t^*} + u_j^* c_{i,j}^* \right) = c_{i,uu} \]  

\[ Gr_{i}^{as} \left( \frac{\partial u_i^*}{\partial t^*} + u_j^* u_{i,j}^* \right) = -p_i^{**} - Gr_{i}^{as} g_i \left[ T_{i}^{*} + \frac{Gr_c}{Gr_i} c^* \right] + u_{i,uu} \]  

\[ Pr Gr_{i}^{as} \left( \frac{\partial T^*}{\partial t^*} + u_j^* T_{i,j}^* \right) = T_{i,uu} \]
\[ Sc \, Gr_t^{0.5} \left( \frac{\partial c_j}{\partial t} + u_j \frac{\partial c_j}{\partial x_j} \right) = c_{ij} \]  

(6.3.9.c)

The appropriate dimensionless groups for the three sets of equations (a), (b) and (c) are shown in Table 6.3.1 (Fluid Dynamics International, 1990).

Table 6.3.1 Dimensionless Systems used in equations 6.3.1.a to 6.3.9.c

<table>
<thead>
<tr>
<th>Quantity</th>
<th>Equation Set 'a'</th>
<th>Equation Set 'b'</th>
<th>Equation Set 'c'</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density</td>
<td>Re</td>
<td>1/Pr</td>
<td>( \text{Gr}^{0.5} )</td>
</tr>
<tr>
<td>Viscosity</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Specific Heat</td>
<td>( \text{Pr} )</td>
<td>( \text{Pr} )</td>
<td>( \text{Pr} )</td>
</tr>
<tr>
<td>Thermal Conductivity</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Capacity</td>
<td>( \text{Sc} )</td>
<td>( \text{Sc} )</td>
<td>( \text{Sc} )</td>
</tr>
<tr>
<td>Diffusivity</td>
<td>1/Re</td>
<td>( \text{Pr} )</td>
<td>1/( \text{Gr}_t^{0.5} )</td>
</tr>
<tr>
<td>Volume Expansion 'Thermal'</td>
<td>( \text{Ra} / (\text{Pr} \text{Re}) )</td>
<td>( \text{Ra} ), ( \text{Pr} )</td>
<td>1</td>
</tr>
<tr>
<td>Volume Expansion 'Concentration'</td>
<td>( \text{Ra} / (\text{Pr} \text{Re}) )</td>
<td>( \text{Ra} ), ( \text{Pr} \text{Le} )</td>
<td>( \text{Gr}_v/\text{Gr}_t )</td>
</tr>
</tbody>
</table>

6.4 INTRODUCTION TO THE COMPUTATIONAL FLUID DYNAMICS

Since the isothermal and non-isothermal flow system under investigation (equations (6.1.11) to (6.1.16)) is a strongly coupled one, no analytical solution is possible. Hence the only way to determine a solution for this system is by introducing a computational analysis. Historically, the time-proven approach to engineering design has been to construct a physical model and conduct laboratory experiments to 'measure the data' affecting the design decisions. In recent years, major advances in computer
technology and computational mechanics have made it possible to construct numerical models and again 'measure the data' obtained from the computational experiment. The computational approach allows the engineer to investigate different model configurations under different physical conditions (some of which may be unattainable in a laboratory setting) in a relatively short time. In the 'computational experiment', disturbances due to measurement devices are eliminated and detailed information on all physical variables throughout the flow domain under investigation is available. However, computational and experimental techniques are complementary and not mutually exclusive approaches to an engineering problem. Numerical simulation does not eliminate the need for building and testing prototypes, but it can considerably reduce the number and variety of such tests required, as well as enhance the effectiveness of testing.

In search for a computer software package to evaluate the governing equations for the isothermal and non-isothermal mass transfer in a flow channel under various cathode angles (α) and channel slopes (β), FIDAP™, the finite element solver developed and distributed by Fluid Dynamics International, Inc., Evanston, IL, U.S.A., appeared to be able to supply all the numerical information necessary to describe the problem under investigation. Among the information FIDAP™ was able to supply were the local mass transfer coefficients, temperature-, density-, and Reynolds number profiles. The FIDAP™ fluid dynamics analysis package is a general purpose computer program that uses the finite element method to simulate many classes of incompressible fluid flows. Two-dimensional, axi-symmetrical and three-dimensional steady state or transient simulations in complex geometries including the effects of temperature and chemical species are possible.

6.5 STEADY STATE ALGORITHMS

The numerical treatment of partial differential equations (PDE) is, by itself, a vast subject. PDE's are the heart of many, if not most, computer analyses or simulations of continuous physical systems, such as fluids, electromagnetic fields, etc.

The solution method for PDE's used in FIDAP™ is based on the finite element method (FEM),
In FEM the flow region is subdivided into a number of small regions, called finite elements. The partial differential equations of fluid mechanics covering the flow region as a whole are replaced by algebraic equations in each element. The system of these equations is then solved simultaneously by numerical techniques to determine the velocities, pressures, temperatures and concentrations throughout the region. Typically, the solution of this system of matrix equations represents the most time consuming aspect of the computational analysis. Therefore, the decision as to which solution algorithm to employ for this phase can govern and ultimately limit the size of the finite element model that can be treated. This is especially true in the case of highly non-linear problems or problems with coupled physical phenomena (i.e., heat-, mass-, and momentum transfer). For Reynolds numbers much greater than unity the convective term is very important; this causes the non-linear character of the equations to become dominant, making the choice of the solution algorithm, its convergence and efficiency a key issue. The strong non-linearity of the equations dictates the use of some type of iterative procedure to effect a solution. The search for a suitable iterative method is complicated by the existence of a multitude of possible procedures and their variants; the book by Ortega and Rheinboldt (1980) provides a survey of available procedures. Some FEM algorithms and their implications on the overall computer- and time requirements will be briefly discussed in the following section.

6.5.1 Finite Element Algorithms

In solving the Navier-Stokes equations numerically, a set of highly non-linear equations has to be solved. This means that an iterative approach has to be used to obtain a solution. Within the FIDAP™ software different numerical solution methods are available: Successive Substitution, Newton-Raphson, Modified Newton, and Quasi-Newton method. Since the solution phase usually accounts for the majority of computer time, a good choice is important for achieving an efficient solution.

In this section, the algorithms available in FIDAP™ for the fully coupled solution will be described briefly and a qualitative assessment of these algorithms in the finite element context will be given. A more detailed account of the individual methods can be found elsewhere (Fluid Dynamics
"The Successive Substitution method is the one mathematicians consider a robust method, that is to say, an iterative method which can be given a poor initial guess and the method will still proceed toward a converged solution. The drawback to this method is the often slow rate of convergence. This method is recommended as a first solution method.

The best known method is the generalized Newton-Raphson procedure. This method has a rate of convergence superior to that of the Successive Substitution iteration but there has to be a reasonable initial guess in order for this method to converge. The convergence is asymptotically quadratic so long as the initial solution vector is within the radius of convergence; unfortunately, this radius of convergence can sometimes be quite small and care must be taken in the choice of the initial vector. Although the radius of convergence of the Newton-Raphson method is somewhat smaller the that of the Successive Substitution method, it is sufficiently large to treat a significant range of problems, and since it converges rapidly, it is the generally recommended solution procedure for the steady-state Navier-Stokes problem (after reference: Fluid Dynamics International, 1990). Unfortunately, the Newton-Raphson method suffers from the same computational drawback as the Successive Substitution - the construction and complete factorization of a non-symmetric matrix is required at each iterative cycle. This fact alone can make both methods prohibitively expensive for three dimensional problems.

The modified Newton-Raphson (also known as the Secant Method) and Quasi-Newton methods are variations on the Newton-Raphson method which attempt to retain the fast convergence of Newton-Raphson without doing all the computations it does.

A compromise between the Newton-Raphson method, where the Jacobian is recomputed at every iteration, and the modified Newton method where the Jacobian is left unchanged, is found in the Quasi-Newton methods. They have been used extensively and are under active development in the field of numerical optimization. In practice, the convergence rate of the Quasi-Newton algorithm often approaches that of the Newton-Raphson method. Quasi-Newton also displays the same sensitivity to the initial solution
vector as the Newton-Raphson method; however, when a suitable initial vector is available, it can be a very powerful solution technique. Of the two, the Quasi-Newton procedure is the recommended one.

Although the Newton-Raphson and the Quasi-Newton methods are powerful solution procedures for the steady state Navier-Stokes equations, they are both somewhat limited in terms of their radius of convergence (e.g. effective Reynolds number range). An appealing technique of overcoming these shortcomings is to first use an algorithm with a larger radius of convergence but possibly slower convergence rate; e.g. Successive Substitution method, then switch to the faster Newton-Raphson or Quasi-Newton algorithm; i.e., combine the algorithms in a sequential manner. In the FIDAP™ package, it is possible to combine the above described methods - Successive Substitution, Newton-Raphson, and modified-Newton into a solution strategy. Thus, for example, the more robust Successive Substitution method can be used to bring the solution vector within the radius of convergence of the Newton-Raphson scheme or the Quasi-Newton method. The faster convergence rates of these methods can then be realized. The correct combination of solution techniques can result in a substantial saving of computer resources.

For the Newton-based methods, if the initial guess vector is not sufficiently close to the solution vector of the system of equations, convergence may not be achieved. To remedy this situation, incremental solution methods have been designed to increase the radius of convergence or to obtain sufficiently close starting vectors for these methods. The incremental loading method is used extensively in non-linear solid mechanics and has a well defined physical interpretation. In the fluid mechanics context, the natural loading parameter is the Reynolds number or the Rayleigh number for Boussinesq type flows; i.e., the Reynolds number is incremented until the solution at the required Reynolds number is attained. The solution at the previous lower Reynolds number is used as an initial vector for the computation of the solution at each higher Reynolds number. The advantage of this technique, which is often employed in practice, is that all the solutions are valid; i.e., solutions are available for a range of Reynolds number.

In FIDAP™, Newton-like methods can be used together with an incremental loading strategy (Fluid Dynamics International, 1990).
6.6 MINIMUM COMPUTATIONAL HARDWARE REQUIREMENTS

Numerical analyses, using the FIDAP™ software package, are limited in size only by practical considerations of computer time and the ultimate capacity of secondary storage devices. Versions of FIDAP™ exist for a wide variety of computers under many different operating systems. FIDAP™ PC/386 is a version of the FIDAP™ Fluid Dynamics Analysis Package for the Intel® 80386™-based Personal Computers. FIDAP™ PC/386 runs under the MS-DOS® operating system using Phar Lap’s 386 DOS-Extender environment.

Fluid Dynamics International supplied completely solved and documented examples of various transport phenomena problems as a part of the FIDAP™ software package. The intention was to enable the researcher to study the programming syntax and to evaluate the computer system performance.

The PC version of FIDAP™ is a fully functional version of the workstation based FIDAP™ package. Without exception, all capabilities of FIDAP™, both computational and graphical, are available in the PC version. At the same time, the hardware limitations of the PC will necessarily restrict the range of problems that can be attempted on these computers. While all two-dimensional and small three-dimensional problems can be simulated with the FIDAP™ PC, it has been proven to be unreasonable to attempt a large-scale three dimensional problem such as the present one on a PC. For future analysis of a three dimensional heat and mass transfer problem in an electrochemical reactor with rectangular cross-section, it is strongly recommended that a switch to a more powerful computer platform, e.g. SILICON-GRAPHICS 4D/380 or CRAY X-MP be made. To illustrate this point, Table 6.6.1 shows comparative computation times for selected problems (FIDAP™ example manual: examples 3, 6, 9, 13 and 18) solved on various computer platforms. Fluid Dynamics International (1990) solved these problems on a 20 MHz Dell 310 personal computer with 80386™ Intel® micro processor, a DEC MicroVAX and the SUN 4/110. The computation times to solve these problems on the computer system used in this study, denoted herein as PC (486/33), were also included in Table 6.6.1. It is important to note that computation times may vary with computer configurations (which includes the amount of memory and the processor and disk access speeds). For various degrees of problem complexities and different computer systems, Table 6.6.1
confirms that smaller transport phenomena problems can be solved on a personal computer, whereas larger ones should be transferred onto a workstation. Hence the problem of enhanced mass transfer in a flow reactor with rectangular cross-section was analyzed only for the 2-dimensional case (x, y direction). To justify this decision, the computer processing times for a given flow problem were compared. The computing times to solve the 2- and 3-dimensional laminar non-isothermal case (Experiment N59) were approximately 2 hours and up to 340 hours, respectively. The computer system used in this study was comprised of:

i. A 80486-DX™ based personal computer operating at 33 MHz

ii. Microsoft®'s MS-DOS® operating system (Version 5.0)

iii. 16 Megabytes (MB) of extended memory, 210 MB hard disk space for application software (e.g. MS-DOS® and FIDAP™) and 210 MB of continuous free hard disk space for the FIDAP™ matrices

iv. A VGA video graphics adaptor

v. One parallel port (mandatory)

vi. A Microsoft® compatible mouse (mandatory)

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### Table 6.6.1 Execution of Selected FIDAP™ Examples on various Computer Systems

<table>
<thead>
<tr>
<th>Problem</th>
<th>PC (386/20) [seconds]</th>
<th>MVAXII [seconds]</th>
<th>SUN4/110 [seconds]</th>
<th>PC (486/33) [seconds]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Laminar, isothermal flow, (FIDAP™ Example 3)</td>
<td>599</td>
<td>914</td>
<td>140</td>
<td>108</td>
</tr>
<tr>
<td>Free surface flow, (FIDAP™ Example 6)</td>
<td>641</td>
<td>992</td>
<td>137</td>
<td>116</td>
</tr>
<tr>
<td>Buoyancy driven flow (FIDAP™ Example 9)</td>
<td>2662</td>
<td>3265</td>
<td>481</td>
<td>227</td>
</tr>
<tr>
<td>Laminar, isothermal flow (FIDAP™ Example 13)</td>
<td>2734</td>
<td>3610</td>
<td>530</td>
<td>363</td>
</tr>
<tr>
<td>Turb. flow, 2 dimensional, (FIDAP™ Example 18)</td>
<td>20283</td>
<td>22658</td>
<td>3210</td>
<td>12383</td>
</tr>
</tbody>
</table>
6.7 ELEMENTS OF THE DEVELOPED FINITE ELEMENT PROGRAM

This section is intended as a reference guide for using the Finite Element program code (see Appendix F) developed for the FIDAP™ PC/386 version 5.0. The individual program steps were documented within the actual code (see Appendix F, Listings F.1 and F.5), but nevertheless a more accurate description of the commands used, will be given here. All the descriptions and definitions of the individual program control cards are adopted from the four FIDAP™ user- and tutorial manuals (Fluid Dynamics International, 1990). The source code development will be described for the more complex 3-dimensional analysis; the procedures described below can be revised for the 2-dimensional problems in a straightforward manner.

Because the flow under consideration is a boundary layer type, the solution for the governing equations can be marched in the downstream direction. While finite element computer codes are powerful tools for simulation of physical phenomena such as fluid flow, the accuracy and cost-effectiveness of the solution is to a large extent dependent on the finite element mesh discretization employed. Since hand-generating even simple grids is a very tedious task, and generating complex three-dimensional meshes is a very difficult, if not impossible, task, it is highly desirable to automate the generation of the mesh. FIDAP™ provides a tool to develop the mesh and the program source code in a straightforward fashion by using the so-called 'control cards'. Using the 3-dimensional program source code supplied in Appendix F (Listing F.5) as an example, a brief description of the control cards used to develop this code are given below.

"The *FIMESH control card is used to invoke the mesh generator. The 3-D command generates the coordinates of all points logically spanning the 3-D volume from the initial point to the final point. The IMAX, JMAX and KMAX keywords are used to specify the limits of the I, J and K dimensions in the logical integer space (Figures 6.7.1 and 6.7.2).

The EXPI, EXPJ and EXPK control cards are used to alter the density of the mesh in the logical coordinate directions. One of the most powerful features in FIMESH is the capability to alter the density of a mesh (i.e., make it finer or coarser) without altering the input file. This is accomplished by defining
FIGURE 6.7.1 Nodal Points for 2-dimensional FEM Analysis
a vector of new indices already specified in the input file. The EXPI, EXPJ and EXPK commands are used to input the vectors containing the new I, J and K indices, respectively.

The function of the POINT control card is twofold. The first is to assign a unique identifier (number) to points in geometric space and the second is to define the correspondence between the geometric definition and the definition of the point in the logical integer space for use in subsequent mesh generation commands. It is recommended that the POINT command be the first command following the last expansion command card (EXPI, EXPJ or EXPK).

The LINE command generates straight line edges from which surfaces can be designed. The SURFACE command produces the coordinates of all points logically spanning the surface from the initial point to the final point. A surface is a plane that is parallel to one of the three logical space planes; i.e., IJ plane, JK plane or IK plane. All four edges of a surface must have either been input or generated prior to surface generation. Points generated by one surface may be used as an edge for another surface.

The BCNODE control card is used to specify nodal point type boundary conditions on the boundaries of the model. The degrees of freedom that can have specified values defined include: x'y'z' components of the velocity (VELO), the x component for the velocity (UX), the temperature (TEMP) and the concentration of species 1 (1SPECIES) individually or all velocities on a single data card.

The ELEMENTS control card specifies which of the generated grid points are to be output as nodes and elements of the finite element mesh. It is important to be aware of the distinction between grid points and nodal points. The BRICK keyword specifies the type of finite elements to be generated. The NODES keyword is used to specify the element order approximation. For a three dimensional problem the elements available are 8 and 27 node BRICKs.

The NUMBER command selects the order of the logical directions to be used for generating the nodal point numbers. Since FIDAP™ uses a profile solver to solve the finite element equations, it is of real importance that the nodes in the mesh be numbered to reduce the profile of the assembled FEM matrix. Different nodal numberings can result in substantial differences in the computer resources required for the execution of FIDAP™. Algorithms which attempt to optimize the nodal numbering of a mesh for
profile-type solvers are usually based on one of two criteria: to minimize the bandwidth of the matrix, where the bandwidth is defined as a maximum difference in any row between the leftmost and the rightmost non-zero entries; or to minimize the profile of the matrix, where the profile is defined as the total number of storage positions within the profile envelope of the matrix. Although it is difficult to generalize, in most cases the reduction of the profile is more suited to the solver employed in FIDAP™. Both these renumbering algorithms are available using the *RENUMBER command.

The master control cards are described in the following section. These control cards completely determine the type of analysis to be performed plus specifying associated optional parameters.

To specify the system of equations to be solved the *PROBLEM control card has to be invoked.

A 3-D analysis is one for which the equations are expressed in cartesian coordinates (x,y,z).

If NONLINEAR is selected, then the convective terms in all momentum-, energy-, turbulent kinetic energy-, turbulent dissipation-, and mass transfer equations are included in the equations discretized.

The STEADY/TRANSIENT keywords select whether a steady state or transient analysis is to be performed. For a steady analysis, the time derivatives in all momentum-, energy-, turbulent kinetic energy-, turbulent dissipation-, and mass transfer equations are discarded; the method then employed to solve the resulting equations is selected using the *SOLUTION master control card.

The TWEAKLY/TSTRONGLY keywords determine whether or not the energy equation is to be solved. For weakly-coupled flows (TWEAKLY), the energy equation is solved but temperature dependence in the momentum equation can only be solved through temperature dependent fluid properties; i.e., the buoyancy term is not present in the momentum equation. For strongly-coupled flows (TSTRONGLY), the complete momentum and energy equations are solved. In all temperature dependent flows, the discretized versions of the momentum and energy equations are solved simultaneously.

The IWEAKLY/ISTRONGLY keywords determine whether the transport equation for species $I$ is to be solved. For weakly-coupled flows (IWEAKLY), the transport equation for species $I$ is solved but species $I$ dependence in the momentum equation can only be solved through species $I$ dependent fluid properties; i.e., the buoyancy term is not present in the momentum equation. For strongly-coupled flows
(ISTRONGLY), the complete momentum and species 1 mass transport equations are solved. In all species 1 dependent flows, the discretized versions of the momentum and species 1 transport equations are solved simultaneously.

The FIXED keyword implies that the region under consideration has no free surface body; i.e., there are no free surfaces or surface changing conditions (dissolving).

