MODELLING AND OPTIMAL CONTROL OF COUNTERCURRENT ION EXCHANGE PROCESS

Ntuthuko Marcus Dube

Supervisor: Prof. Dr. Raynichka Tzoneva

Department of Electrical Engineering

Peninsula Technikon

A dissertation submitted to fulfil requirements for Master of Technology (MTech) in Electrical Engineering, Peninsula Technikon, Bellville. January 2002.

STATEMENT

The content of this dissertation represents the work of the author (N.M Dube) and the opinions contained herein are the author's and do not necessarily reflect that of Peninsula Technikon. Apart from normal guidance and instructions from the supervisor (Prof. R. Tzoneva) the work presented herein is the author's.

No part of this work has been submitted by the author in support of an application for another degree or any other qualification, from the Peninsula Technikon or any other Institution of learning.

This dissertation is dedicated to my mother and father
Josephine Nokuthula and Luke Pika Dube

ACKNOWLEDGEMENTS

The author would like to thank everybody who was directly or indirectly involved in this project more especially the Creator who kept me healthy. All those words of encouragement from my family and friends did contribute and here are the results.

Firstly the author would like to mention the NATIONAL RESEARCH FOUNDATION (NRF) previously the FOUNDATION OF RESEARCH AND DEVELOPMENT (FRD) who funded the project from the beginning till its end of which without them this project will not have been impossible.

Prof. Raychnika Tsoneva for her inspirational supervision and guidance. She became a mother, a mentor, a tutor and the light. She proved that, "*until you realize how little do you know, you really need to start looking for that knowledge.*" Mr. Paul Tennant for believing in me and paving me the way to start my post diploma studies. The author does not to forget all the other staff members of Electrical Engineering, Peninsula Technikon who took interest in this project, especially Dr. Ogembo M. Kachienga including staff of Mechanical Engineering Department who were part of the research activity of the Faculty of Engineering of Peninsula Technikon. The author would also like to thank all the individuals from the Chemical Engineering Department of Peninsula Technikon who were involved in the project.

ACKNOWLEDGEMENTS

My colleagues (MTech's) at Electrical Engineering, Peninsula Technikon for the support and pain absorption. *"Guys whoever does not believe in you, does not believe in the power of existence and thus marginalizing himself, hold up the truth."* Your personal support to me was more than just brotherhood, you were a home away from home (you know what we went through). My other colleagues BTech's and Diploma students who worked on the plant.

People who were indirectly involved in this project are my family, thank you, to you all for your support and understanding.

If at any stage during the proceedings of this project you ever asked the author the question, "*HOW IS YOUR PROJECT GOING*?" it means you were indirectly involved in this project, thank you for your support. If you believe that at any one time you offered the author support and you feel that it is not reflected above, count yourself in, thank you very much.

ABSTRACT

The problem for wastewater treatment is very important these days because of population increase and industry development. Ion exchange technology has proved its positive qualities in domestic lives and industry, but the ion exchange process still needs deeper understanding and improving. That is why a new pilot plant has been built in Chemical Engineering Department at Peninsula Technikon.

In addition to the treatment of domestic effluents there are other processes which the ion exchange is suited for, such as the recovery and the reuse of industrial effluents. The proposed control system is developed in such a way that it does not have to depend on a certain effluent.

The selection of the process and the development of techniques for its control fall into the national needs of improving people's lives (e.g. giving them excess to clean water) and quality of the environmental conditions (treatment of toxic waste substances).

There is a need for an application area for modeling and control methods developed in the field of control engineering for the Department of Electrical Engineering; on the other hand a need of control techniques for the development in the Department of Chemical Engineering technological process. This multidisciplinary liaison between the two Engineering departments promotes joint research activities and relevance between them. It also equips the graduating engineer with the relevant experience into working in a team of multidisciplinary engineering fields.

ABSTRACT

Community and industrial relevance of the research study is that in addition to treatment of domestic effluents, the ion exchange process is also particularly well suited for: the desalination of hard waters, combined neutralization and desalination of alcohol effluents, such as mine drainage, acid mineral tailings, paper bleach affluent, the pretreatment of reverse osmosis feed for the removal of organic and colloidal particulate.

OBJECTIVE

Ion exchange processes used for the removal of salts from water employ a method that involves passing water through cylindrical columns of cation-exchange and anion-exchange beads in the H^+ and OH^- respectively. The emphasis of the research work will be on the reuse of domestic water supplies, starting with a secondary sewage effluent as feed to the cation-column and producing water of a potable quality.

The pilot plant for the ion exchange for treatment of sewage effluents was then built in the Department of Chemical Engineering at the University of Cape Town between 1980 and 1982 but this system does not exist anymore. But the problem for wastewater treatment is so important these days that a new pilot plant has been built in the Chemical Engineering Department. A new control system had to be designed and this project formed the basis for the research work done to design and implement this control system. The ion exchange process and the control system developed in 1980 had some disadvantages according to their proper and optimal work:

- As the up-flow cycle times were determined separately for each column on the basis of the feed concentration, resin capacity and the required stoichiometric ratio, the cycles come in and out of phase.
- The startup time for the process was too long. The time taken from the process to reach a new steady state when the feed water concentration changed was also too long.
- The used model considered only steady state operating conditions.

OBJECTIVE

- The closed loop controls were not built into the previous system.

On the bases of the above mentioned disadvantages the following problem for optimal control may be formulated:

- To maintain the operating conditions such that the required quality of water is produced at a constant throughput for the minimum consumption of regenerant chemicals.