If the flow is specified as TURBULENT on the *PROBLEM control card, the desired turbulent viscosity model is chosen using the *VISCOSITY control card. The zero equation mixing length model is specified by the mixing keyword and the $k$-$\varepsilon$ model by the K.E. keyword. If the turbulent keyword is specified, one of these *VISCOSITY models must be chosen for the various FLUID element groups. The various constants required for the $k$-$\varepsilon$ model are input on the *VISCOSITY data cards.

FIDAP™ is capable of solving any combination of the momentum equation for the velocity and pressure fields, the energy equation for temperature and transport equation for species 1. Which combination of equations is actually solved depends on the sets of keywords. The momentum equation is solved if the MOMENTUM keyword is specified.

If *EXECUTION(DATACHECK) is specified, the program will read the entire data file and perform numerous consistency checks on the input data as well as providing information on computer requirements without executing the analysis. It is strongly recommended this option be used the first time any input file is submitted to FIDAP™. Once an input file successfully executes in the DATACHECK mode the job should be resubmitted with the NEWJOB keyword for a complete analysis of the problem.

The *PRESSURE(MIXED) control card specifies the numerical approach to be used for the incompressibility constraint and the discretization of the pressure variable. When the MIXED keyword is used, the pressure variable is discretized and contributes an additional degree of freedom to the system of unknowns to be solved for. The value of the MIXED keyword is value of the clipping constant used to clip small pivots encountered when performing the LU decomposition of the global coefficient matrix. It has a default value of $10^4$. If more than 10 pivots are encountered during the matrix decomposition phase this value should be reduced by one to two orders of magnitude.
The *SOLUTION master control card is used to specify the numerical method to be involved to solve the nonlinear system of equations which arises in a steady-state analysis or at each step of an implicit time integration simulation. The S.S. keyword selects the Successive Substitution as the iterative method used to obtain the nonlinear steady state solution.

The *POSTPROCESS card controls the output of the various solution quantities, velocities, pressure, temperatures, concentrations, etc., to the FDPOST post-processor file.

The nodal data and any associated boundary and initial conditions will be read from the FIMESH interface file if the *NODES(FIMESH) keyword is specified. A *NODES(FIMESH) control card must appear after the execution of the FIMESH module in the FIPREP input file.

Two different BRICK elements are available in the *ELEMENTS master control card: a linear 8 node element and a 27 node quadratic element. Finite elements for fluid flow simulation can be characterized by the velocity-pressure approximation used. In the case of the LINEAR pressure approximation, two different sets of basis function can be employed. If the Local keyword is specified, the linear basis functions lie in the local coordinate space of the element, while if the GLOBAL keyword is invoked, the basis function lie in the global coordinate space (i.e., x,y,z).  

The OUTFLOW keyword specifies a special outflow boundary condition element group. In certain situations, the natural outflow boundary condition that arises from the application of the finite element method to the flow equations, is not suitable. One particular situation is often encountered, namely: Outflow boundaries of flows involving buoyancy forces and gradients generate significant temperature gradients across the outflow boundary. A similar situation arises for buoyancy forces and gradients generated by species dependent density variations. At such a boundary, the temperature or concentration variation results in a pressure gradient which is in conflict with the default stress-free boundary condition applied in FIDAP™. The result of these incompatibilities is typically spurious separation and recirculation at the outflow boundary.

The value of the MDENS keyword is the set number of the fluid density model associated with the element group - this model is defined using the *DENSITY master control card. Constant or variable
density models are available.

In the same way, as described above, the control cards MCOND, MSPHT, MVISC, MEXP, M1EXP and MIDIFF can be used for defining the thermal conductivity, specific heat capacity, fluid viscosity, volume-expansion coefficient due to temperature, volume-expansion coefficient due to concentration, and diffusivity of species 1, by invoking the master control cards *CONDUCTIVITY, *SPECIFICHEAT, *VISCOSITY, *VOLUMEXPANSION(SEF=1), *VOLUMEXPANSION(SEF=2) and *DIFFUSIVITY, respectively.

The *ICNODE master control card is used to input the initial conditions which are applied directly to nodal points in the mesh; e.g. initial values for temperature, concentration of species 1, etc. For transient analysis these values are used as the initial condition at the initial time, while for a steady-state analysis they are used as the initial guess for the nonlinear iterative solution method.

The DISSIPATION / NODISSIPATION keywords determine whether the viscous dissipation term is to be included in the energy equation. In addition, when the DISSIPATION option is selected, the dissipation term $\mu \dot{\gamma}$ is multiplied by the value of the dissipation keyword. This option is provided in the event that a non-dimensionalization which requires a multiplicative factor in front of the dissipation term is being employed. Another situation where use of this factor may be required is when British units are being employed. The default value of the DISSIPATION keyword is 'Constant = 1.0'.

Each density model input using the *DENSITY master control card has a set number associated with it. This set number is then used to associate the model with a particular element group using the MDENS keyword on the *ELEMENT master control card. In many applications, the assumption of constant density together with the Boussinesq approximation is no longer sufficient, because the variation of density with temperature and possibly species concentration is too large. The conservation of mass equation must take these density variations into account. In the VARIABLE density approach, for a steady state problem, the continuity equation becomes

$$ \nabla \cdot (\rho \mathbf{u}) $$

(6.7.1)
With this formulation, the equation of state defining variation of density used in FIDAP\texttrademark{} is:

\[
\rho = \rho_0 \left[ 1 - B_1 (T - T_0) \right] \left[ 1 - B_{11} (c_{1e} - c_{10}) \right] \left[ 1 - B_{12} (c_{2e} - c_{20}) \right] 
\] (6.7.2)

It has to be noted, that the density follows the variation outlined in equation (6.7.2) in all terms in which the density appears. In this case, the body force term is not included in the momentum equation. If a body force term \( pg \) is desired in the momentum equation, to include the gravity effects, it must be explicitly included using the *BODYFORCE master control card. The values for the required expansion coefficients are specified using the *VOLUMEXPANSION master control card, while the MEXP and M1EXP keywords on the *ELEMENTS master control card associate the volume expansion coefficient model with a particular element group. In summary, there are some points to note regarding the use of the variable density model. These include:

i. One or more of the keywords TSTRONGLY, 1STRONGLY or 2STRONGLY must be specified on the *PROBLEM control card.

ii. A fully coupled solution approach must be used, i.e., the segregated solver cannot be used.

iii. A MIXED formulation for the pressure must be used.

iv. There is no variation of density with respect to pressure or time.

v. The Boussinesq body force term is not included in the momentum equation. If the body force term is desired in the momentum equation, it must be included using the *BODYFORCE control card.

The viscosity model is defined by a function curve using the *\backslash ISOSITY(CURVE) master control card. The independent variable for the functional dependence may be temperature and/or species \( l \) (selected by the TEMPERATURE or ISPECiES keywords, respectively); if no dependence is explicitly specified, the default is TEMPERATURE. If a turbulence model is being chosen one of the two keywords
must be added:

i. The MIXING keyword specifies a eddy viscosity mixing-length type turbulence model. When this model is chosen the TURBULENT keyword must be specified on the *PROBLEM control card and the time averaged Navier-Stokes, energy and concentration equations are solved. Up to four constants must be input for the MIXING length model; they are the laminar viscosity, the turbulent Prandtl number, and the turbulent Schmidt numbers for species 1 and 2. Additional constants may be input if required by the subroutine USRMXL.

ii. A K.E. *VISCOSITY property set is used for two closely related purposes:

- to specify a k-ε turbulence model for a simulation and input the required constants for the model. This function is recognized by associating the property set with a fluid element group.

- to input the constants required by a near-wall boundary model (WALL elements) when a k-ε turbulence model is being used for the fluid elements. This function is recognized by associating the property set with a WALL element group. In general, the same property set serves the purpose of specifying the k-ε model and the entry of the constants for a near-wall boundary model. In either case the TURBULENT keyword must be specified on the *PROBLEM control card.

To account for the thermal conductivity, heat capacity and diffusivity the same rules apply by using the control cards *CONDUCTIVITY (CONSTANT), *SPECIFICHEAT (CONSTANT) and *DIFFUSIVITY (CURVE), respectively.

The *VOLUMEXPANSION card is used to input data for a volume-expansion coefficient fluid property data set. Each volume-expansion coefficient model input using the *VOLUMEXPANSION control card has a set number associated with it. This set number is then used to associate the model with a particular element group using the MEXP keyword for a temperature-dependent buoyancy term and the M1EXP keyword for a species 1 dependent buoyancy term on the *ELEMENT control card.
The *BODYFORCE control card specifies the data for a bodyforce acting on the fluid. It should be noted that the body force is input as a volumetric force; i.e., force/unit mass is required.

Since FIDAP\textsuperscript{TM} uses a profile solver to solve the finite element equations, it is of great importance that the nodes in the mesh be numbered so as to reduce the skyline profile of the assembled FEM matrix. The *RENUMBER card is used to perform the optimal renumbering of the nodes in the mesh so as to reduce the size of the global coefficient matrix" (Fluid Dynamics International, 1990).

6.8 NUMERICAL SIMULATIONS AND CONDITIONS INVESTIGATED

As outlined above the 2- and 3-dimensional FIDAP\textsuperscript{TM} source code for isothermal and non-isothermal horizontal flow ($\alpha = 0^\circ$, $\beta = 0^\circ$) was developed. Because of the computer and time requirements involved for solving the 3-dimensional isothermal and non-isothermal case it was decided to reduce the numerical analysis to a 2-dimensional ($x, y$) one. This decision also limited the scope of theoretical analysis which could be performed. Since the cathode angle ($\alpha$) is a function of the $y, z$ direction and the problem was investigated in the $x, y$ direction, it was only possible to analyze the influence of the channel slope ($\beta$) on the mass transfer coefficient.

The numerically simulated experimental conditions for the isothermal mass transfer investigations employed cathode angle ($\alpha$) of 0 degrees and channel slopes ($\beta$) of 0, 30, 60, 90 degrees and Reynolds numbers of approximately 200 to 1900. The isothermal simulations were performed for a bulk solution temperature of $T_1 = 25^\circ C$.

The non-isothermal simulations were performed for a cathode angle ($\alpha$) of 0 degrees and channel slopes ($\beta$) of 0, 30, 60, 90 degrees and Reynolds numbers of approximately 200 to 1900. The maximum temperature difference between the cathode surface and bulk solution was approximately $+30^\circ C$.

All numerically simulated parameters (e.g. velocity, bulk solution temperature and concentration, and cathode surface temperature) for the isothermal and non-isothermal experiments were set exactly as they were in the actual physical experiment (e.g. Experiment 11: $U = 2$ cm/s, $T_1 = 24.7^\circ C$, $\alpha = 0^\circ$,
\[ \beta = 0^\circ \].

In order to perform the numerical analysis of the laminar isothermal and non-isothermal flow situations the initial- (I.C.) and boundary conditions (B.C.), listed in Table 6.8.1, were used.

<table>
<thead>
<tr>
<th>CONDITION (I.C. or B.C.)</th>
<th>TYPE</th>
<th>LOCATION</th>
</tr>
</thead>
<tbody>
<tr>
<td>B.C.</td>
<td>( U_i = U_{in} )</td>
<td>CONSTANT VELOCITY AT THE CHANNEL INLET</td>
</tr>
<tr>
<td>B.C.</td>
<td>( T_i )</td>
<td>CONSTANT TEMPERATURE AT THE CHANNEL INLET</td>
</tr>
<tr>
<td>B.C.</td>
<td>( u = 0 )</td>
<td>ALL CHANNEL WALLS (no slip condition)</td>
</tr>
<tr>
<td>B.C.</td>
<td>( T_a )</td>
<td>CONSTANT CATHODE TEMPERATURE</td>
</tr>
<tr>
<td>B.C.</td>
<td>( C_{oo} = C_{bulk} )</td>
<td>CONSTANT CONCENTRATION AT THE CHANNEL INLET</td>
</tr>
<tr>
<td>B.C.</td>
<td>( C_{oo} = 0 )</td>
<td>CONSTANT CATHODE CONCENTRATION</td>
</tr>
<tr>
<td>I.C.</td>
<td>( T_i )</td>
<td>APPLIED INITIALLY AT EACH NODAL POINT</td>
</tr>
<tr>
<td>I.C.</td>
<td>( C_{oo} )</td>
<td>USE GOOD GUESS, IT WILL BE APPLIED TO EACH NODAL POINT</td>
</tr>
</tbody>
</table>

The FIDAP\textsuperscript{TM} software simulations for the isothermal and non-isothermal combined convection studies supplied, among other quantities (see reference manuals: Fluid Dynamics International, 1990), the density-, temperature-, Reynolds number profiles, and the local mass transfer coefficients. The mass flux normal to a given boundary was calculated by the FIDAP\textsuperscript{TM} software to be:

\[ N_a = -D \frac{\partial c_i}{\partial x_i} \]  

(6.8.1)

where \( D \) is the diffusion coefficient. The local mass transfer coefficient, \( K \), is then given by:
\[ K = \frac{N_A}{c - c_{ref}} \]  

To compare the numerically obtained local mass transfer coefficients (equation 6.8.2) to the experimental average ones, the local mass transfer coefficients were integrated over the total length of the cathode using the trapezoidal rule.

In the course of the theoretical analysis the governing equations were solved in the dimensional form. This approach posed some difficulty in solving the governing equations with orders of magnitude difference between the individual quantities (e.g., density, viscosity, velocity). These difficulties sometimes caused the solution, due to poor guesses of the initial conditions, either to converge very slowly or not at all. A non-dimensional formulation can sometimes result in a reduction of the large differences in orders of magnitude that can occur between terms in an equation. After extensive discussions with Fluid Dynamics International, it was recommended that the governing equations should be solved in their dimensionless form, which should simplify the analysis significantly.

6.9 RESULTS AND DISCUSSION OF THE NUMERICAL SIMULATIONS

Since it was decided to analyze the problem of isothermal and non-isothermal mass transfer in a flow reactor in a 2-dimensional way, it had to be verified that no large errors in the final solution would be introduced by excluding the third dimension. A 2- and a 3-dimensional numerical analysis was performed for the non-isothermal experiment N59. The results of these numerical simulations are shown in a number of different plots. Figure 6.9.1 depicts for experiment N59 the 3-dimensional finite element mesh plot as generated by FIDAP™. Figure 6.9.2 compares the local mass transfer coefficient for the 2-dimensional case to the 3-dimensional one. The local mass coefficients for the 3-dimensional case were determined in the centre of the flow channel (z = 1 cm). Examination of these 2- and 3-dimensional data shows essentially no difference between the local mass transfer coefficients; and it seems that the assumption of analyzing the problem as a 2-dimensional one will not introduce any additional errors. The
local mass transfer coefficients from the 2-dimensional simulation were used to calculate the average mass transfer coefficient. The deviation from the experimentally obtained overall mass transfer coefficient was approximately 2%. The Reynolds number profile at the downstream edge of the cathode is shown in Figure 6.9.3. The highest Reynolds numbers can be observed in the upper third of the channel and the lowest ones can be found at the cathode surface where the density is lower than in the bulk solution. For presentation reasons only 10 different Reynolds number profiles were drawn. It can be concluded that in the z-direction the non-isothermal problem is a unsymmetrical one. The density contour plot (Figure 6.9.4) shows a lower density at the cathode surface than in the bulk solution. Furthermore the constant density lines at the upstream edge of the cathode show some irregularities which may be caused by the finite element mesh size. The same irregularities are found in the temperature contour plot (Figure 6.9.5).

A detailed evaluation of the isothermal numerical results, their comparison to the effects observed in the laboratory and their similarity to experimental isothermal local Sherwood numbers found in the literature is given in section 6.9.1. The results from the non-isothermal theoretical analysis are presented in section 6.9.2.

6.9.1 Results of the Isothermal Laminar Simulations

Isothermal numerically simulated data were obtained for the same apparatus dimensions as discussed in Chapter 4. The numerical conditions were set to match the experimental ones in order to be able to compare them directly. Isothermal laminar experimental conditions at various Reynolds numbers and channel slopes ($\beta$) of 0, 30, 60 and 90° were evaluated. As discussed before, the cathode angle ($\alpha$) was kept constant at 0°. Figure 6.9.1.1 shows numerically obtained local mass transfer coefficients, at various cathode positions along the x-direction, for the simulation of the isothermal laminar experiments 11, 11769, 12790, and 13209 (Re = 400, Sc = 1270, $\alpha = 0°$, and $T_i = 25°C$). It should be noted, that with increasing channel elevation ($\beta$) not only the local mass transfer coefficient increases but also the point at which fully developed mass transfer occurs is shifted closer to the downstream edge of the mass transfer surface. Fully developed mass transfer conditions are defined by the position along the cathode
FIGURE 6.9.2 Local and Average M.T. Coefficient (N59)
FIGURE 6.9.4 Density Contour Plot – Simulation of Experiment N59
FIGURE 6.9.5 Temperature Profile – Simulation of Experiment N59
at which no change in the concentration boundary layer thickness (i.e. no change in local mass transfer coefficient) occurs. As the concentration boundary layer develops from zero thickness at the upstream edge of the cathode to a finite value, the local mass transfer coefficient will reduce from $\infty$ to a constant value. Since the local mass transfer coefficient at the upstream edge of the cathode is assumed to be $\infty$, the first local mass transfer coefficient value for the graphs in Figure 6.9.1.1 was obtained and plotted for $x = 3 \text{ mm}$. A small error in the calculation of the average mass transfer coefficient was introduced by assuming that the local mass transfer coefficient, found at $x = 3 \text{ mm}$, was the valid one for the upstream edge of the cathode ($x = 0 \text{ mm}$). This assumption was necessary in order to be able to integrate numerically the local mass transfer coefficients over the total length of the mass transfer surface and to obtain the average mass transfer coefficient.

Table 6.9.1.1 offers a comparison between experimentally ($K_{\text{exp}}$) and numerically ($K_{\text{sim}}$) obtained average mass transfer coefficients for isothermal laminar mass transfer in a horizontal flow reactor ($\alpha = 0^\circ, \beta = 0^\circ$).

**Table 6.9.1.1  Comparison of Experimentally and Numerically obtained Average Mass Transfer Coefficients for the Laminar Isothermal Case of $\alpha = 0^\circ, \beta = 0, 30, 60$ and 90°**

<table>
<thead>
<tr>
<th>$K_{\text{sim}}$ $(10^4 \text{ cm/s})$</th>
<th>$K_{\text{exp}}$ $(10^4 \text{ cm/s})$</th>
<th>Re [-]</th>
<th>$T_1$ [°C]</th>
<th>$T_2$ [°C]</th>
<th>$\alpha$ [°]</th>
<th>$\beta$ [°]</th>
<th>Laboratory Exp. #</th>
<th>$\Delta K$ [%] (Exp. = 100%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.832</td>
<td>2.038</td>
<td>266</td>
<td>24.7</td>
<td>N/A</td>
<td>0</td>
<td>0</td>
<td>11</td>
<td>-10.1</td>
</tr>
<tr>
<td>3.984</td>
<td>3.811</td>
<td>1829</td>
<td>24.9</td>
<td>N/A</td>
<td>0</td>
<td>0</td>
<td>13</td>
<td>-4.5</td>
</tr>
<tr>
<td>2.063</td>
<td>2.274</td>
<td>246</td>
<td>25.0</td>
<td>N/A</td>
<td>0</td>
<td>30</td>
<td>11769</td>
<td>-9.2</td>
</tr>
<tr>
<td>4.289</td>
<td>4.397</td>
<td>1744</td>
<td>25.0</td>
<td>N/A</td>
<td>0</td>
<td>30</td>
<td>11779</td>
<td>-2.5</td>
</tr>
<tr>
<td>2.311</td>
<td>2.424</td>
<td>224</td>
<td>25.0</td>
<td>N/A</td>
<td>0</td>
<td>60</td>
<td>12790</td>
<td>-4.7</td>
</tr>
<tr>
<td>4.729</td>
<td>4.865</td>
<td>1748</td>
<td>25.1</td>
<td>N/A</td>
<td>0</td>
<td>60</td>
<td>12793</td>
<td>-2.8</td>
</tr>
<tr>
<td>$K_{SDM}$</td>
<td>$K_{EXP}$</td>
<td>$Re$</td>
<td>$T_1$</td>
<td>$T_2$</td>
<td>$\alpha$</td>
<td>$\beta$</td>
<td>Laboratory</td>
<td>$\Delta K$ [%]</td>
</tr>
<tr>
<td>----------</td>
<td>----------</td>
<td>------</td>
<td>-------</td>
<td>-------</td>
<td>---------</td>
<td>---------</td>
<td>------------</td>
<td>---------------</td>
</tr>
<tr>
<td>(10^4 cm/s)</td>
<td>(10^4 cm/s)</td>
<td>[-]</td>
<td>[°C]</td>
<td>[°C]</td>
<td>[°]</td>
<td>[°]</td>
<td>Exp. #</td>
<td>(Exp. = 100%)</td>
</tr>
<tr>
<td>2.565</td>
<td>2.586</td>
<td>245</td>
<td>24.9</td>
<td>N/A</td>
<td>0</td>
<td>90</td>
<td>13209</td>
<td>-0.9</td>
</tr>
<tr>
<td>5.115</td>
<td>4.949</td>
<td>1632</td>
<td>25.0</td>
<td>N/A</td>
<td>0</td>
<td>90</td>
<td>13213</td>
<td>+11.1</td>
</tr>
</tbody>
</table>

Examination of the results of 24 numerical simulations ($\alpha = 0°$, $\beta = 0°$, 30°, 60° and 90°), which includes the information given in Table 6.9.1.1, shows that for laminar isothermal flow conditions, the maximum difference between the experimentally and the numerically obtained overall mass transfer coefficient is approximately ±11%.

Pickett and Ong (1974) studied the cathodic reduction of m-nitrobenzene sulphonate acid (sodium salt) in a vertical electrochemical flow cell with separate anode and cathode compartments (40 mm width x 7 mm height) under limiting current conditions. The entrance length to equivalent diameter ratio was approximately 77. Values for average mass transfer coefficients were reported which were obtained by calculating mass transfer rates to electrodes of various length ($L = 2, 10.3, 30.5, 60, 90, 120, 149$ mm). Using the physical properties for this electrolyte system reported by Postlethwaite et al. (1972) and the mass transfer coefficients given by Pickett and Ong (Pickett and Ong, 1974, Figure 3), the corresponding local Sherwood numbers were calculated. These Sherwood numbers can then be compared to the numerically obtained local Sherwood numbers for isothermal vertical electrolyte flow ($\beta = 90°$) under limiting current conditions (Figure 6.9.1.2). The data of Pickett and Ong's results (Re = 100 and Re = 2000) are compared to the results of the simulation for the isothermal experiments 13209 (Re = 245) and 13212 (Re = 1632), respectively. Although the numerical conditions (Reynolds number) could have been set to compare directly with the results presented by Pickett and Ong (1974), it was decided to use conditions in the close vicinity of the above which were at the same time investigated in the experimental part of this study (i.e. experiments 13209 and 13212). Figure 6.9.1.2 indicates that Pickett and Ong's results for the Reynolds number of 100 are, as expected, slightly lower than the numerical results for
FIGURE 6.9.1.1 Local Isothermal MTC (Simulation)
FIGURE 6.9.12 Numerically obtained Local Sh.-Numbers compared to Pickett and Ong (1974)

\[ \beta = 90^\circ \]

- \( 13209 \) (Re = 2452)
- \( 13212 \) (Re = 1632)
- Pickett and Ong (Re = 100)
- Pickett and Ong (Re = 2000)

Cathode Position [cm] (x-Direction)

Local Sherwood Number [-]
Re = 245 (13209) while those at Re = 2000 are higher than the simulated values for Re = 1632. At 3 cm from the upstream edge of the cathode (Re = 100) the local Sherwood number reaches a constant value of approximately 60. As the Reynolds number value increases this point will shift more to the downstream edge of the cathode. It can be concluded that the numerical results agree well with those reported by Pickett and Ong. The only exception is the local mass transfer coefficient at the upstream edge of the electrode, which is approximately 20% larger than the one obtained through numerical analysis. This discrepancy may be explained by such differences between the laboratory experiment and the numerical model as:

i. Small effects by excluding the third dimension in the numerical analysis

ii. The 'hard' boundary condition at the upstream edge of the cathode (\( C_a = 0 \)) caused the numerical result, as discussed before, to be 'noisy' for the first \( \approx 3 \) mm.

iii. The Plexiglas\(^{-\text{TM}}\) - cathode assembly fit was assumed an ideal one but in the laboratory set-up a small but noticeable gap between the Plexiglas\(^{-\text{TM}}\) - cathode assembly was needed to compensate for thermal expansion of the cathode. This might have caused some additional disturbances in the flow field, which were not accounted for in the model.

This discrepancy in the local mass transfer coefficient at the upstream edge of the electrode may also attribute to the differences found between the numerical and experimental results obtained during the course of this project.

The local Sherwood numbers close to the upstream edge of the cathode are greater than those further downstream due to the much thinner mass transfer boundary layer near the electrode upstream edge. For the numerically derived data, local mass transfer coefficients at the position \( x > 13 \) cm are approximately the same, which implies that fully developed mass transfer will occur at \( L/D_e \) in the range of 8 to 10. This seems to agree very well with mass transfer data in fully developed hydrodynamic flow reported by Pickett and Ong (1974) and Schütz (1964) for channel flow and pipe flow, respectively.
6.9.2 Results of the Non-Isothermal Laminar Simulations

The 2-dimensional non-isothermal laminar numerical simulation (x, y direction) was performed for a cathode angle (\(\alpha\)) of 0°, channel slopes (B) of 0, 30, 60 and 90°, \(T_1 = 25^\circ\text{C}\), \(T_2 = 35, 55^\circ\text{C}\), and \[200 \leq \text{Re} \leq 1900\].

For any of the investigated non-isothermal laminar conditions the mass transfer coefficients close to the upstream edge of the electrode are greater than those further downstream due to the much thinner mass transfer boundary layer near to the electrode leading edge. Figure 6.9.2.1 shows typical values of local mass transfer coefficient calculated for various channel elevations (B). As discussed in the previous section, the local mass transfer values at the upstream edge of the electrode (x = 0) were omitted. The influence of the channel slope (B) on the local mass transfer coefficient should be noted. With increasing channel slope (B) fully developed mass transfer was found to appear closer to the downstream edge of the mass transfer surface. Unlike the isothermal case (see Figure 6.9.1.1), the local mass transfer coefficients did not reach their fully developed ("final") value. It can be said that the mass transfer boundary layer for the non-isothermal laminar flow region was in the developing stage and only mass transfer entrance effects were observed. With a fully developed hydrodynamic boundary layer and the thermal- and concentration boundary layer development starting at the same point, this observation suggests that a fully developed mass transfer boundary layer will not be encountered in electrochemical reactors with a mass transfer surface to equivalent diameter ratio of less than 9 (L/d_e ≤ 9).

Table 6.9.1.1 offers a comparison between experimentally (K_{exp}) and numerically (K_{sim}) obtained average mass transfer coefficients for non-isothermal laminar mass transfer in a horizontal flow reactor (\(\alpha = 0^\circ\), \(B = 0^\circ\)).
Re=400
Sc=1270
α=0°
ΔT=30 °C

—N59 - β=0°
...N1266 - β=30°
-N2051 - β=60°
-N2344 - β=90°

FIGURE 6.9.2.1 Local Non-Isothermal MTC (Simulation)
Table 6.9.2.1  Comparison of Experimentally and Numerically obtained Average Mass Transfer Coefficients for the Laminar Non-Isothermal Case of $\alpha = 0^\circ$, $\beta = 0^\circ$, 30°, 60°, 90°

<table>
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<th>$K_{\text{num}}$ $(10^4 \text{ cm/s})$</th>
<th>$K_{\text{exp}}$ $(10^4 \text{ cm/s})$</th>
<th>Re</th>
<th>$T_1$ [°C]</th>
<th>$T_2$ [°C]</th>
<th>$\alpha$</th>
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<th>Exp. #</th>
<th>$\Delta K$ [%] (Exp. = 100%)</th>
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Examination of the results of 17 numerical simulations ($\alpha = 0^\circ$, $\beta = 0^\circ$, 30°, 60° and 90°), which includes the information given in Table 6.9.2.1, shows that for laminar non-isothermal flow conditions, the maximum difference between the experimentally and the numerically obtained overall mass transfer coefficient is about ±9%. Some of the differences between the non-isothermal laminar experimental and 2-dimensional numerical simulation results may be attributed to:

i. Effects of the third dimension

ii. The 'hard' boundary condition at the upstream edge of the cathode ($C_p = 0$)

iii. The heat flux through the base of the cathode was assumed to be constant in the course of one laboratory experiment, while the numerical simulation assumed a constant temperature condition.

iv. The Plexiglas™- cathode assembly fit was assumed to be ideal but the actual apparatus...
had a small but noticeable gap between the Plexiglas™ and the cathode. This was present to allow for thermal expansion of the cathode, and might have caused additional disturbances in the flow field which were not accounted for in the model.

v. Even after careful machine shop work, small but noticeable surface 'roughnesses' were detected in the flow channel and on the electrode surface. These 'roughnesses' may have caused some deviation from the assumed ideal conditions.

vi. Experimental error, especially of the cathode surface temperature measurement. A detailed estimation of all experimental errors is given in Appendix J.

The convergence of the numerical solution was found to be very susceptible to the chosen initial values and the relaxation factors during the course of the 2-and 3-dimensional analysis. As discussed in section 6.8, for faster convergence in future analyses it might be more advisable to use the non-dimensional governing equations in order to avoid larger differences (up to three order of magnitudes) between individual model parameters (e.g. density and viscosity values).

6.9.3 Results of the Isothermal and Non-isothermal Turbulent Simulations

To perform isothermal or non-isothermal simulations the governing equations (6.2.2.4 to 6.2.2.9) will have to be solved. These equations contain empirical constants (see section 6.2.2). A thorough literature search was unsuccessful in locating any work which would have allowed prediction or reasonable estimation of the values for these empirical factors.

Over the years, the k-ε model has been tested, optimized and fine tuned against a wide range of flows of practical interest. Fluid Dynamics International (1990) has accumulated the existing information and implemented it into FIDAP™ as default values for the k-ε model. Fluid Dynamics International however emphasized that these default model constants are optimized for the adequate prediction of a wide range of flows. It was stated that the performance of the k-ε model is known to be very sensitive to small changes in some of the model constants (the concentration in particular). Thus, strictly speaking, for a given flow problem, an investigator may have to fine-tune these constants to obtain
better agreement between the model predictions and experimental results. In the absence of empirical data, an effort to obtain them from ad hoc numerical tuning was recommended.

Due to the computer limitations encountered in the study and the lack of the 'eddy' data, only one preliminary numerical simulation was performed. Using the 'trial and error technique' to adjust the FIDAP™ constants, a physically meaningful result for the isothermal experiment (I3196) was obtained. As shown in Figure 6.9.3.1, the local Sherwood numbers were plotted as a function of the cathode position. For comparison the local Sherwood numbers obtained by Pickett and Ong (1974) were included for completeness. For developing isothermal turbulent mass transfer in a vertical flow channel, the numerical data are in good agreement with one set presented by Pickett and Ong. Figure 6.9.3.1 shows also, that the difference between the overall Sherwood numbers for the isothermal turbulent experiment I3196, obtained in the experimental investigation and during the course of the numerical analysis, is approximately 8%. No claim can be made in this study that a complete turbulent numerical analysis was performed. The only intention in carrying out this single simulation run was to indicate that, with more information and computing power available, the turbulent problem can be solved in future studies.
CHAPTER SEVEN
CONCLUSIONS

The results of the three thousand (3277) isothermal laminar and turbulent mass transfer experiments that were performed are first considered. Observations were made at Schmidt numbers from 800 to 2300 and Reynolds numbers from 200 to 32000 by variation of temperature and flow rate. The bulk solution temperature was varied from 25°C to 55°C. The transition from the laminar to the turbulent flow region in this study was found to occur at a Reynolds number range from 1800 to 2000. These results, which are lower than the generally accepted Reynolds number transient value of 2300 for pipe flow, may be explained by the effect of the rectangular cross-section in the channel employed here in creating corner turbulent effects at lower flow rates than in a pipe with circular cross-section.

For comparison with existing work, Figure 5.3.1.1 has shown typical (25°C) results on horizontal and vertical channel to be in satisfactory agreement, validating the experimental apparatus and techniques employed here. Since no comparable data for forced convective mass transfer at various slopes and angles of channel or flat plate exist, the empirical correlations presented here (Equations 5.3.1.3, 5.3.1.4b, 5.3.2.2 and 5.3.2.3b) give the first comprehensive presentation for the effect of configurational changes in the flow channel (rotation and slope). The entire picture has been summarized by presenting the correlations in the form of 3-dimensional figures, Figures 5.3.1.6 and 5.3.2.3 for laminar and turbulent flow respectively. Applicable for the range of Reynolds numbers and Schmidt numbers under investigation, but restricted to a developing diffusion boundary layer regime, increases in mass transfer at the liquid-solid interface can be as high as 90%. The combined effects of channel rotation and channel slope are thus now clearly portrayed. This almost doubling of mass transfer rates that can be achieved provides the bounds for flow reactor design where mass transfer at the solid-liquid interface is of major interest.
Mechanistically the effect on mass transfer can be understood, in a simplified way, through the secondary flows involved at the electrode surface due to the effect of the small density differences arising with the formation of the diffusion boundary layer. These are magnified both by rotation of the cathode surface towards the vertical and also by elevation of the surface towards the vertical. While flow visualisation studies would be needed to examine this in detail, the increased mass transfer is clearly due to changes in such secondary flows corresponding to the channel configurational pattern.

The some two thousand (2394) non-isothermal experiments, performed with the bulk solution maintained at 25°C and the cathode surface temperature varied from 35°C to 55°C, introduced additional major effects. The thermal gradient thus established introduced a developing thermal boundary layer with resulting convective effects. An important observation, which may be noted from Figure 5.4.2.1, was the result that mass transfer in the laminar flow regime, at Re ≤ 1900 (Range: 1800 - 2000), is independent of the Reynolds number. Wragg and Ross (1967), who studied superposed laminar free and forced convective mass transfer in a vertical annular flow reactor, found a significant increase of mass transfer due to free convective effects. Most importantly they concluded that mass transfer in the laminar flow region seemed to be controlled solely by natural convection. Thus in applications in which the flow velocity is sufficiently low and temperature differences are encountered, the natural convection effect must also be significant. This would be expected in the present work which was concerned mainly with the effect of the thermal gradient on the overall mass transfer coefficient for flow in a rectangular duct. For a fully developed hydrodynamic boundary layer, with simultaneous developing concentration and thermal boundary layers, Reynolds numbers from 200 to 1900, Schmidt numbers from 800 to 2300 and combined Grashof numbers from 2·10^4 to 9·10^4, the experimental data for laminar non-isothermal mass transfer have been successfully correlated. As previously noted (Section 3.4) the proposal for a combined Grashof number to relate the free convective effects arising from simultaneous heat and mass transfer was first made by Mathers et al. (1957). Here, the combined Grashof number, with the equivalent diameter as the characteristic length (Equations 5.4.2.1b and 5.4.2.2b) has been used. These equations cover the entire range of configurational affects of rotation and slope of the surface for laminar flow conditions. The
3-dimensional picture, Figure 5.4.2.6, has illustrated the full range that has been explored. The success of this approach in correlating data for a wide range of conditions give further substantiation to the predominant role played by natural convection in laminar flow situations.

The flow patterns to be expected under these circumstances of combined heat and mass transfer at various rotational angles (α) and channel elevations (β) can be considered in a simplified way by reference to Figure 7.1:

At α = 0°, the secondary flows in a horizontal flow channel with the an upwards facing heated mass transfer surface are of a column type. The less dense material, rising from the mass transfer surface, will superimpose a vertical velocity component to the main bulk flow. This could cause some recirculation effects close to the vertical channel walls. However, if the mass transfer surface covers only a fraction of the entire channel width, as in the work of Ajersch (1990), it is expected that the recirculation effects will further increase along the side of the mass transfer surface. This is described further, below.

At α = 90°, one of the vertical walls is the heated mass transfer surface. The less dense material will raise along the vertical surface due to the existing buoyancy forces. The vertical fluid motion combined with the bulk flow would appear to cause a 'spiral' type fluid flow.

At α = 180°, the heated mass transfer surface is facing downwards in the flow channel. During this type of operation a semi-stable layer covering the mass transfer surface is being formed. Within this layer small vortices may form causing the liquid to mix with the adjacent 'fresh' bulk electrolyte.

For rotational angles (α), other than the above, a combination of the described secondary flow effects is expected to occur.

The effect of the rotational angle (α) on the secondary flows will have superimposed on it the phenomena introduced by the channel elevation (β). At 0° ≤ β ≤ 90°, a formation of longitudinal vortices along the mass transfer surface is expected to occur.

The present data are approximately 10% lower than those presented by Ajersch (1990) (Figure
5.4.2.1). The flow adjacent to a heated mass transfer surface is thought to be of a laminar convection boundary layer type but at low Reynolds numbers the probability of having recirculation is high. The flow pattern is greatly affected by the natural convection effects which may create secondary flows. These secondary flows will convect additional 'fresh' electrolyte to the mass transfer surface. The development of those secondary flows will be greatly influenced by the ratio of the electrode width to the channel width. To study the wall effects on the mass transfer coefficient, the ideal arrangement would consist of electrodes of finite width in an infinite wide flow channel. In addition to the streamline forced convective bulk flow, ‘fresh’ electrolyte will be convected by means of secondary flow pattern from the outside to the mass transfer surface (Figure 7.1). However in a flow arrangement with the electrode covering the whole width of the flow channel these secondary flows will be reduced. Since Aijersch’s work employed an apparatus with an electrode width to channel width ratio of 0.4 and the present study investigated mass transfer at an electrode which covered the whole width of the channel, these reduced secondary flows in the latter system would appear to account for the decrease in the overall mass transfer coefficient. No other non-isothermal free and forced convective mass transfer data for channel flow have been found in the literature.

In the turbulent flow region, for Reynolds numbers greater than 1900 (Range: 1800 -2000), the Sherwood number, as indicative of mass transfer, increases with increasing flow rates (e.g. Figure 5.4.2.1). It can be concluded that the mass transfer is controlled by both free and forced convection, and that the forced convective effects are dominant. This claim can be further substantiated by examining the exponents of the (GRSc) group and the Reynolds number in regression equations 5.4.3.1b and 5.4.3.2b. While the exponent of the (GRSc) group, which is very small (0.06), indicates an almost negligible contribution of the buoyancy forces on the overall mass transfer; the exponent of the Reynolds number (0.66), an indication of the forced convective component on the overall mass transfer, has essentially reached the value proposed by Pickett (1977) for mass transfer in the turbulent flow regime.

For the extensive conditions under investigation (200 ≤ Re ≤ 32000; 800 ≤ Sc ≤ 2300; 0° ≤ α ≤ 180°; 0° ≤ β ≤ 90°) it may be concluded that non-isothermal operation can enhance mass
transfer at a liquid-solid interface approximately four times over the isothermal case under the optimum selection of channel surface rotation, channel elevation, temperature gradient and flow conditions (Figure 5.4.3.3). These significant increases can be clearly understood mechanistically through consideration of the secondary flows caused by the joint introduction of both thermal and diffusion boundary layers on a section of the channel flow system. Overall the results now obtained provide new comprehensive guidance for optimizing design of flow reactors in which mass transfer at the liquid-solid interface is operationally significant.

The theoretical analysis that was performed to determine the local mass transfer coefficients in the rectangular channel operated under laminar isothermal and non-isothermal flow conditions was based on a computational study of the flow system. The coupled governing equations, which were derived from first principles, were solved by using the FIDAP™ finite element software package. However, limitations in the computer resources allowed only for an analysis of the influence of the channel elevation and not of rotational effects on the local and overall mass transfer coefficient.

A similar limitation led to a 2-dimensional analysis as described in section 6.9.1. The justification for omitting the dimension in the z-direction was demonstrated, when the local mass transfer coefficients for the 2-dimensional infinite wide channel (z-direction) were compared to those obtained through a 3-dimensional analysis in the centre of the channel (y → 0, z = 1 cm). An important observation from this comparison, which may be noted from Figure 6.9.2, was that the local mass transfer coefficients in the centre of the channel (y → 0, z = 1 cm) are not influenced by the effect of the channel walls.

The computer simulation provided an insight on the dependence of the local laminar isothermal mass transfer coefficients on flow channel elevation. In agreement with the experimental laminar isothermal results, the overall mass transfer coefficient showed an increase with increasing channel slope (θ → 90°). Figure 6.9.1.1 indicates that the local mass transfer coefficient is a function both of distance along the electrode and the channel slope. It also appears that the concentration boundary layer develops more slowly with increasing channel slope. For such conditions the flow adjacent to the mass transfer surface seems is increasingly affected by natural convection effects which create longitudinal vortices.
Comparison of the local mass transfer coefficients with available experimental information in the literature (Pickett and Ong, 1974) clearly showed that these predictions are accurate within the limitations under which they were made. As discussed by Pickett (1977), Fahidy (1985) and others, such results indicate that the thickness of the concentration boundary layer at any specified position along the electrode decreases with increasing slope. The average mass transfer coefficients numerically derived in this simulation agreed within ±11% of the experimental values obtained as shown in Table 6.9.1.1. This is a satisfactory validation of the model that has been derived and applied by using FIDAP™.

Local non-isothermal mass transfer coefficients were also computed for channel elevations (β) ranging from 0° to 90°. The results as given in Figure 6.9.2.1 revealed that an increase in channel slope resulted in an increase in the overall mass transfer coefficient. Comparison between the isothermal and the non-isothermal local mass transfer coefficients show, that the non-isothermal boundary layer development with respect to the position along the electrode length is much slower than for the comparable isothermal case. It is thus clear that both the thermal gradient and the channel slope are important factors in establishing the magnitude of both the local and overall mass transfer coefficients. Secondary flow patterns determine the development of the concentration and thermal boundary layers. The numerically derived average mass transfer coefficients agreed within ±9% of the experimental values obtained in this study which again is excellent validation of the simulation model.

While average mass transfer coefficients were obtained experimentally, numerical simulations of the local mass transfer coefficients have been made with 2-dimensional restrictions. While the conditions were for both developing thermal and diffusion boundary layer, there is clear evidence in the simulated local mass transfer coefficients that they have reached or almost reached their final developed values. Thus values of mass transfer coefficients for fully developed thermal and diffusion boundary layers can be determined through this approach. Although the simulations that have been made are limited in range as described above, the deduction that they can provide mass transfer coefficients under fully developed conditions is important. A tabulation of the simulation results has therefore been provided in Appendix M for reference.
No analytical solution for the turbulent flow in the rectangular channel could be presented due to the lack of information on some of the modelling constants required for the governing equations. However one numerical simulation for the isothermal turbulent flow case was accomplished using a 'trial and error' approach. In this case the local mass transfer coefficients were found to be in good agreement with those presented by Pickett and Ong (1974). Such an approach indicates that derivations in this way may be promising for the much more complex turbulent systems.

This thesis has presented an analytical and experimental investigation of mass transfer in a channel reactor at various surface angles ($\alpha$) and channel elevations ($\beta$). Due to the experimental and computational limitations encountered during the course of this project, many questions are still unanswered. Future research activities related to this study could include:

i. measurement of the local mass transfer coefficients in the flow reactor with developing thermal and diffusion boundary layer under isothermal and non-isothermal conditions

ii. measurement of the local and average mass transfer coefficients in the flow reactor with fully developed thermal and diffusion boundary layers under isothermal and non-isothermal conditions

iii. measurement of the local and average mass transfer coefficients in the flow reactor with developing hydrodynamic, thermal and diffusion boundary layers under isothermal and non-isothermal conditions

iv. the analysis of secondary flows caused by the density gradients using e.g. a flow visualisation

v. an experimental/numerical investigation to determine the model constants needed for the turbulent flow simulations

vi. an isothermal and non-isothermal 3-dimensional numerical analysis and simulation for the whole range of surface angles and channel slopes, especially for $0^\circ \leq \alpha \leq 180^\circ$ and $60^\circ > \beta < 90^\circ$

vii. boundary layer stability studies which inevitably arise in connection with such flows.
APPENDIX A

ESTIMATION OF ELECTROLYTE PROPERTIES

A.1 FLUID PROPERTIES SPREAD SHEET

Densities, viscosities, diffusivities, thermal conductivities and heat capacities were taken from Eisenberg et al. (1956), Sutey and Knudsen (1967), Reiss et al. (1963), Bourne et al. (1985), Storck and Coeuret (1977), Bazán and Arvía (1965), Landolt-Börnstein (1936) and Riedel (1950) for various caustic soda/potassium ferricyanide/potassium ferrocyanide electrolyte solutions compositions, as summarized in Table A.1.1. The reference numbers in Table A.1.1 correspond to:

1: Eisenberg, Tobias and Wilke (1956)
2: Sutey and Knudsen (1967)
3: Reiss, Hanratty and van Shaw (1963)
4: Bourne, Dell’Ava, Dossenbach and Post (1985)
5: Storck and Coeuret (1977)
6: Bazán and Arví (1965)
7: Landolt-Börnstein (1936)
8: Riedel (1950)
Table A.1.1 Electrolyte Properties reported in the Literature

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A.2  ELECTROLYTE DENSITIES AND VISCOSITIES

The available literature data for densities (see section 3.5.2 and Table A.1) and viscosities (see section 3.5.3 and Table A.1) were correlated by performing multiple regression. All the density results are best described by

\[ \rho \left[ \frac{g}{cm^3} \right] = 0.001 (1006 + 41.9C_{NaOH} + 151.58C_{Ferm} + 233.95C_{Frem} - \\
\quad -0.267T - 0.691C_{NaOH}^2 + 9.550C_{Ferm}^2 - 82.73C_{Frem}^2 - 0.00229T^2) \]  
(A.2.1)

The papers above quoted were also used to estimate the electrolyte viscosity. Using the multiple regression analysis the results found in the literature were best correlated by

\[ \nu \left[ \frac{cm^2}{s} \right] = 0.02 + 0.00195C_{NaOH} + 0.0032C_{Ferm} - 0.00267C_{Frem} - 0.000632T - \\
\quad + 3.9 \cdot 10^{-5}C_{NaOH}^2 + 0.0029C_{Ferm}^2 + 0.0305C_{Frem}^2 + 7 \cdot 10^{-4}T^2 \]  
(A.2.2)

Since the regression coefficient, $r^2$, was 0.999 in both cases, it can be concluded that the solution properties calculated by equations A.2.1 and A.2.2 agree to the literature value to within 1%.

A.3  DIFFUSION COEFFICIENTS

Data reported in the literature about the diffusion coefficients of potassium ferro- and ferricyanide are for ternary solutions are tabulated in Table A.1.1. Based on these data and the correlation reported in section 3.5.1 the multiple regression correlation can be written as

\[ D \left[ \frac{cm^2}{s} \right] = 0.01 \left( 5.5207 \cdot 10^{-4} - 1.4448 \cdot 10^{-4}C_{NaOH} - 5.989 \cdot 10^{-4}C_{Ferm} - 3.367 \cdot 10^{-4}C_{Frem} - \\
\quad + 6.22 \cdot 10^{-6}T - 1.27 \cdot 10^{-6}C_{NaOH}^2 + 1.258 \cdot 10^{-3}C_{Ferm}^2 + 9.26 \cdot 10^{-4}C_{Frem}^2 + 1.7 \cdot 10^{-7}T^2 \right) \]  
(A.3.1)
The diffusion coefficient can be estimated by equation A.3.1 to within 1% of the values found in the literature.

A.4 THERMAL CONDUCTIVITIES AND HEAT CAPACITIES

As discussed in section 3.5.4, these solution properties were estimated using data available for pure aqueous sodium hydroxide solutions. The thermal conductivity of the electrolyte solution used in this study was estimated by:

\[
\lambda \left[ \frac{J}{\text{cm s}^2\text{C}} \right] = 0.01\left(0.5841 + 0.0063161 C_{\text{NaOH}} + 0.001541 T - 0.000283 C_{\text{NaOH}}^2 - 2.76 \cdot 10^{-4} T^2 \right)
\]

(A.4.1)

Equation A.4.2 was utilized to predict the heat capacity of the bulk solution:

\[
\epsilon_p \left[ \frac{J}{g \text{K}} \right] = 4.18 - 0.229 C_{\text{NaOH}} + 1.425 \cdot 10^{-4} T + 0.02903 C_{\text{NaOH}}^2 - 3.748 \cdot 10^{-13} T^2
\]

(A.4.2)

The regression coefficient was 0.999 in both correlations, enabling the calculated solution properties to agree to the literature value to within 1%.
APPENDIX B

ROTAMETER CALIBRATIONS

Three rotameters, with overlapping but different volumetric flow ranges, were used in the electrochemical reactor assembly. They were constructed from different glass tubes and stainless steel floats. The calibration was performed by using the "bucket and stopwatch" method with the appropriate safety precautions (full alkaline resistance coverall, rubber boots, rubber gloves, safety goggles and face shield). The flow rate, which was adjusted (limited) for each experiment by the use of an orifice, was found to vary linearly with the scale reading in all three measurement ranges. The calibration curves for 25°C and a nominal electrolyte solution (2 mol/l NaOH, 0.025 mol/l K₄[Fe(CN)₆], 0.025 mol/l K₃[Fe(CN)₆]) and the regression equations for the three rotameters (Small, Medium and Large) is shown in Figure B.1. The same procedure was employed for volumetric flow measurements at different temperature readings of 30°C to 55°C performed in 5°C increments.

The volumetric flow results for the small, medium and large rotameter calibration are best correlated by equations B.1 to B.3, respectively.

\[
\dot{V} \left[ \frac{m^3}{s} \right] = 0.4989 \cdot 10^{-6} \cdot S_S \sqrt{\frac{(7940 - \rho_B) \cdot 1085}{6855 \rho_B}} \\
\dot{V} \left[ \frac{m^3}{s} \right] = 1.7599 \cdot 10^{-6} \cdot S_M \sqrt{\frac{(7940 - \rho_B) \cdot 1085}{6855 \rho_B}} \\
\dot{V} \left[ \frac{m^3}{s} \right] = 6.0820 \cdot 10^{-6} \cdot S_L \sqrt{\frac{(7940 - \rho_B) \cdot 1085}{6855 \rho_B}}
\]

where \( \dot{V} \) is the volumetric flow rate in m³/s, \( \rho_B \) is the bulk density in kg/m³, and \( S_S \) [%], \( S_M \) [%], \( S_L \) [%]
are the small, medium and large rotameter readings, respectively.
FIGURE B.1 Rotameter Calibration

$T_B = 25^\circ C$

Small Rotameter
Medium Rotameter
Large Rotameter

Volume Flow Rate [$10^{-3}$ m$^3$/s]

Rotameter Reading [%]
APPENDIX C

SAMPLE CALCULATIONS

Apparatus Parameter:

Height of Channel:

\[ y_1 = 1.5 \text{ cm} \]  \hspace{1cm} (C.0)

Width of Channel:

\[ z_1 = 2.0 \text{ cm} \]  \hspace{1cm} (C.1)

Equivalent Diameter:

\[ d_e = 2 \frac{y_1 z_1}{y_1 + z_1} \frac{1.5 \text{ cm} \cdot 2.0 \text{ cm}}{1.5 \text{ cm} + 2.0 \text{ cm}} = 1.714 \text{ cm} \]  \hspace{1cm} (C.2)

Electrode Area:

\[ A = 15 \text{ cm} \cdot 2 \text{ cm} = 30 \text{ cm}^2 \]  \hspace{1cm} (C.3)

Raw Data (Experiment: NR1):

Rotameter : 24.9 cm³/s @ S-50

Voltage (Eₐ) : 50.26 mV

\[ T_1 : 25.1 \text{ °C} \]

\[ T_2 : 35.0 \text{ °C} \]

\[ T_3 : 36.2 \text{ °C} \]

\[ T_4 : 25.5 \text{ °C} \]
\[ C_{\text{NaOH}} : 1.9935 \text{ mol/l} \]
\[ C_{\text{ferr}} : 0.0248 \text{ mol/l} \]
\[ C_{\text{ferro}} : 0.0251 \text{ mol/l} \]

Limiting Current:

\[ I_L = \frac{E_L}{R_L} = \frac{50.26 \text{ mV}}{1.238 \Omega} = 40.60 \text{ mA} \]  \hspace{1cm} (C.4)

Electrolyte Properties:

Surface Temperature

\[ T_s = \frac{T_2 + T_3}{2} = \frac{35.0^\circ\text{C} + 36.2^\circ\text{C}}{2} = 35.6^\circ\text{C} \]  \hspace{1cm} (C.5)

Surface Ferrocyanide concentration:

\[ C_{\text{ferro}} = 0.0248 \frac{\text{mol}}{l} + 0.0251 \frac{\text{mol}}{l} = 0.0499 \frac{\text{mol}}{l} \]  \hspace{1cm} (C.6)

Film Temperature:

\[ T_F = \frac{T_1 + T_3}{2} = \frac{25.1^\circ\text{C} + 35.6^\circ\text{C}}{2} = 30.35^\circ\text{C} \]  \hspace{1cm} (C.7)

Film Ferrocyanide concentration:

\[ C_{\text{ferro}} = 0.0251 \frac{\text{mol}}{l} + 0.0124 \frac{\text{mol}}{l} = 0.0375 \frac{\text{mol}}{l} \]  \hspace{1cm} (C.8)
Film Ferricyanide concentration:

\[ C_{\text{Fricn}} = \frac{0.0248 \text{ mol}}{2} = 0.0124 \text{ mol} \]  

(C.9)

All of the following electrolyte properties and dimensionless numbers were calculated by means of the appropriated equations. The coefficients of the individual equations are displayed only for few significant digits but the final results of the computation were taken from the attached spread sheets were at least 9 or more significant figures were used (Lotus spread sheet files and 'Data Summary' (see Appendix L)).

Density:

\[
\rho \left[ \frac{g}{cm^3} \right] = 0.001 \left( 1006 + 41.9 C_{\text{Kodif}} + 151.58 C_{\text{Ferm}} + 233.95 C_{\text{Fero}} - 0.267 T - 0.691 C_{\text{Kodif}}^2 + 9.550 C_{\text{Ferm}}^2 - 82.73 C_{\text{Fero}}^2 - 0.00229 T^2 \right)
\]  

(C.10)

From equation (C.10) the electrolyte bulk density can be estimated.

\[
\rho_b = 0.001 \left( 1006 + 41.9 \cdot 1.9935 + 151.58 \cdot 0.0248 + 233.95 \cdot 0.0251 - 0.267 \cdot 25.1 ... - 0.691 \cdot 1.9935^2 + 9.55 \cdot 0.0248^2 - 82.73 \cdot 0.0251^2 - 0.00229 \cdot 25.1^2 \right)
\]

\[
\rho_b = 1.08822 \frac{g}{cm^3}
\]

The electrolyte densities for the film-, and surface conditions were estimated similarly to the above procedure.
Viscosity:

\[
\nu \left[ \frac{cm^2}{s} \right] = 0.02 + 0.00195C_{NaOH} + 0.0032C_{Jern} - 0.00267C_{jern} - 0.000632T - \\
+ 3.9 \cdot 10^{-3}C_{NaOH}^2 + 0.0029C_{Jern}^2 + 0.0305C_{jern}^2 + 7 \cdot 10^{-8}T^2
\]  

(C.11)

From equation (C.10) the electrolyte bulk density can be estimated.

\[

v_F = 0.02 + 0.00195 \cdot 1.9935 + 0.0032 \cdot 0.0124 - 0.00267 \cdot 0.0375 - 0.000632 \cdot 30.35 - \\
+ 3.9 \cdot 10^{-3} \cdot 1.9935^2 + 0.0029 \cdot 0.0124^2 + 0.0305 \cdot 0.0375^2 + 7 \cdot 10^{-8} \cdot 30.75^2
\]

\[
v_F = 0.01134 \frac{cm^2}{s}
\]

Diffusivity:

\[
D \left[ \frac{cm^2}{s} \right] = 0.01 \left[ 5.5207 \cdot 10^{-4} - 1.4448 \cdot 10^{-4} C_{NaOH} - 5.989 \cdot 10^{-4} C_{Jern} - 3.367 \cdot 10^{-4} C_{jern} - \\
+ 6.22 \cdot 10^{-8} T + 7.28 \cdot 10^{-9} C_{NaOH}^2 + 1.258 \cdot 10^{-5} C_{Jern}^2 + 9.26 \cdot 10^{-8} C_{jern}^2 + 1.7 \cdot 10^{-7} T^2 \right]
\]

(C.12)

\[
D_F = 0.01 \left[ 5.5207 \cdot 10^{-4} - 1.4448 \cdot 10^{-4} \cdot 1.9935 - 5.989 \cdot 10^{-4} \cdot 0.0124 - 3.367 \cdot 10^{-4} \cdot 0.0375 + 6.22 \cdot 10^{-8} \cdot \\
- 30.35 + 7.28 \cdot 10^{-9} \cdot 1.9935^2 + 1.258 \cdot 10^{-5} \cdot 0.0124^2 + 9.26 \cdot 10^{-8} \cdot 0.0375^2 + 1.7 \cdot 10^{-7} \cdot 30.35^2 \right]
\]

\[
D_F = 6.239 \cdot 10^{-4} \frac{cm^2}{s}
\]

Thermal conductivity:

\[
\lambda \left[ \frac{J}{cm \cdot s \cdot ^\circ C} \right] = 0.01 \left( 0.5841 + 0.0063161C_{NaOH} + 0.001541T - 0.000283C_{NaOH}^2 - 2.76 \cdot 10^{-8} T^2 \right)
\]

(C.13)

\[
\lambda = 0.01 \left( 0.5841 + 0.0063161 \cdot 1.9935 + 0.001541 \cdot 30.35 - 0.000283 \cdot 1.9935^2 - 2.76 \cdot 10^{-8} \cdot 30.35^2 \right)
\]

\[
\lambda = 0.006398 \frac{J}{cm \cdot s \cdot ^\circ C}
\]
Heat capacity:

\[ c_p \left[ \frac{J}{g \cdot K} \right] = 4.18 - 0.229 C_{NaOH} + 1.425 \cdot 10^{-3} T + 0.02903 C^2_{NaOH} - 3.748 \cdot 10^{-13} T^2 \]  \hspace{1cm} (C.14)

\[ c_p = 4.18 - 0.229 \cdot 1.9935 + 1.425 \cdot 10^{-3} \cdot 30.35 + 0.02903 \cdot 1.9935^2 - 3.748 \cdot 10^{-13} \cdot 30.35^2 \]

\[ c_p = 3.8377 \frac{J}{g \cdot K} \]

The individual electrolyte properties, which have been calculated using the above equations, are summarized in Table C.1.

**Table C.1 Summary of the Electrolyte Properties for Experimental Run NR1**

<table>
<thead>
<tr>
<th>Properties</th>
<th>Bulk</th>
<th>Surface</th>
<th>Film</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>( C_{NaOH} ) [mol/l]</td>
<td>1.9935</td>
<td>1.9935</td>
<td>1.9935</td>
<td></td>
</tr>
<tr>
<td>( C_{act} ) [mol/l]</td>
<td>0.0248</td>
<td>0</td>
<td>0.0124</td>
<td></td>
</tr>
<tr>
<td>( C_{rem} ) [mol/l]</td>
<td>0.0251</td>
<td>0.0499</td>
<td>0.0375</td>
<td></td>
</tr>
<tr>
<td>( T ) [°C]</td>
<td>25.1</td>
<td>35.6</td>
<td>30.35</td>
<td></td>
</tr>
<tr>
<td>( \rho ) [g/cm³]</td>
<td>1.08822</td>
<td>1.09011</td>
<td>1.08615</td>
<td>( \rho ) evaluated at ( T_B )</td>
</tr>
<tr>
<td>( v ) [cm²/s]</td>
<td>0</td>
<td></td>
<td>0.01134</td>
<td></td>
</tr>
<tr>
<td>( D ) [cm²/s]</td>
<td></td>
<td></td>
<td>6.239\cdot 10^{-4}</td>
<td></td>
</tr>
<tr>
<td>( \lambda ) [J/(cm s K)]</td>
<td>0.006398</td>
<td></td>
<td>0.006398</td>
<td></td>
</tr>
<tr>
<td>( c_p ) [J/(g K)]</td>
<td></td>
<td></td>
<td>3.8377</td>
<td></td>
</tr>
</tbody>
</table>
Calculations of Dimensionless Numbers

**Bulk velocity, U:**

\[
U = \frac{\dot{V}}{\nu_{11} \cdot \nu_{12}} = \frac{24.9 \frac{cm^3}{s}}{1.5 cm \cdot 2.0 cm} = 8.3 \frac{cm}{s}
\]  
(C.15)

**Reynolds number:**

\[
Re = \frac{U d_s}{\nu_f} = \frac{8.3 \frac{cm}{s} \cdot 1.714 cm}{0.01134 \frac{cm^2}{s}} = 1257
\]  
(C.16)

**Schmidt number:**

\[
Sc = \frac{\nu_f}{D_F} = \frac{0.01134 \frac{cm^2}{s}}{6.239 \cdot 10^{-6} \frac{cm^2}{s}} = 1802
\]  
(C.17)

**Prandtl number:**

\[
Pr = \frac{c_p \rho_f \nu_f}{\lambda}
\]

\[
Pr = \frac{3.8377 \frac{J}{g \cdot cm^2 \cdot s} \cdot 1.08615 \frac{g}{cm^3} \cdot 0.01134 \frac{cm^2}{s}}{0.006398 \frac{J}{cm \cdot s \cdot K}}
\]

\[
Pr = 7.39
\]
Sherwood number:

\[ Sh = \frac{I_e d_e}{z_e F DA_c c_{in-b}^m} \]  
(C.19)

\[ Sh = \frac{40.6 \cdot 10^{-3} A \cdot 1.714 \text{ cm}}{1 \cdot 96487 \frac{As}{mol} \cdot 6.239 \cdot 10^{-6} \text{ cm}^2 \cdot 0.0248 \frac{mol}{1000 \text{ cm}^3} \cdot 30 \text{ cm}^2 \cdot 0.0248 \frac{mol}{1000 \text{ cm}^3}} \]

\[ Sh = 154 \]

Density difference:

\[ \Delta \rho_e = \rho_g - \rho_s = 1.08822 \frac{g}{\text{cm}^3} - 1.09011 \frac{g}{\text{cm}^3} = -0.00189 \frac{g}{\text{cm}^3} \]  
(C.20)

Grashof number of mass transfer:

\[ Gr_e = \frac{g d_e^2 \Delta \rho_e}{\nu^2 \rho_f} \]  
(C.21)

\[ Gr_e = \frac{981 \frac{cm}{s^2} \cdot 1.714 \text{ cm}^3}{\left(0.01134 \frac{cm^2}{s}\right)^2 \cdot 1.08615 \frac{g}{\text{cm}^3}} \left(-0.00189 \frac{g}{\text{cm}^3}\right) = -66570 \]
Expansion:

\[
\left( \frac{dp}{dT} \right)_p = -4.671 \cdot 10^{-4} \frac{g}{cm^3 \cdot ^\circ C}
\]  \hspace{1cm} (C.22)

Grashof number of heat transfer:

\[
Gr_i = \frac{gd_s^3 \Delta \rho_T}{v^2 \rho_f} = \frac{gd_s^3}{v^2 \rho_f} \left( \frac{dp}{dT} \right)_p (T_s - T_0)
\]  \hspace{1cm} (C.23)

\[
Gr_i = \frac{981 \frac{cm}{s^2} \cdot 1.7714 \frac{cm^3}{s^3}}{0.01134 \frac{cm^2}{s}} \cdot 1.08615 \frac{g}{cm^3} \cdot 4.671 \cdot 10^{-4} \frac{g}{cm^3 \cdot ^\circ C} \cdot (25.1^\circ C - 35.6^\circ C)
\]

\[
Gr_i = 1.734 \cdot 10^4
\]

Combined Grashof number:

\[
GR_c = Gr_i \left( \frac{Sc}{Pr} \right)^{0.5} Gr_i \hspace{1cm} (C.24)
\]

\[
GR_c = -66841 + \left( \frac{1817}{7.38} \right)^{0.5} \cdot 1.734 \cdot 10^4 = 2.641 \cdot 10^6
\]
APPENDIX D
DATA FILE SYSTEM

To simplify future access to the experimental data obtained in this study all the information listed in the 'Data Summary' (see Appendix L) were entered in Lotus-123® Release 3.1 spread sheet files and stored on floppy disks. A summary of the spread sheet file sizes containing the experimental data is given in Table D.1. All diskettes are stored at the Research Supervisors office.

Table D.1 Spread Sheet File Size

<table>
<thead>
<tr>
<th>Spread Sheet File Content</th>
<th>File Name</th>
<th>File Size [kB]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Isothermal Preliminary Experiments (IP1..IP356)</td>
<td>GBIP1.WK3</td>
<td>620</td>
</tr>
<tr>
<td>Isothermal Replicate Experiments (IR1..IR200)</td>
<td>GBIR1.WK3</td>
<td>350</td>
</tr>
<tr>
<td>Isothermal Experiments (II..I3277)</td>
<td>GBII4.WK3</td>
<td>6470</td>
</tr>
<tr>
<td>Non-Isothermal Preliminary Experiments (NP1..NP504)</td>
<td>GBNP1.WK3</td>
<td>1512</td>
</tr>
<tr>
<td>Non-Isothermal Replicate Experiments (NR1..NR150)</td>
<td>GBNR1.WK3</td>
<td>332</td>
</tr>
<tr>
<td>Non-Isothermal Experiments (N1..N2394)</td>
<td>GBN4.WK3</td>
<td>7350</td>
</tr>
</tbody>
</table>

The Microsoft MS-DOS® (Version 5.0) BACKUP utility was used to transfer these spread sheet data files onto 3.5" 1.44 MB high density floppy diskettes. The procedure for restoring these data files to a hard disk is given below.

The DOS® RESTORE command restores one or more files from the backup disks. The format
of the command is

```
[drive:][path] RESTORE dl: file_spec [/P][/S]
```

where the following is true:

i. *drive:* is the disk drive containing the file RESTORE.COM. RESTORE is an external DOS* command; therefore, DOS* must read the file from disk into memory each time the command is invoked. If no disk is specified, DOS* uses the current default drive.

ii. *path* is a series of subdirectories DOS* must traverse to find the file RESTORE.COM. If no path is specified, DOS* uses the current working directory.

iii. *dl* is the disk drive containing the backup disk.

iv. *file_spec* is the name of the file(s) to back up from the backup disk (*dl*). This file specification also tells RESTORE where the files should be restored to.

v. */P* requests RESTORE to prompt the user before restoring files that have been modified, or marked as read-only since the last backup.

vi. */S* requests RESTORE to restore files in all of the subdirectories present.

To illustrate the above, an example is given below:

```
C\DOS> RESTORE B: C/S
```

RESTORE will restore all of the files on the backup disk in drive B to the hard disk in drive C, including files contained in subdirectories.
APPENDIX E

PLOTTED EXPERIMENTAL RESULTS
FIGURE E2  Isothermal Data
FIGURE E3  Isothermal Data
FIGURE E5  Isothermal Data
FIGURE E7  Isothermal Data
FIGURE E10  Isothermal Data
FIGURE B20 Isothermal Data
FIGURE B23 Non-Isothermal Data

Re

25/25/0/0
25/35/0/0
25/45/0/0
25/55/0/0

qS

4000
1000
100
10
FIGURE E24  Non-Isothermal Data
FIGURE E25  Non-Isothermal Data
FIGURE E27  Non-Isothermal Data
FIGURE E30  Non-Isothermal Data
FIGURE E31 Non-Isothermal Data
FIGURE E34  Non-Isothermal Data
FIGURE E35  Non-Isothermal Data
FIGURE B41 Non-Isothermal Data
FIGURE E42  Non-Isothermal Data
APPENDIX F

FIDAP™ FINITE ELEMENT PROGRAM

The FIPREP input files for the 2- and 3-dimensional numerical simulation for the problem 25/55/0/0 (Experiment N59) are shown in Listings F.1 and F.5, respectively. The x, y, z coordinates in the physical model space as well as the corresponding integer coordinates can be found in Figure 6.7.1. A more detailed explanation of the individual commands is also given in Chapter 6.7. It should be noted, that none of the 150 page nodal data output files (approximately 2 MB) are listed in this Appendix due to space considerations. Nevertheless they are on magnetic media readily available in the research supervisons office (see Appendix D).
The generation of the FIDAP input file for the 2-dimensional laminar non-isothermal flow problem was accomplished using FiPREP. Listing F.1 summarizes the input file to FiPREP.

Listing F.1  FIECHO File for 2-dimensional Numerical Simulation of Experiment N59

*TITLE
G.B. 25/55/0/0, VEL=2.5 CM/S, N59
*FIMESH(2-D, IMAX=7, JMAX=3)
EXPI
  /1 2 3 4 5 6 7
  1 0 17 0 33 0 49
EXPJ
  /1 2 3
  1 0 15
POINT
  1 1 1 1 0 0
  2 3 1 1 127 0
  3 5 1 1 142 0
  4 7 1 1 229 0
  5 1 3 1 0 1.5
  6 3 3 1 127 1.5
  7 5 3 1 142 1.5
  8 7 3 1 229 1.5
LINE
  1 2 2 4
  2 3
  3 4
  5 6 2 4
  6 7
  7 8
  1 5
  2 6
  3 7
  4 8
SURFACE
  1 8
ELEMENTS (QUADRILATERAL, NODES=9, ALL)
ELEMENTS (BOUNDARY, NODES=3)
  4 8
BCNODE(VELO)
  4 0
  5 8 0
BCNODE(UX)
/BCNODE(UY)
  1 4
/1 5
/5 8
BCNODE(TEMPERATURE)
  1 5 25
  1 2 25
  2 3 55
  5 8 25
BCNODE(1SPECIES)
  1 5 2.5E-5
  1 2 2.5E-5
  2 3 0
  5 8 2.5E-5
END
*PROBLEM(NONLINEAR,TSTRONGLY,1STRAONGLY)
*SOLUTION(S.S.=25,ACCF=.2,VELCONV=0.0015,RESCONV=0.0007)
*EXECUTION(NEWJOB)
*OPTIONS(UPWINDING)
*UPWINDING
  1 1 0 0 2 0 0 0 2 0 0
*PRESSURE(MIXED=1.0E-99,DISCONTINUOUS)
*ICNODE(TEMPERATURE,CONSTANT=25)
*ICNODE(1SPECIES,CONSTANT=0)
*NODES(FMESH)
*ELEMENTS(QUADRILATERAL,NODES=9,FMESH,MEXP=1,M1EXP=2)
*ELEMENTS(OUTFLOW,NODES=3,FMESH)
*DENSITY(VARIABLE=1.0896,TEMPERATURE,1SPECIES)
*VISCOSITY(CURVE=9,TEMPERATURE)
  -1.0E10, 25, 30, 35, 40, 45, 50, 55, 1.0E10
  0.0127, 0.0127, 0.0114, 0.0103, 0.0094, 0.0089, 0.0086, 0.0086
*CONDUCTIVITY(CONSTANT=0.00155)
*SPECIFICHEAT(CONSTANT=0.915,TEMPERATURE)
*DIFFUSIVITY(CURVE=9,TEMPERATURE)
  -1.0E10, 25, 30, 35, 40, 45, 50, 55, 1.0E10
  5.4325E-6, 5.4325E-6, 6.2094E-6, 7.0061E-6, 7.7584E-6, 8.3833E-6,
  8.7956E-6, 8.9358E-6, 8.9358E-6
*VOLUMEXPANSION(SET=1,CONSTANT=4.671E-4,TEMPERATURE,REFTEMP=15)
*VOLUMEXPANSION(SET=2,CONSTANT=-6.91E-5,1SPECIES,REFTEMP=15)
*BODYFORCE(CONSTANT)
  0 -981.0
*RENUMBER
*END
The FIOUT file illustrates the FIPREP output file which summarizes the problem and problem structure to be analyzed (Listing F.2).

**Listing F.2  FIPREP File for 2-dimensional Numerical Simulation of Experiment N59**

```
              FFFFFFFF   I I I I I   P P P P P P P   R R R R R R   E E E E E E E   P P P P P P P
              FFFFFFFF   I I I I I   P P P P P P P   R R R R R R   E E E E E E E   P P P P P P P
               FF   I I   P P   P P   R R   R R   E E   P P   P P
               FF   I I   P P   P P   R R   R R   E E   P P   P P
               FF   I I   P P   P P   R R   R R   E E   P P   P P
               FF   I I   P P   P P   R R   R R   E E   P P   P P
               FF   I I   P P   P P   R R   R R   E E   P P   P P
               FF   I I I I I   P P   R R   R R   E E E E E E E   P P
               FF   I I I I I   P P   R R   R R   E E E E E E E   P P

FIDAP INPUT PREPARATION PROGRAM
===================================

RELEASE 1.5.05

```

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**FIPREP CARD IMAGE LISTING**

08-DEC-91 AT 23:33:43  PAGE NO: 1

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<td>5 0 2.5E-5</td>
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<tr>
<td>54</td>
<td>END</td>
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<tr>
<td>55</td>
<td>*PROBLEM (NONLINEAR, STRONGLY, STRONGLY)</td>
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<tr>
<td>56</td>
<td>*SOLUTION (S.S.=25, ACCF=.2, VELCONV=0.0015, RESCONV=0.0007)</td>
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<tr>
<td>57</td>
<td>*EXECUTION (NFIJOB)</td>
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<tr>
<td>58</td>
<td>*OPTIONS (UPWINDING)</td>
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<tr>
<td>59</td>
<td>*UPWINDING</td>
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<td>1 1 0 0 2 0 0 0 2 0 0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>61</td>
<td>*PRESSURE (MIXED=1.0E-99, DISCONTINUOUS)</td>
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<tr>
<td>62</td>
<td>*INCNODE (TEMPERATURE, CONSTANT=25)</td>
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<tr>
<td>63</td>
<td>*INCNODE (SPECIES, CONSTANT=0)</td>
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<tr>
<td>64</td>
<td>*NODES (FINISH)</td>
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<td>65</td>
<td>*ELEMENTS (QUADRILATERAL, NODES=9, FINESH, MEXP=1, NLIEVENT=2)</td>
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<tr>
<td>66</td>
<td>*ELEMENTS (OUTFLOW, NODES=3, FINESH)</td>
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<td>67</td>
<td>*DENSITY (VARIABLE=1.0896, TEMPERATURE, SPECIES)</td>
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<td>68</td>
<td>*VISCOSITY (CURVE=9, TEMPERATURE)</td>
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<td>71</td>
<td>*CONDUCTIVITY (CONSTANT=0.0015)</td>
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<td>72</td>
<td>*SPECIFICHEAT (CONSTANT=0.915, TEMPERATURE)</td>
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<td>73</td>
<td>*DIFFUSIVITY (CURVE=9, TEMPERATURE)</td>
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<td>77</td>
<td>*VOLUMEXPANSION (SET=1, CONSTANT=4.671E-4, TEMPERATURE, REFTEMP=15)</td>
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<td>*VOLUMEXPANSION (SET=2, CONSTANT=-6.91E-5, SPECIES, REFTEMP=15)</td>
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<td>*BODYFORCE (CONSTANT)</td>
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<td>80</td>
<td>0 -981.0</td>
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<td>81</td>
<td>*RENUMBER</td>
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<td>82</td>
<td>*END</td>
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******************************************************** END OF INPUT LISTING ***************
NUMBER OF NODES \hspace{2cm} (NUMNP) = 735
NUMBER OF ELEMENTS SCANNED \hspace{2cm} (NELEM) = 168
RENUMBERING OPTION \hspace{2cm} (LORRN) = 1
\hspace{2cm} = 2, MINIMISE BANDWIDTH
BANDWIDTH OF RENUMBERED MATRIX \hspace{2cm} (IBAND) = 34
PROFILE OF RENUMBERED MATRIX \hspace{2cm} (IPROF) = 13785
TOTAL CORE MEMORY FOR RENUMBERING \hspace{2cm} (INTOT) = 14936
WORKING SPACE MEMORY FOR RENUMBERING \hspace{2cm} (ISPACE) = 2283
TOTAL RENUMBERING TIME \hspace{2cm} (SEC.S) = 3.510
The philosophy involved in developing a nodal point mesh for a finite element analysis is to distribute the nodal points according to the anticipated field variables. For large gradients in the field variables, the nodal points should be dense; and for small gradients, the nodal points should be sparse. Therefore, it is necessary to make an estimate of how the field variables change in different regions of the problem domain when generating a mesh. FIMESH allows the user to generate boundary edges of the mesh with the desired spacing of nodal points by using line generators which preserve this spacing. The FIMESH control file for the 2-dimensional flow domain is shown in Listing F.3.

Listing F.3 FIMESH File for 2-dimensional Numerical Simulation of Experimental N59

```
* * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * *
* FFFFFFFF IIIIII MMM MMM EEEEEEEE SSSSSSS HH HH *
* FFFFFFFF IIIIII MMM MMM EEEEEEEE SSSSSSS HH HH *
* FF II MMMM MMMM EE SSS HH HH *
* FF II MMM MMM MMM MMM EE SSS HH HH *
* FF II MMM MMM MMM MMM EE SSS HH HH *
* FFFFFF II MMM MMM MMM MMM EEEEE SSSSSSS HHHHHHHHHH *
* FFFFFF II MMM MMM MMM MMM EEEEE SSSSSSS HHHHHHHHHH *
* FF II MMM MMM MMM MMM MMM MMM MMM SSS HH HH *
* FF II MMM MMM MMM MMM MMM MMM MMM SSS HH HH *
* FF II MMM MMM MMM MMM MMM MMM MMM SSS HH HH *
* FF II MMM MMM MMM MMM MMM MMM MMM SSS HH HH *
* FF II MMM MMM MMM MMM MMM MMM MMM SSS HH HH *
* FF II MMM MMM MMM MMM MMM MMM MMM SSS HH HH *
* FF II MMM MMM MMM MMM MMM MMM MMM SSS HH HH *
* FF II MMM MMM MMM MMM MMM MMM MMM SSS HH HH *
* FF II MMM MMM MMM MMM MMM MMM MMM SSS HH HH *
* FF II MMM MMM MMM MMM MMM MMM MMM SSS HH HH *
* FF II MMM MMM MMM MMM MMM MMM MMM SSS HH HH *
* FF II MMM MMM MMM MMM MMM MMM MMM SSS HH HH *
* FF II MMM MMM MMM MMM MMM MMM MMM SSS HH HH *
* FF II MMM MMM MMM MMM MMM MMM MMM SSS HH HH *
* FF II MMM MMM MMM MMM MMM MMM MMM SSS HH HH *
* FF II MMM MMM MMM MMM MMM MMM MMM SSS HH HH *
* FDDAP MESH GENERATION PROGRAM *
* ******************************************** *
* RELEASE ( 5.05 ) *
* ******************************************** *

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* G.B. 25/55/0/0, VEL=2.5 CM/S, N59 08-DEC-91 23:33:52 *
* PROBLEM DIMENSION *
* ******************************************** *
* 2-D GENERATION *
* IMAX = 7 JMAX = 3 KMAX = 1 MAX NO. OF POINTS = 300 *
```
### POINT DEFINITIONS

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<th>JO</th>
<th>KO</th>
<th>I</th>
<th>J</th>
<th>K</th>
<th>X</th>
<th>Y</th>
<th>Z</th>
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<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
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<td>0.000000E+00</td>
<td>0.000000E+00</td>
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<tr>
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<td>1</td>
<td>1</td>
<td>1</td>
<td>17</td>
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<td>0.000000E+00</td>
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<tr>
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<td>7</td>
<td>1</td>
<td>1</td>
<td>49</td>
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<td>1</td>
<td>229.000</td>
<td>0.000000E+00</td>
<td>0.000000E+00</td>
</tr>
<tr>
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<td>1</td>
<td>3</td>
<td>1</td>
<td>1</td>
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<td>1</td>
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<td>1.50000</td>
<td>0.000000E+00</td>
</tr>
<tr>
<td>7</td>
<td>5</td>
<td>3</td>
<td>1</td>
<td>17</td>
<td>15</td>
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<td>127.000</td>
<td>1.50000</td>
<td>0.000000E+00</td>
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<tr>
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<td>229.000</td>
<td>1.50000</td>
<td>0.000000E+00</td>
</tr>
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</table>

### LINE DEFINITIONS

| STRAIGHT LINE GENERATED FROM POINT | 1 | 1 | 1 | 1 | TO POINT | 2 | 17 | 1 | 1 |
| STRAIGHT LINE GENERATED FROM POINT | 2 | 17 | 1 | 1 | TO POINT | 3 | 33 | 1 | 1 |
| STRAIGHT LINE GENERATED FROM POINT | 3 | 33 | 1 | 1 | TO POINT | 4 | 49 | 1 | 1 |
| STRAIGHT LINE GENERATED FROM POINT | 4 | 49 | 1 | 1 | TO POINT | 5 | 1 | 15 | 1 |
| STRAIGHT LINE GENERATED FROM POINT | 5 | 1 | 15 | 1 | TO POINT | 6 | 17 | 15 | 1 |
| STRAIGHT LINE GENERATED FROM POINT | 6 | 17 | 15 | 1 | TO POINT | 7 | 33 | 15 | 1 |
| STRAIGHT LINE GENERATED FROM POINT | 7 | 33 | 15 | 1 | TO POINT | 8 | 49 | 15 | 1 |
| STRAIGHT LINE GENERATED FROM POINT | 8 | 49 | 15 | 1 | TO POINT | 9 | 1 | 1 | 1 |
| SUB-SURFACE GENERATED FROM POINT | 1 | 1 | 1 | 1 | TO POINT | 9 | 1 | 1 | 1 |
| SUB-SURFACE GENERATED FROM POINT | 4 | 49 | 1 | 1 | TO POINT | 8 | 49 | 15 | 1 |

### SURFACE DEFINITIONS

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<th>ELEMENT GROUP DEFINITION</th>
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### BOUNDARY CONDITIONS - NODE POINT

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<th>POINT1</th>
<th>POINT2</th>
<th>VALUE1</th>
<th>VALUE2</th>
<th>NCUR</th>
<th>FAC</th>
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<td>0.000000E+00</td>
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<td>CONS</td>
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<td>CONS</td>
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<td>3</td>
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<td>0.000000E+00</td>
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<td>0.000000E+00</td>
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TOTAL MESH DATA
=================================
TOTAL NUMBER OF NODES .............. 735
TOTAL NUMBER OF ELEMENTS .......... 175
NUMBER OF ELEMENT GROUPS .......... 2


TIMING INFORMATION
=================================

G.B. 25/55/0/0, VEL=2.5 CM/S, N59

POINTS AND EDGE POINTS GENERATION ........ 1.87
SURFACE POINTS GENERATION ............. 0.16
CURVE DRIVEN POINTS GENERATION ........ 0.00
3-D MESH POINTS GENERATION ............ 0.00
AREA CALCULATIONS .................... 0.00
VOLUME CALCULATIONS ................... 0.00
RELAXATION CALCULATIONS ............... 0.00
BOUNDARY CONDITIONS GENERATION ....... 1.70
ELEMENT CONNECTIVITY FORMATION ........ 0.23
MERGE CALCULATIONS .................. 0.00
OUTPUT FILE GENERATION ............... 0.66
PLOT .................................. 0.00

TOTAL GENERATION (SEC) = 5.28

****** END OF FIMESH RUN ******

***** FIDAP INPUT FILE SUCCESSFULLY CREATED
After each numerical simulation FIDAP™ generates a job status file, FDSTAT. This file contains information about the computer resources needed to solve the problem, as well as convergence information. Sample FDSTAT outputs for the 2-and 3-dimensional analysis are given in Listings F.4 and F.8, respectively.

Listing F.4  FDSTAT File for the 2-dimensional Numerical Simulation of Experiment N59

==========================================================================
FIDAP ( 5.05 ) 08-DEC-91 AT 23:34:31
THIS SOFTWARE IS A LICENSED PRODUCT OF FLUID DYNAMICS INTERNATIONAL, INC. AND MAY ONLY BE USED ACCORDING TO THE TERMS OF THAT LICENSE ON THE SYSTEM IDENTIFIED IN THE LICENSE AGREEMENT. COPYRIGHT (C) 1990 BY FLUID DYNAMICS INTERNATIONAL, INC. ALL RIGHTS RESERVED.
==========================================================================
G.B. 25/55/0/0, VEL=2.5 CM/S, N59

PROBLEM DEFINITION & CONTROL INFORMATION INPUT, NO. OF ERRORS = 0
TIME FUNCTION INFORMATION INPUT, NO. OF ERRORS = 0
NODAL DATA INPUT, NO. OF ERRORS = 0
CONSTRAINED D.O.F.S INPUT, NO. OF ERRORS = 0
LOAD FORCES INPUT, NO. OF ERRORS = 0
VOLUMETRIC FORCES INPUT, NO. OF ERRORS = 0
INITIAL CONDITIONS INPUT, NO. OF ERRORS = 0
PROBLEM ELEMENT INFORMATION INPUT COMPLETED
MATRIX PROFILE STRUCTURE COMPUTED

TOTAL SYSTEM DATA

-----------------------------------------------
NON SYMMETRIC MATRIX BLOCK STRUCTURE
NUMBER OF EQUATIONS .............. (NQN) = 3045
NUMBER OF MATRIX ELEMENTS ......... (NENN) = 441265
MEAN HALF BANDWIDTH ............. (MIBBN) = 72
MAXIMUM HALF BANDWIDTH ........... (MAXBN) = 156
STORAGE FOR ONE BLOCK (INTEGER WORDS) .... (MEMRN) = 882536
NUMBER OF BLOCKS .............. (NBLKNS) = 1
MAX. TOTAL STORAGE AVAILABLE (INTEGER WORDS) (MTOT) = 3000000
ADD. MEMORY FOR GLOBAL MATRIX TO BE IN CORE (MCMCIN) = 0
(0 = IN CORE)

FILE STRUCTURE DATA

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   RECORDS LENGTH RECORDS LENGTH
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33 1 776 1 776 3
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<th>VEL</th>
<th>DINORM</th>
<th>DNORM</th>
<th>REL.ERR.</th>
<th>F</th>
<th>RENORM</th>
<th>RNORM</th>
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--- ITERATION ANALYSIS FOR ITERATION NUMBER ---

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<th>RENORM</th>
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#### Computation Complete for Load Step 1
TIMING INFORMATION
G.B. 25/55/0/0, VEL=2.5 CM/S, N59

INPUT PHASE . . . . . . . . . . . . . . . . . . . . . . . . . . . . . 7.97

BREAKDOWN FOR SINGLE ITERATION

GENERATION OF ELEMENT MATRICES
      AND FORCE VECTORS -  6.70
      ASSEMBLY OF FULL SYSTEM -  0.77
      LU DECOMPOSITION/SOLUTION -  32.84
      MATRIX FREE ITERATION -  0.00

STOKES SOLUTION . . . . . . . . . . . . . . . . . . . . . . . . . . . 0.00

STEADY SOLUTION/TIME INTEGRATION PHASE . . . . . . . . . . . . . . 745.55

      UPDATING FOR NONLINEARITY -  121.75
      ASSEMBLY -  15.15
      LU DECOMPOSITION -  590.05
      MATRIX FREE ITERATION -  0.00
      LINE SEARCHES -  0.00
      SEARCH DIRECTIONS -  0.00
      PRESSURE RECOVERY -  0.38

TOTAL SOLUTION TIME (SEC) =  753.52

******* FIDAP RUN COMPLETED *******
Listing F.5 FIECHO File for the 3-dimensional Numerical Simulation of Experiment N59

*TITLE
G.B. 25/55/0/0, VEL=2.5 CM/S, N59
*FIMESH(3-D, IMAX=9, JMAX=5, KMAX=5)

EXPI
1 0 9 0 0 0 29 0 37
EXPJ
1 0 0 0 11
EXPK
1 0 0 0 11

POINT
1 1 1 5 0. 0. 0.
10 3 1 5 127. 0. 0.
11 7 1 5 142. 0. 0.
2 9 1 5 229. 0. 0.
/
3 9 5 5 229. 0. 1.5
12 7 5 5 142. 0. 1.5
13 3 5 5 127. 0. 1.5
4 1 5 5 0. 0. 1.5
/
5 1 1 1 0. 2. 0.
14 3 1 1 127. 2. 0.
15 7 1 1 142. 2. 0.
6 9 1 1 229. 2. 0.
/
7 9 5 1 229. 2. 1.5
17 7 5 1 142. 2. 1.5
18 3 5 1 127. 2. 1.5
8 1 5 1 0. 2. 1.5

LINE
1 10
10 11
11 2
2 3
3 12
12 13
13 4
1 4
5 14
14 15
15 6
6 7
7 17
17 18
18 8
8 5
1 5
2 6
3 7
4 8
10 13
11 12
14 18
15 17
13 18
12 17
10 14
11 15
SURFACE
1 13
10 12
11 3
5 18
14 17
15 7
1 14
10 15
11 6
4 18
13 17
12 7
1 8
2 7
3-D
1 7
BCNODE(VELO)
/NEAR SIDE WALL
1 3 0.
/BOTTOM
1 6 0.
/FAR SIDE WALL
5 7 0.
/TOP
4 7 0.
BCNODE(UX)
/INFLOW PLANE
1 8 2.5
BCNODE(TEMP)
/INFLOW PLANE
1 8 25
/BOTTOM PLANE
10 15 55
BCNODE(1SPECIES)

/INFLOW PLANE
1 8 2.5E-5
/BOTTOM PLANE
10 15 0
ELEMENTS(4BRICK, NODES=27)
1 7
NUMBER
2 3 1
END
*PROBLEM(3-D, NONLINEAR, STEADY, TSTRONGLY, 1STRONGLY, FIXED, MOMENTUM)
*EXECUTION (NEWJOB)
*PRESSURE (MIXED)
*SOLUTION (S.S. = 200)
*POSTPROCESS
*NODES (FIMESH)
*ELEMENTS (BRICK, NODES=27, FIMESH, LINEAR, GLOBAL, MDENS=1, MCOND=1,
MSPHRT=1, MVISC=1, MEXP=1, M1EXP=2, M1DIFF=1)
*ICNODE (TEMP, CONSTANT=25)
/VISCOSITY - LINEAR BELOW 15 AND ABOVE 55
*VISCOSITY (SET=1, CURVE=17, TEMPERATURE)
 -1E10 5 10 15 20 25 30 35 40 45 50 55 60 65 70 75 1E10
 0.041 0.0211 0.0186 0.164 0.0144 0.0127 0.0114 0.0103 0.0094, 
 0.0089, 0.0086 0.0086 0.0089 0.0095 0.0104 0.0115 0.0130
/DENSITY AT REFERENCE TEMP OF 15 DEG C
*DENSITY (SET=1, VARIABLE=1.0896, TEMPERATURE, 1SPECIES)
/SPECIFIC HEAT CONSTANT
*SPECIFICHEAT (SET=1, CONSTANT=0.915)
/THERMAL CONDUCTIVITY CONSTANT
*CONDUCTIVITY (SET=1, CONSTANT=0.00155)
/VOLUME EXPANSION DUE TO TEMPERATURE
*VOLUMEXPANSION (SET=1, CONSTANT=4.671E-4, TEMPERATURE, REFTEMP=15)
/VOLUME EXPANSION DUE TO CONCENTRATION CHANGE IN FERRI CYANIDE
*VOLUMEXPANSION (SET=2, CONSTANT=-6.91E-5, 1SPECIES, REFTEMP=15)
/DIFFUSIVITY OF FERRI CYANIDE - LINEAR BELOW 15 AND ABOVE 55 DEG C
*DIFFUSIVITY (SET=1, CURVE=14, TEMPERATURE)
 -1E10 5 10 15 20 25 30 35 40 45 50 55 80 1E10
 2.E-6 3.0389E-6 3.5177E-6 4.0750E-6 4.7147E-6 5.4325E-6 6.2094E-6,
 7.0061E-6 7.7584E-6 8.3833E-6 8.7956E-6 8.9358E-6 8.96E-6 9.9E-6
*BODYFORCE (CONSTANT)
0 -981 0
*RENUMBER (PROFILE)
*END

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### FIPREP Card Image Listing

**01-JAN-92 AT 11:58:44**

**Page No.: 3**

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**1**

- **MINIMISE PROFILE**
- **MINIMISE BANDWIDTH**
- **BANDWIDTH OF RENUMBERED MATRIX**
- **PROFILE OF RENUMBERED MATRIX**
- **TOTAL CORE MEMORY FOR RENUMBERING**
- **WORKING SPACE MEMORY FOR RENUMBERING**
- **TOTAL RENUMBERING TIME**
Listing F.7 FIMESH File for the 3-dimensional Numerical Simulation of Experiment N59

```
FFFFFFFFF I I I I I I M M M M M M M EEEEEEEEE ESSSSSSSS SSSSSSS HH HH
FFFFFFFFF I I I I I I M M M M M M M EEEEEEEEE ESSSSSSSS SSSSSSS HH HH
FF I I M M M M M M M M M M M E E SSS HH HH
FF I I M M M M M M M M M M M E E SSS HH HH
FF I I M M M M M M M M M M M E E SSS HH HH
FF I I M M M M M M M M M M M E E SSS HH HH
FF I I I I I I M M M M M M M EEEEEEEEE SSSSSSSSS SSSSSSS HH HH
FF I I I I I I M M M M M M M EEEEEEEEE SSSSSSSSS SSSSSSS HH HH
FIDAP MESH GENERATION PROGRAM

RELEASE ( 5.05 )
```

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G.B. 25/55/0/0. VEL=2.5 CM/S. N59

01-JAN-92 11:50:53

**Problem Dimension**

```
3-D GENERATION

IMAX = 9  JMAX = 5  KMAX = 5  MAX NO. OF POINTS = 300
```

**Point Definitions**

```
POINT  IO  JO  KO  I  J  K  X   Y   Z
     1  1  1  1  1  1  0.000000E+00  0.000000E+00  0.000000E+00
     10  1  1  5  9  1  11  127.000000E+00  0.000000E+00  0.000000E+00
     11  1  1  5  9  1  11  142.000000E+00  0.000000E+00  0.000000E+00
     2  1  1  5  37 1  11  229.000000E+00  0.000000E+00  0.000000E+00
     3  9  5  5  37 1  11  229.000000E+00  0.000000E+00  1.50000
     12  7  5  5  29 1  11  142.000000E+00  0.000000E+00  1.50000
     13  3  5  5  9  1  11  127.000000E+00  0.000000E+00  1.50000
     4  1  5  5  1  1  11  0.000000E+00  0.000000E+00  1.50000
     5  1  1  1  9  1  1  0.000000E+00  2.000000E+00  0.000000E+00
     14  1  1  1  9  1  1  127.000000E+00  2.000000E+00  0.000000E+00
     15  7  1  1  29 1  1  142.000000E+00  2.000000E+00  0.000000E+00
     6  9  1  1  37 1  1  229.000000E+00  2.000000E+00  0.000000E+00
     7  9  5  1  37 1  11  229.000000E+00  2.000000E+00  1.50000
     17  7  5  1  29 1  11  142.000000E+00  2.000000E+00  1.50000
     16  3  5  1  9  1  11  127.000000E+00  2.000000E+00  1.50000
     8  1  5  1  1  1  11  0.000000E+00  2.000000E+00  1.50000
```
### LINE DEFINITIONS

```
STRAIGHT LINE GENERATED FROM POINT 1 ( 1, 1, 11 ) TO POINT 10 ( 9, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 10 ( 9, 11, 11 ) TO POINT 11 ( 29, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 11 ( 29, 11, 11 ) TO POINT 2 ( 37, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 2 ( 37, 11, 11 ) TO POINT 3 ( 37, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 3 ( 37, 11, 11 ) TO POINT 12 ( 29, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 12 ( 29, 11, 11 ) TO POINT 13 ( 9, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 13 ( 9, 11, 11 ) TO POINT 4 ( 1, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 4 ( 1, 11, 11 ) TO POINT 14 ( 9, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 14 ( 9, 11, 11 ) TO POINT 15 ( 29, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 15 ( 29, 11, 11 ) TO POINT 6 ( 37, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 6 ( 37, 11, 11 ) TO POINT 7 ( 37, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 7 ( 37, 11, 11 ) TO POINT 16 ( 9, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 16 ( 9, 11, 11 ) TO POINT 8 ( 1, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 8 ( 1, 11, 11 ) TO POINT 5 ( 1, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 5 ( 1, 11, 11 ) TO POINT 9 ( 37, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 9 ( 37, 11, 11 ) TO POINT 17 ( 29, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 17 ( 29, 11, 11 ) TO POINT 10 ( 9, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 10 ( 9, 11, 11 ) TO POINT 11 ( 29, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 11 ( 29, 11, 11 ) TO POINT 12 ( 29, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 12 ( 29, 11, 11 ) TO POINT 13 ( 9, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 13 ( 9, 11, 11 ) TO POINT 14 ( 9, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 14 ( 9, 11, 11 ) TO POINT 15 ( 29, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 15 ( 29, 11, 11 ) TO POINT 16 ( 9, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 16 ( 9, 11, 11 ) TO POINT 17 ( 29, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 17 ( 29, 11, 11 ) TO POINT 18 ( 9, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 18 ( 9, 11, 11 ) TO POINT 19 ( 37, 11, 11 )
STRAIGHT LINE GENERATED FROM POINT 19 ( 37, 11, 11 ) TO POINT 20 ( 37, 11, 11 )
```

### SURFACE DEFINITIONS

```
SURFACE GENERATED FROM POINT 1 ( 1, 1, 11 ) TO POINT 13 ( 9, 11, 11 )
SURFACE GENERATED FROM POINT 10 ( 9, 11, 11 ) TO POINT 12 ( 29, 11, 11 )
SURFACE GENERATED FROM POINT 11 ( 29, 11, 11 ) TO POINT 3 ( 37, 11, 11 )
SURFACE GENERATED FROM POINT 5 ( 1, 1, 11 ) TO POINT 18 ( 9, 11, 11 )
SURFACE GENERATED FROM POINT 14 ( 9, 11, 11 ) TO POINT 17 ( 29, 11, 11 )
SURFACE GENERATED FROM POINT 15 ( 29, 11, 11 ) TO POINT 7 ( 37, 11, 11 )
SURFACE GENERATED FROM POINT 1 ( 1, 1, 11 ) TO POINT 9 ( 37, 11, 11 )
SURFACE GENERATED FROM POINT 10 ( 9, 11, 11 ) TO POINT 11 ( 29, 11, 11 )
SURFACE GENERATED FROM POINT 11 ( 29, 11, 11 ) TO POINT 12 ( 29, 11, 11 )
SURFACE GENERATED FROM POINT 12 ( 29, 11, 11 ) TO POINT 13 ( 9, 11, 11 )
SURFACE GENERATED FROM POINT 13 ( 9, 11, 11 ) TO POINT 14 ( 9, 11, 11 )
SURFACE GENERATED FROM POINT 14 ( 9, 11, 11 ) TO POINT 15 ( 29, 11, 11 )
SURFACE GENERATED FROM POINT 15 ( 29, 11, 11 ) TO POINT 16 ( 9, 11, 11 )
SURFACE GENERATED FROM POINT 16 ( 9, 11, 11 ) TO POINT 17 ( 29, 11, 11 )
SURFACE GENERATED FROM POINT 17 ( 29, 11, 11 ) TO POINT 18 ( 9, 11, 11 )
SURFACE GENERATED FROM POINT 18 ( 9, 11, 11 ) TO POINT 19 ( 37, 11, 11 )
SURFACE GENERATED FROM POINT 19 ( 37, 11, 11 ) TO POINT 20 ( 37, 11, 11 )
```

### 3-D REGION DEFINITIONS

```
3-D REGION GENERATED FROM POINT 1 ( 1, 1, 1 ) TO POINT 7 ( 37, 11, 11 )
```

### BOUNDARY CONDITIONS - NODE POINT

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<td>0.00000E+00</td>
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</table>
```
ELEMENT GROUP DEFINITION

ELEMENTS GENERATED FROM POINT 1 (1, 1, 1) TO POINT 7 (37, 11, 11)

NODAL NUMBERING

NUMBERING SEQUENCE J K I

TOTAL MESH DATA

TOTAL NUMBER OF NODES 4477
TOTAL NUMBER OF ELEMENTS 456
NUMBER OF ELEMENT GROUPS 1
GROUP 1: NO. OF ELEMENTS 450, NO. OF NODES 27

TIMING INFORMATION

G.B. 25/55/0/0, VEL=2.5 CM/S, N59

POINTS AND EDGE POINTS GENERATION 4.45
SURFACE POINTS GENERATION 1.37
CURVE DRIVEN POINTS GENERATION 0.00
3-D MESH POINTS GENERATION 101.56
AREA CALCULATIONS 0.00
VOLUME CALCULATIONS 0.00
RELAXATION CALCULATIONS 0.00
BOUNDARY CONDITIONS GENERATION 10.27
ELEMENT CONNECTIVITY FORMATION 1.32
MERGE CALCULATIONS 0.00
OUTPUT FILE GENERATION 5.11
PLOT 0.00

TOTAL GENERATION (SEC) = 125.78

******* END OF FIMESH RUN *******

***** FIDAP INPUT FILE SUCCESSFULLY CREATED
Listing F.8  FDSTAT File for the 3-dimensional Numerical Simulation of Experiment N59

=============== FIDAP (5.05) 01-JAN-92 AT 12:09:58 ===============

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==================================

G.B. 25/55/0/0, VEL=2.5 CM/S, N59

PROBLEM DEFINITION AND CONTROL INFORMATION INPUT, NO. OF ERRORS = 0
TIME FUNCTION INFORMATION INPUT, NO. OF ERRORS = 0
NODAL DATA INPUT, NO. OF ERRORS = 0
CONSTRAINED D.O.F.S INPUT, NO. OF ERRORS = 0
LOAD FORCES INPUT, NO. OF ERRORS = 0
VOLUMETRIC FORCES INPUT, NO. OF ERRORS = 0
INITIAL CONDITIONS INPUT, NO. OF ERRORS = 0

PROBLEM ELEMENT INFORMATION INPUT COMPLETED
MATRIX PROFILE STRUCTURE COMPUTED

TOTAL SYSTEM DATA

NON SYMMETRIC MATRIX BLOCK STRUCTURE

NUMBER OF EQUATIONS ............... (NON) = 23085
NUMBER OF MATRIX ELEMENTS ........... (NEMN) = 35756645
MEAN HALF BANDWIDTH ............... (MNBW) = 774
MAXIMUM HALF BANDWIDTH ........... (MAXBW) = 1792
STORAGE FOR ONE BLOCK (INTEGER WORDS) .... (MENORN) = 1416854
NUMBER OF BLOCKS ............... (NBLKS) = 51
MAX. TOTAL STORAGE AVAILABLE (INTEGER WORDS) (MNTOT) = 3000000
ADD. MEMORY FOR GLOBAL MATRIX TO BE IN CORE (MNCIN) = 66985974
(0 = IN CORE)

FILE STRUCTURE DATA

UNIT NO. LOGICAL LOGICAL REC. NO. PHYSICAL PHYSICAL REC. KBYTES
RECORDS LENGTH RECORDS LENGTH
32 30 62678 10 62678 2448
33 1 2368 1 2368 9
38 51 1416854 1122 65536 267232

==== LOAD STEP 1 ======== AT LOAD 0.1000E+01 INCREMENT 0.1000E+01 ======

***** EXECUTION ERROR (6701)*****

FATAL ERROR IN WRITING ON UNIT ( 38 )
LOGICAL RECORD ( 35 ), PHYSICAL RECORD ( 770 )
OF ( 22 ) PHYSICAL RECORDS BEING WRITTEN
SYSTEM DEPENDENT ERROR STATUS NUMBER = 2
This run was terminated by FIDAP™, due to the fact that the file FORT.38 (UNIT 38) required more continuous free hard disk space (287.2 MB) than there was available. This listing was included to illustrate that for complex 3-dimensional numerical simulations workstation based computers are required to perform these computational tasks.
APPENDIX G
DATA COLLECTION PROGRAM

The Turbo Pascal* program development environment has changed over the past couple of years in a way that the third party subroutine suppliers could not keep up with this progress. At the time the data collection program was developed Turbo Pascal*’s 5th revision was already released, but unfortunately no software drivers for the DT-752, DT-756 and DT-2801 computer boards were available. Due to this problem the data collection software code had to be written in the less structured Turbo Pascal* Version 3.0 format. The program listing is shown in Listing G.1.
Listing G.1  Source Code of the Data Collection Program

PROGRAM COPPERBLOCK (INPUT, OUTPUT);

{Si PCLDEFS.TP}
{Si PCLERRS.PAS}
{Si AVGVOLTS.PAS}

{Program structure originally developed by M. Ajersch; Modified by G. Bendich.}
{Last Update: June 27, 1990, Version 1.05}

LABEL 555, 666;

VAR

STATUS, TCTYPE:               INTEGER;
CHANNEL:                       CHAR;
Z, DUMMY, INDEX, X, Y, POSITION: INTEGER;
PRINTCOUNT:                    REAL;
CHVOLTS, TCVOLTS, DEGREES:    REAL;
TSURF1, TSURF2, SURFACETEMP:   REAL;
ERRORTEST, SETPOINT:          REAL;
FULL, LOOP, PRACTICE:          BOOLEAN;
DATE, FILENAME, EXPERIMENT:   STRING[25];
SURE.SIZE:                     CHAR;
ROTMETER:                      REAL;
THERMO:                        TEXT;

T: ARRAY[1..10] OF REAL;

******************************************************************************

{Procedure CHECKCOMMAND enables keyboard commands to be entered while the main
program is still running.}

PROCEDURE CHECKCOMMAND;

VAR CMD: CHAR;
BEGIN

READ(Kbd.CMD);
CMD := UPCASE(CMD);
CASE CMD OF

'R':  DUMMY := 501; {Reset the program}
'H':  DUMMY := 502; {Quit the program}
'F':  DUMMY := 504; {Save the temperatures in a datafile}

ELSE WRITE(CHR(7)); {BEEP at bogus command}
END (CASE)
END;  {CHECKCOMMAND}
PROCEDURE PRINTTEMPERATURE;

VAR POSITION, COUNT, FUN: INTEGER;

BEGIN

GOTOXY(28,24); WRITE(SETPOINT:4:0);
GOTOXY(5,17); WRITE(T[1]:4:2);
GOTOXY(76,17); WRITE(T[4]:4:2);
GOTOXY(23,18); WRITE(T[2]:4:2);
GOTOXY(57,18); WRITE(T[3]:4:2);
GOTOXY(28,25); WRITE(T[5]:4:2);

PRINTCOUNT := 0;

END;  (PRINTTEMPERATURE)

BEGIN  (COPPERBLOCK)

STATUS := INITIALIZE;
STATUS := SELECTBOARD(1);
TCTYPE := ORD('T');
FULL := FALSE;
PRACTICE := FALSE;
CLRSCR;
WRITE('TEST RUN ONLY [Y/N]? : ');
READ(SURE);
IF SURE = 'Y' THEN PRACTICE := TRUE
ELSE
BEGIN
  WRITELN;
  WRITELN;
  WRITE('ENTER DATE : ');
  READ(DATE);
  WRITELN;
END:
(Input the surface temperature setpoint)

666: GOTOXY(1,5);
DUMMY := 0;
LOOP := FALSE;
FULL := FALSE;
PRINTCOUNT := 25;
ANALOGDATAVALUE := 0;
STATUS := DACVALUE(0, ANALOGDATAVALUE);
STATUS := DACVALUE(1, ANALOGDATAVALUE);
WRITELN;

REPEAT

IF PRACTICE = TRUE THEN GOTO 555;
CLRSCR;
WRITELN('DATE: ','DATE');
WRITELN;
WRITE('ENTER FILENAME WHERE DATA WILL BE STORED: '); 
READ(FILENAME);
EXPERIMENT := FILENAME;
WRITELN;
WRITELN;
555: WRITE('INPUT CATHODE SURFACE TEMPERATURE SETPOINT: '); 
READ(SETPOINT);
WRITELN;
WRITELN;
WRITE('SURE [Y/N]? : '); 
READ(SURE);
WRITELN;
IF SETPOINT > 60 THEN BEGIN
  WRITELN;
  WRITELN('SETPOINT TOO HIGH! PERSPEX WILL MELT!');
  WRITELN('PLEASE TRY AGAIN. [Hit any key to continue]');
  REPEAT UNTIL KEYPRESSED;
  LOOP := FALSE;
END
ELSE
  IF (SURE = 'Y') OR (SURE = 'y') THEN LOOP := TRUE;
UNTIL LOOP = TRUE;

**************************************************************************

(Draw the copper electrode and thermocouple positions)

CLRSCR;
FOR INDEX := 1 TO 25 DO WRITE(#0);
WRITE('CATHODE TEMPERATURE PROFILE');
GOTOXY(2,10); WRITE('UP');
GOTOXY(2,11); WRITE('STREAM');
GOTOXY(73,10); WRITE('DOWN');
GOTOXY(73,11); WRITE('STREAM');
(Draw top, sides, and bottom of electrode)

GOTOXY(10,3); WRITE(*218);
FOR INDEX := 1 TO 59 DO WRITE(*196);
WRITE(*191);
FOR Y := 4 TO 19 DO
BEGIN
GOTOXY(10,Y); WRITE(*179);
GOTOXY(70,Y); WRITELN(*179);
END;

GOTOXY(10,Y); WRITE(*192);
FOR INDEX := 1 TO 59 DO WRITE(*196);
WRITE(*217);

(Draw the thermocouple positions)

GOTOXY(17,18); WRITELN('T2: ');
GOTOXY(52,18); WRITELN('T3: ');
GOTOXY(1,17); WRITELN('T1: ');
GOTOXY(71,17); WRITELN('T4: ');
GOTOXY(1,24); WRITE('SURFACE TEMP. SETPOINT : ');
GOTOXY(1,25); WRITE('RESERVIOR TEMPERATURE : ');
GOTOXY(25,21); WRITE(' ELECTROLYTE FLOW ======= >> ');
GOTOXY(35,11); WRITE('CATHODE');
GOTOXY(23,15); WRITE('ALL TEMPERATURES IN DEG. CELSIUS');
GOTOXY(68,24); WRITE('G.B. JUNE 90');
GOTOXY(68,25); WRITE('VERSION 1.05');

(Draw the cartridge heater)

GOTOXY(5,4); WRITE(*218);
GOTOXY(4,5); WRITE(*205); WRITE(*181);
GOTOXY(4,6); WRITE(*205); WRITE(*181);
GOTOXY(5,7); WRITE(*192);

GOTOXY(6,4); FOR INDEX := 1 TO 62 DO WRITE(*196);
WRITE(*191);
GOTOXY(6,7); FOR INDEX := 1 TO 62 DO WRITE(*196);
WRITE(*217);

GOTOXY(10,4); WRITE(*193);
GOTOXY(10,7); WRITE(*194);
GOTOXY(10,5); WRITE(*0);
GOTOXY(10,6); WRITE(*0);
GOTOXY(68,5); WRITE(*179);
GOTOXY(68,6); WRITE(*179);
(Draw the channel walls)

GOTOXY(1,19);  FOR INDEX := 1 TO 9 DO WRITE(*205);
WRITE(*207);
GOTOXY(70,19);  WRITE(*207);
FOR INDEX := 1 TO 9 DO WRITE(#205);
GOTOXY(3,19);  WRITE(#223); WRITE(#223);
GOTOXY(73,19);  WRITE(#223); WRITE(#223);
GOTOXY(1,23);  FOR INDEX := 1 TO 79 DO WRITE(#205);

(Draw the menu box)

GOTOXY(72,1);  WRITE(*201);
FOR INDEX := 1 TO 7 DO WRITE(*205);
GOTOXY(80,1);  WRITE(*187);
FOR INDEX := 1 TO 5 DO
BEGIN
  GOTOXY(72,INDEX+1);
  WRITE(*186,','*186);
END;
GOTOXY(72,7);  WRITE(*200);
FOR INDEX := 1 TO 7 DO WRITE(*205);
GOTOXY(80,7);  WRITE(*188);
GOTOXY(73,2);  WRITE('R)eset');
GOTOXY(73,4);  WRITE('H)alt');
GOTOXY(73,6);  WRITE('F)ile');

******************************************************************************
(Main LOOP. Get the temperature readings and print them on screen)
******************************************************************************

WHILE DUMMY < 501 DO BEGIN

IF KEYPRESSED THEN CHECKCOMMAND;

(Get the 5 temperature readings)

FOR INDEX :=1 TO 5 DO
BEGIN
  CHANNEL := INDEX;
  STATUS := MEASUREVOLTS(CHANNEL, CHVOLTS);
  TCVOLTS := CHVOLTS/400;
  STATUS := VOLTSTODEGREES(TCTYPE, TCVOLTS, DEGREES);
  T[INDEX] := DEGREES;
END;

(Print the 5 temperatures to the screen)

IF ROUND(PRINTCOUNT/25) = 1 THEN PRINTTEMPERATURE;
PRINTCOUNT := PRINTCOUNT + 1;
CASE DUMMY OF

501:  GOTO 666;  (Reset program)

502:  BEGIN   (Exit the program)
       CLRSCR;
       GOTOXY(1,1);
       WRITELN('PROGRAM TERMINATED AT YOUR REQUEST');
       END;

503:  BEGIN   (Exit program, temperature too high)
       CLRSCR;
       GOTOXY(1,1);
       FOR INDEX := 1 TO 10 DO WRITELN(' ');
       GOTOXY(1,1);
       WRITELN('***************************************************');
       WRITELN('PROGRAM TERMINATED. CATHODE TEMPERATURE TOO HIGH FOR PERSPEX.');
       WRITELN(' ');
       WRITELN('THANKS FOR USING THE DATA COLLECTION SYSTEM ');
       WRITELN(' ');
       WRITELN('***************************************************');
       WRITELN; WRITELN; WRITELN;
       END;

504:  BEGIN   (Dump all data into file)
       ASSIGN( THERMO, FILENAME);
       REWRITE( THERMO );
       WRITELN( THERMO, 'FILE: ', EXPERIMENT );
       WRITELN( THERMO, 'DATE: ', DATE );
       WRITELN( THERMO );
       WRITELN( THERMO, 'TEMPERATURE PROFILE' );
       WRITELN( THERMO );
       FOR INDEX := 1 TO 6 DO WRITELN( THERMO, 'T[', INDEX, ']' = ',T[INDEX]:3:1 );
       WRITELN( THERMO );
       WRITELN( THERMO, 'BULK TEMP. UPSTREAM : ', T[7]:3:1 );
       WRITELN( THERMO, 'DOWNSTREAM TEMP. : ', T[8]:3:1 );
       WRITELN( THERMO, 'ANODE TEMP. : ', T[9]:3:1 );
       WRITELN( THERMO );
       WRITELN( THERMO, 'SURFACE TEMP.SETPT.: ', SETPOINT:3:1 );
       WRITELN( THERMO, 'AVG. SURFACE TEMP. : ', SURFACETEMP:3:1 );
       LOOP := FALSE;
       SIZE := 'M';
       WRITELN;
       WRITELN;
       ROTAMETER := 100;
       WRITELN; WRITELN;
       WRITELN( THERMO );
       WRITELN( THERMO, 'ROTAMETER SIZE' );
       WRITELN( THERMO, 'ROTAMETER READING : ', ROTAMETER:5:1 );
WRITELN(THRON);
CLOSE(THRON);

CLSCLR;
WRITE('DATA DUMPED IN FILE ');
WRITELN(FILENAME);
WRITELN;
WRITE('CONTINUE [Y/N]? : '); READ(SURE);
IF SURE = 'Y' THEN GOTO 600
ELSE BEGIN
   WRITELN;
   WRITELN;
   WRITELN('THANKS FOR USING THE DATA COLLECTION SYSTEM');
   EXIT;
END;

END;  {CASE 504)

END;  {CASE}

STATUS := TERMINATE;
END.
APPENDIX H

LITERATURE REVIEW SPREAD SHEET

Publications found in various journals which were of some interest to this project were analyzed and the information (e.g. flow field, temperature range, ...) summarized in form of a spread sheet (see Lotus File: GBHMT1.WK3). The list of the individual papers, which was taken directly from the Lotus spread sheet, is presented in Listing H.1.
LISTING H.1 Alphabeticallst Listing from the Data Collection Spread Sheet (FILE: GBHMT1.WK3)

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APPENDIX I

THE EFFECT OF IONIC MIGRATION ON THE LIMITING CURRENT

Faraday's law was used to calculate the mass transfer rate to the electrode to the measured limiting current (see section 2.2)

\[ N_A = \frac{I_L}{z_i F A} \]  \hspace{1cm} (I.1)

It was assumed that an excess of supporting electrolyte in a very dilute electrolyte solution will prevent the effect of ionic migration. Following the approach presented by Dawson (1968), equation I.1 can be modified to include the effect of migration (Dawson, 1968; Wilke et al., 1952):

\[ N_A = \frac{I_L}{z_i F A} \left( 1 - T_A \right) \]  \hspace{1cm} (I.2)

\( T_A \) is the transference number for the ferricyanide ions under investigation, and is a measure of the fraction of the total current carried by the ion. The transference number is given by (Dawson, 1968)

\[ T_A = \frac{C_{ferm} \Lambda_{ferm}}{\sum C_i \Lambda_i} \]  \hspace{1cm} (I.3)

The equivalent conductance for each of the ions in a very dilute electrolyte system is presented in Table I.1.
Table 1.1 Equivalent Conductances for the Ferricyanide / Ferrocyanide System

<table>
<thead>
<tr>
<th>Ion</th>
<th>$A^0$ (cm$^2$ / equiv. ohm) (25°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>OH$^-$</td>
<td>197.0</td>
</tr>
<tr>
<td>Na$^+$</td>
<td>50.0</td>
</tr>
<tr>
<td>K$^+$</td>
<td>73.5</td>
</tr>
<tr>
<td>1/3 Fe(CN)$_6$$^{3-}$</td>
<td>101.0</td>
</tr>
<tr>
<td>1/4 Fe(CN)$_6$$^{4-}$</td>
<td>111.0</td>
</tr>
</tbody>
</table>

The nominal electrolyte composition used in the present work was approximately:

NaOH 2.00 mol/l
K$_3$Fe(CN)$_6$ 0.025 mol/l
K$_4$Fe(CN)$_6$ 0.025 mol/l

By neglecting the effect of concentration on the equivalent conductance, equation 1.3 can be used to approximate the transference number (after Dawson, 1968):

$$T_A = \frac{0.025 \frac{mol}{1000cm^3} \cdot 101 \frac{cm^2}{\Omega}}{(0.025(101 + 111) + 0.175 \cdot 73.5 + 2.0 \cdot 197.6 + 2.0 \cdot 50.0) \frac{mol}{1000cm^3} \frac{cm^2}{\Omega}}$$

$$T_A = 4.92 \cdot 10^{-3}$$

Since the result of the above calculation is much less than one, it can be concluded that the assumption to neglect the effect of ionic migration is a valid one. This assumption will introduce an error into the estimation of the mass transfer coefficient of less than 0.5%.
APPENDIX J

ESTIMATION OF MEASUREMENT ERROR

The estimated percentage error for each measured variable over the experimental range under investigation is summarized in Table J.1

**Table J.1 Estimation of the Measurement Errors**

<table>
<thead>
<tr>
<th>Variable</th>
<th>Value or Range</th>
<th>Maximum Probable Error</th>
<th>Maximum Probable Error</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>(Absolute)</td>
<td>(Relative [%])</td>
</tr>
<tr>
<td><strong>L</strong></td>
<td>1.5 cm ... 15 cm</td>
<td>0.005 cm</td>
<td>0.25</td>
</tr>
<tr>
<td><strong>d_L</strong></td>
<td>1.714 cm</td>
<td>0.005 cm</td>
<td>0.5</td>
</tr>
<tr>
<td><strong>A</strong></td>
<td>30 cm²</td>
<td></td>
<td>0.5</td>
</tr>
<tr>
<td><strong>T</strong></td>
<td>25°C ... 55°C</td>
<td>0.25</td>
<td>1.0</td>
</tr>
<tr>
<td><strong>C_{NaOH}</strong></td>
<td>0.025 mol/l; 50 ml Titration</td>
<td>0.1 ml</td>
<td>0.2</td>
</tr>
<tr>
<td><strong>C_{HCl}</strong></td>
<td>2.0 mol/l; 25 ml Titration</td>
<td>0.1 ml</td>
<td>0.4</td>
</tr>
<tr>
<td><strong>Rotameter</strong></td>
<td>10 ... 100</td>
<td>0.5 units</td>
<td>5.0</td>
</tr>
<tr>
<td><strong>I_L</strong></td>
<td>10 mA ... 700 mA</td>
<td></td>
<td>2.5</td>
</tr>
<tr>
<td><strong>Angle (α)</strong></td>
<td>0° ... 180°</td>
<td>1°</td>
<td>0.5</td>
</tr>
<tr>
<td><strong>Slope (θ)</strong></td>
<td>0° ... 90°</td>
<td>1°</td>
<td>1</td>
</tr>
</tbody>
</table>
The maximum probable error for the calculated variables is summarized in Table J.2.

**Table J.2 Maximum Probable Errors**

<table>
<thead>
<tr>
<th>Variable</th>
<th>Source of Error</th>
<th>Maximum Probable Error (Absolute)</th>
<th>Maximum Probable Error (Relative [%])</th>
</tr>
</thead>
<tbody>
<tr>
<td>ρ</td>
<td>Temperature</td>
<td></td>
<td>0.1</td>
</tr>
<tr>
<td></td>
<td>$C_{p,\text{cml}}$</td>
<td></td>
<td>0.4</td>
</tr>
<tr>
<td></td>
<td>Correlation</td>
<td></td>
<td>2.2</td>
</tr>
<tr>
<td></td>
<td>TOTAL</td>
<td></td>
<td>2.9</td>
</tr>
<tr>
<td>ν</td>
<td>Temperature</td>
<td></td>
<td>0.4</td>
</tr>
<tr>
<td></td>
<td>$C_{v,\text{cml}}$</td>
<td></td>
<td>0.4</td>
</tr>
<tr>
<td></td>
<td>Correlation</td>
<td></td>
<td>3.4</td>
</tr>
<tr>
<td></td>
<td>TOTAL</td>
<td></td>
<td>4.2</td>
</tr>
<tr>
<td>D</td>
<td>Temperature</td>
<td></td>
<td>0.4</td>
</tr>
<tr>
<td></td>
<td>$C_{D,\text{cml}}$</td>
<td></td>
<td>0.4</td>
</tr>
<tr>
<td></td>
<td>Correlation</td>
<td></td>
<td>4.3</td>
</tr>
<tr>
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<td></td>
<td>5.1</td>
</tr>
<tr>
<td>Volume Flow</td>
<td>Rotameter Reading</td>
<td>2</td>
<td>2</td>
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<tr>
<td></td>
<td>Calibration</td>
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<td>1</td>
</tr>
<tr>
<td></td>
<td>TOTAL</td>
<td></td>
<td>3</td>
</tr>
</tbody>
</table>

Mickley et al. (1957) described an error estimation scheme based on the partial derivative approach. This method for estimation of maximum probable error will be used to estimate the error in the dimensionless variables. In the following section the error in the Reynolds number (equation J.1) for experiment NR1 will be calculated using the above method. The data were taken from the 'Data Summary' (Appendix L).

\[ Re = \frac{Ud}{v} \]  

(J.1)
The partial derivatives needed for use in this method are:

\[
\frac{\partial Re}{\partial U} = \frac{d_s}{v} \tag{J.2}
\]

\[
\frac{\partial Re}{\partial d_s} = \frac{U}{v} \tag{J.3}
\]

\[
\frac{\partial Re}{\partial v} = -\frac{U d_s}{v^2} \tag{J.4}
\]

Consequently, \(\Delta Re\), can be estimated using equation (J.5)

\[
\Delta Re = \frac{\partial Re}{\partial U} \Delta U + \frac{\partial Re}{\partial d_s} \Delta d_s + \frac{\partial Re}{\partial v} \Delta v \tag{J.5}
\]

By replacing the partial derivatives equation (J.5) can be written:

\[
\Delta Re = \frac{d_s}{v} \Delta U + \frac{U}{v} \Delta d_s - \frac{U d_s}{v^2} \Delta v \tag{J.6}
\]

Using the information from Tables J.1 and J.2, \(\Delta U\), \(\Delta d_s\), and \(\Delta v\) can be calculated:

- \(\Delta U = 2.49 \cdot 10^4 \text{cm/s} \) (3.0% of 8.3 cm/s)
- \(\Delta d_s = 8.57 \cdot 10^2 \text{cm} \) (0.5% of 1.714 cm)
- \(\Delta v = 4.77 \cdot 10^4 \text{cm}^2/\text{s} \) (4.2% of 0.01134 cm²/s)

With the above information, \(\Delta Re\) can be calculated:

\[
\Delta Re = \frac{1.714\text{cm} \cdot 2.49 \cdot 10^4 \text{cm/s}}{0.01134 \text{cm}^2/\text{s}} \cdot \frac{8.3 \text{cm}}{s} + \frac{8.3 \text{cm}}{s} \cdot 0.00857 \text{cm} + \frac{8.3 \text{cm}}{s} \cdot 1.714 \text{cm} \cdot 4.77 \cdot 10^4 \text{cm}^2/\text{s} \cdot \frac{0.01134 \text{cm}^2}{s} \tag{J.7}
\]

\[
\Delta Re = 96.7
\]
In order to obtain the maximum error, the sign of $\Delta v$ was taken positive in the above equation (1.7). Since the approximate value of the Reynolds number is 1255 the maximum percentage error is $\pm 7.7\%$. The same procedure was used to determine the maximum error of the other dimensionless groups (see Table J.3).

It is evident that equation J.7 probably overestimates the error involved in the measurement. It takes no account of the possibility of compensating errors. Even more serious is its failure to take into account the method used to obtain the original estimates of the uncertainties in the directly measured quantities. But nevertheless this method can be used for a reasonable estimate of the maximum probable error.

### Table J.3 Maximum Probable Error for the Dimensionless Groups

<table>
<thead>
<tr>
<th>Dimensionless Group</th>
<th>Equation Number</th>
<th>Maximum Probable Error [%] (Worst Case)</th>
</tr>
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<tbody>
<tr>
<td>Re</td>
<td>C.16</td>
<td>8</td>
</tr>
<tr>
<td>Pr</td>
<td>C.18</td>
<td>6</td>
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<tr>
<td>Sc</td>
<td>C.17</td>
<td>9</td>
</tr>
<tr>
<td>Gr</td>
<td>C.23</td>
<td>10</td>
</tr>
<tr>
<td>Gr_t</td>
<td>C.21</td>
<td>12</td>
</tr>
<tr>
<td>Sh</td>
<td>C.19</td>
<td>10</td>
</tr>
</tbody>
</table>

The data presented above are error estimates for a single data point. However, within a set of experiments some of the measurement errors are eliminated by holding a quantity constant, e.g. the cathode angle and the channel slope do not change.
APPENDIX K
NOMENCLATURE

Nomenclature

\( a_{\text{ox}} \)  Activity of oxidized species
\( a_{\text{red}} \)  Activity of reduced species
\( a_i \text{ to } a_7 \)  Regression coefficient
\( a_r \)  Tafel constant
\( A \)  Cathode area, cm\(^2\)
\( b \)  0.5 \cdot Channel height, cm
\( b_r \)  Tafel constant
\( c \)  Concentration, mol/cm\(^3\)
\( c_{10} \)  Concentration of species 1, mol/cm\(^3\)
\( c_{20} \)  Concentration of species 2, mol/cm\(^3\)
\( c_{10}^{\text{ref}} \)  Reference concentration of species 1, mol/cm\(^3\)
\( c_{20}^{\text{ref}} \)  Reference concentration of species 2, mol/cm\(^3\)
\( c_1 \text{ to } c_6 \)  Constants in various equations
\( c_i \)  Concentration of species i, mol/cm\(^3\)

\( \nabla c_i \)  Concentration gradient
\( c_p \)  Thermal capacity, J/(kg K)
\( c_{\text{ref}} \)  Reference concentration, mol/cm\(^3\)
\( c_e \)  Empirical constant
\( C_{03} \)  Bulk concentration of ferricyanide ions in Table A.1.1, mol/l

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C_04  Bulk concentration of ferrocyanide ions in Table A.1.1, mol/l
C_A  Concentration of species A, mol/l
C_B  Bulk concentration, mol/l
C_{femB}  Bulk concentration of ferricyanide ions, mol/l
C_{femF}  Film concentration of ferricyanide ions, mol/l
C_{femS}  Surface concentration of ferricyanide ions, mol/l
C_{femB}  Bulk concentration of ferrocyanide ions, mol/l
C_{femF}  Film concentration of ferrocyanide ions, mol/l
C_{femS}  Surface concentration of ferrocyanide ions, mol/l
C_{Na}  Bulk concentration of caustic soda in Table A.1.1, mol/l
C_{NaOH}  Bulk concentration of caustic soda, mol/l
C_{NaOHF}  Film concentration of caustic soda, mol/l
C_{NaOHs}  Surface concentration of caustic soda, mol/l
C_o  Concentration of species at the electrode surface, mol/l
C_{redox}  Concentration of species i, mol/l
d_e  Equivalent diameter, cm
D_{03}  Diffusion coefficient of potassium ferricyanide ions, cm²/s
D_{04}  Diffusion coefficient of potassium ferrocyanide ions, cm²/s
D  Diffusion coefficient, cm²/s
D_P  Diffusion coefficient of ferricyanide ion at film conditions, cm²/s
D_T  Turbulent diffusion coefficient, cm²/s
E  Applied voltage, V
E_L  Voltage drop at 1.238 Ohm resistor, V
E_r  Cell voltage, V
E_r^o  Electrode potential under standard condition, V
F  Faraday's constant = 96,485 A s/mol
\( g \)  
Acceleration of gravity = 981 \( \text{cm/s}^2 \)

\( \Delta G \)  
Change in free energy, J/mol

\( h \)  
Heat transfer coefficient, J/(m\(^2\) s K)

\( i \)  
Current, A/cm\(^2\)

\( i_L \)  
Limiting current density, A/cm\(^2\)

\( l_m \)  
Mixing length, m

\( I \)  
Isothermal experiment

\( IR \)  
Preliminary isothermal experiment

\( IR \)  
Repeated isothermal experiment

\( I_L \)  
Limiting current, A

\( k \)  
Turbulent kinetic energy, J

\( K \)  
Average mass transfer coefficient, cm/s

\( L_m \)  
Mixing length, cm

\( l_t \)  
Turbulent length, cm

\( L \)  
Cathode length, cm

\( n \)  
Exponents in equation (3.4.5), (3.4.6)

\( N \)  
Non-isothermal experiment

\( NP \)  
Preliminary non-isothermal experiment

\( NR \)  
Repeated non-isothermal experiment

\( P \)  
Fluid pressure, N/mm\(^2\)

\( O \)  
Order of ....

\( R \)  
Radius, cm

\( R_e \)  
Cell resistance, \( \Omega \)

\( R_g \)  
Gas constant, 8.3143 J/(K mol)

\( R_L \)  
'Voltage drop' resistor, 1.238 \( \Omega \)
$S_s$  Small rotameter reading, \%  
$S_m$  Medium rotameter reading, \%  
$S_l$  Large rotameter reading, \%  
$s$  Function of the aspect ratio  
$t$  Time, s  
$T$  Absolute temperature, K  
$T_A$  Transference Number  
$T_0$  Reference temperature, °C  
$T_1$  Upstream bulk solution temperature (x=30mm), °C  
$T_2$  Upstream cathode surface temperature (x=10mm), °C  
$T_3$  Downstream cathode surface temperature (x=140mm), °C  
$T_4$  Downstream bulk solution temperature (x=180mm), °C  
$T_B$  = $T_1$ = Bulk temperature, °C  
$T_C$  = $T_2$ = Upstream cathode temperature, °C  
$T_F$  = ($T_1 + T_2$)/2, Film temperature, °C  
$T_S$  = ($T_3 + T_4$)/2, Cathode surface temperature, °C  
$\Delta T$  = $T_2 - T_1$, °C  
$\mu_i$  Ionic mobility, cm$^2$-mol/(J·s)  
$u_x$  Local velocity in x-direction, cm/s  
$u_y$  Local velocity in y-direction, cm/s  
$u_z$  Local velocity in z-direction, cm/s  
$U$  Mean velocity, cm/s  
$U_0$  Maximum velocity, cm/s  
$V$  Volumetric flow rate, m$^3$/s  
$W$  Mass flow, kg/s  
$x$  x-distance from leading edge of cathode, cm
$x_e$  Hydrodynamic entrance length, cm

$y$  $y$-distance from leading edge of cathode, cm

$y_1$  Channel height, 1.5 cm

$z$  $z$-distance from leading edge of cathode, cm

$z_1$  Channel width, 2.0 cm

$z_e$  Number of electrons in reduction reaction
Dimensionless Groups

Le = 1/Pe

Nu = h d /k

Pe = U d /α

Pr = ν/α

Prₘ = Empirical constant

Prᵣ = (cᵣ μᵣ /λᵣ)

Prₜ = Empirical constant

Re = (ρU d /μ)

Sc = μ(ρD)

Scᵣ = μᵣ(ρ Dᵣ)

Sh = K d /D

Shᵣ = K d /x

St = Sh(Re Sc) = hₜ /((ρ F U Cₜ)

Gr = (Δρ g L²)/ρ ν²

Grᵣ = (Δρᵣ g L²)/ρ ν²

GRₘ = Grₘ + (Pr/Sc)½ Grₚ

GRₜ = Grₚ + (Sc/Pr)½ Grₚ

Gz = W cₚ /k L
Greek Letters

\( \alpha \) Cathode angle, °

\( \alpha_c \) Mass diffusivity, cm²/s

\( \alpha_i \) Ionic constant of species i, J/(m² s K)

\( \alpha_s \) Thermal diffusivity, cm²/s

\( \beta \) Channel slope, °

\( \beta_v \) Volumetric expansion coefficient due to temperature gradient, 1°C

\( \beta_c \) Volumetric expansion coefficient due to concentration gradient, cm³/mol

\( \beta_{c1} \) Volumetric expansion coefficient due to concentration gradient 1, cm³/mol

\( \beta_{c2} \) Volumetric expansion coefficient due to concentration gradient 2, cm³/mol

\( \Gamma \) Ionic strength, mol/l

\( \gamma \) Aspect ratio

\( \delta_h \) Hydrodynamic boundary layer thickness, cm

\( \delta_c \) Concentration boundary layer thickness, cm

\( \delta_t \) Thermal boundary layer thickness, cm

\( \varepsilon \) Viscous dissipation rate of turbulent kinetic energy

\( \eta \) Overpotential, V

\( \eta_c \) Concentration overpotential, V

\( \eta_o \) Ohmic overpotential, V

\( \eta_s \) Surface overpotential, V

\( \Lambda_e \) Equivalent conductance, cm²/Ω

\( \lambda \) Thermal conductivity, W/(m K) (denoted as \( k_t \) in the 'Data Summary': Appendix L)

\( \lambda_r \) Turbulent thermal conductivity, W/(m K)

\( \lambda_w \) Thermal conductivity of water, W/(m K)

\( \mu \) Effective dynamic viscosity, Pa s

\( \mu_o \) Dynamic viscosity, Pa s
\( \mu_t \quad \text{Turbulent dynamic viscosity, Pa s} \)

\( \nu \quad \text{Kinematic viscosity, cm}^2/\text{s} \)

\( \nu_f \quad \text{Kinematic viscosity at film conditions, cm}^2/\text{s} \)

\( \nu_T \quad \text{Turbulent (eddy) kinematic viscosity, cm}^2/\text{s} \)

\( \rho_o \quad \text{Reference density, g/cm}^3 \)

\( \rho_b \quad \text{Bulk density, g/cm}^3 \)

\( \rho_f \quad \text{Film density, g/cm}^3 \)

\( \rho_s \quad \text{Surface density, g/cm}^3 \)

\( \Delta \rho_c \quad \text{Density difference due to concentration gradient alone, g/cm}^3 \)

\( \Delta \rho_t \quad \text{Density difference due to thermal gradient alone, g/cm}^3 \)

\( \Sigma \quad \text{Sum of ...} \)

\( \nabla \phi \quad \text{Ionic migration across an electric field} \)

\( \chi \quad \text{Function of aspect ratio} \)

\( \omega \quad \text{Angle velocity, rad/s} \)

\( \nabla p \quad \text{Pressure force on element per unit volume} \)

\( \nabla \tau \quad \text{Viscous force on element per unit volume} \)
APPENDIX L

RAW DATA AND RESULTS

It was decided not to include the raw data and the derived quantities in the thesis, due to space considerations (491 additional pages) and the fact that all experimental results are plotted in Appendix E. A complete summary of all experimental raw data and the derived quantities presented in this work is available in a 491 page "Data Summary" stored in the Research Supervisors office and on 3.5" high density diskettes saved under the disk operating system MS-DOS® Version 5.0 (see Appendix D).

The 'Data Summary' contains the data of the Isothermal Preliminary (IP1..IP336), Isothermal Replicate (IR1..IR200) and Isothermal (I1..I3277) experiments.

The isothermal 'Data Summary' includes:

- Experiment Number
- Rotameter reading, [size, %]
- Volumetric flow rate, [m³/s]
- Bulk velocity, [m/s]
- Voltage drop across $R = 1.238\Omega$, [mV]
- Limiting current, [mA]
- Bulk solution temperature, $T_b$, [°C]
- Caustic soda bulk concentration, [mol/l]
- Potassium ferricyanide bulk concentration, [mol/l]
- Potassium ferrocyanide bulk concentration, [mol/l]
- Density, [kg/m³]
- Viscosity, [m²/s]
- Diffusivity, [m²/s]
- Cathode angle, [°]
- Channel slope, [°]
- Reynolds number, [-]
- Schmidt number, [-]
- Average mass transfer coefficient, [m/s]
- Sherwood number, [-]
- Date when experiment was performed, [DD/MM/YY]
- Experiment when electrodes were cleaned
- Experiment when electrolyte composition was determined

In addition the 'Data Summary' contains data of the Non-Isothermal Preliminary (NP1..NP504),
Non-Isothermal Replicate (NR1..NR150) and Non-Isothermal (N1..N2394) experiments.

The non-isothermal 'Data Summary' includes:

- Experiment Number
- Rotameter reading, [size, %]
- Volumetric flow rate, [m$^3$/s]
- Bulk velocity, [m/s]
- Voltage drop across $R = 1.238\Omega$, [mV]
- Limiting current, [mA]
- Temperature, $T_1$, [°C]
- Temperature, $T_2$, [°C]
- Temperature, $T_3$, [°C]
- Temperature, $T_4$, [°C]
- Surface temperature, [°C]
- Film temperature, [°C]
- Cathode heater voltage, [V]
- Cathode heater amperage, [A]
- Caustic soda bulk concentration, [mol/l]
- Potassium ferricyanide bulk concentration, [mol/l]
- Potassium ferrocyanide bulk concentration, [mol/l]
- Bulk density, [kg/m$^3$]
- Density at film conditions, [kg/m$^3$]
- Density at surface conditions, [kg/m$^3$]
- Viscosity at film conditions, [m$^2$/s]
- Diffusivity at film conditions, [m$^2$/s]
- Thermal conductivity, [J/(m·s·K)]
- Heat capacity, [J/(kg·K)]
- Cathode angle, [°]
- Channel slope, [°]
- Reynolds number, [-]
- Schmidt number, [-]
- Prandtl number, [-]
- Grashof number for heat transfer, [-]
- Grashof number for mass transfer, [-]
- Combined Grashof number for mass transfer, [-]
- Average mass transfer coefficient, [m/s]
- Sherwood number, [-]
- Date when experiment was performed, [DD/MM/YY]
- Experiment when electrodes were cleaned
- Experiment when electrolyte composition was determined
APPENDIX M

SIMULATION RESULTS

While the experimental conditions were for both developing thermal and diffusion boundary layers, there is clear evidence in the plots of the simulated local mass transfer coefficients that they have reached or almost reached their final value. Thus values of mass transfer coefficients for fully developed thermal and diffusion boundary layers can be estimated through this approach (see Table M.1).

<table>
<thead>
<tr>
<th>Re</th>
<th>Sc</th>
<th>Pr</th>
<th>β</th>
<th>T₁</th>
<th>T₂</th>
<th>K_{EXP} (10^4 cm/s) developing flow</th>
<th>K_{SIM} (@ x = 15 cm) (10^4 cm/s) fully developed B.L.</th>
</tr>
</thead>
<tbody>
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<td>266</td>
<td>2349</td>
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<td>$K_{Sim}$ (@ $x = 15$ cm)</td>
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REFERENCES


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