According to goals, the main purpose of the research project is to develop a strategy for optimal identification and control synthesis and to use them for simulation and the real time with the new pilot plant. The research projected arriving at a deeper understanding of the ion exchange process, using new methods and models for its description and control.

LIST OF FIGURES

LIST OF FIGURES

LIST OF FIGURES

LIST OF TABLES

ABBREVIATIONS

A/D	Analog to Digital
AC	Alternating current
ADC	Analog to Digital Converter
ADPT5050	Adapter-5050
AGND	Analog Ground
BV	Bed Volume
CCIX	Continuous Countercurrent Ion Exchange
CD_ROM	Compact Disk_Read Only Memory
CH0 – CH15	Channels 0 to 15
COD	Chemical Oxygen Demand
CPU	Central Processing Unit
DAQ	Data acquisition
DB-50	50 PIN Connector
DB50F/M	Female/Male Interface Cable
DC	Direct current
DIO	Digital Input/Output Lines
DIOIN	Digital Input/Output Line Input
DIOUT	Digital Input/Output Line Output
DMA	Direct Memory Access
DVB	Divinylbenzene
EDR	Eagle Technology Drivers
FIFO	First In First Out
I/O	Input/output
IDC5	Input DC module
IDC-50	Male type connector
IDE-50	Male connector

ABBREVIATIONS

IX	Ion Exachange
J1 – J17	Jumper settings from 1 to 17
LabVIEW	Laboratory Virtual Instruments Engineering Workbench
LSB	Least Significant Bit
MIMO	Multiple Input Multiple Output
MINTEK	Council for Minerals Technology
NI	National Instruments
NIM	National Institute of Metallurgy (later MINTEK)
NIM^{TM}CCIX	National Institute of Metallurgy Continuous Countercurrent Ion Exchange (Trade mark)
NMS	National Management Services
OAC5A	Output AC module
PB 24H	Analog/Digital Mounting Rack
PC 38X	Current Interface Card
PC 71	Current to Voltage Converter Card
PC	Personal Computer
PC30GA_ADV	Data Acquisition Interface Card
pH	Acidic/Alkalinity Scale
PI	Proportional Intergral
PID	Proportional Integral Derivative
RO	Reverse Osmosis
SE	Single Ended Input
SISO	Single Input Single Output
TDS	Total Dissolved Oxygen
TL1 – TL7	Connector block 1 to 7

NOMENCLATURE

(Chapter One)

C_f	Liquid concentration in the 1st stage [*mol/l*]
C_t	Liquid concentration in the last stage [*mol/l*]
e_v	Voidage
F_R	Resin flow rate [*mol/h*]
F_L	Liquid flow rate [*mol/h*]
h	Resin holdup
H^+	Hydrogen ion
H	Hydrogen
Na	Sodium
Na^+	Sodium ion
$NaCl$	Sodium chloride
OH^-	Hydroxide
q_f	Resin concentration in the 1st stage [*mol/l*]
q_t	Resin concentration in the last stage (top) [*mol/l*]
T	Periodic time [h]
V_R	Resin volume [*l*]

NOMENCLATURE

(Chapter Two)

a	Area where the exchange occurs
a_A	Solution phase concentration of ion species A
a_B	Solution phase concentration of ion species B
$\bar{a}_A$	Resin phase concentration of ion species A
$\bar{a}_B$	Resin phase concentration of ion species B
Ap	Apparent area of the total area of the stage plate
A^+	A cation attached to a cation exchange medium
B^+	A co-ion (cation) taking part in a cation exchange
$\underline{C}$	Total ion concentration in solution phase
C_A	Concentration of species A in solution phase
$Ca(OH)_2$	Lime (Calcium Hydroxide)
C^*	Resin operating capacity [eq/m^3]
C_A^*	Ion concentration of species A at resin surface in equilibrium
Ca^{++} (Ca^{2+})	Calcium ion
$CaCl_2$	Calcium Chloride
C_B	Concentration of species B in solution
C_d	Concentration of d component
Cl^-	Chloride ion
$-COOH$	Carboxylic acid group
D	Diffusion coefficient
$\underline{D}$	Diffusion constant
$\bar{C}$	Resin capacity [kg.eq.m^{-3}]
$\bar{C}_A$	Concentration of ion species A in resin phase
$\bar{C}_B$	Concentration of ion species B in resin phase
C_i	Solution phase concentration of i component
$\bar{C}_i$	Resin phase concentration of i component

NOMENCLATURE

(Chapter Two)

H	Hydrogen
H^+	Hydrogen ion
H_2O	Water molecule
$J_{d,z}$	Flux of diffusion of ion species of concentration C_d
m_A	Ionic concentration of ion species A in solution phase
m_B	Ionic concentration of ion species B in solution phase
m_A^*	Distribution coefficient
$\overline{m}_A$	Concentration of ion species A in resin phase
$\overline{m}_B$	Concentration of ion species B in resin phase
Mg	Magnesium
Mg^{++} (Mg^{2+})	Magnesium ion
Na	Sodium
Na^+	Sodium ion
$N(CH_3)_2$	Tertiary amine
NCH_3OH	Quaternary ammonium group
OH^-	Hydroxide
$\bar{x}_A$	Equivalent fraction of resin in A in resin phase
$\overline{m}_B$	Equivalent fraction of resin in B in resin phase
k_a	Thermodynamic equilibrium constant
kD	Diffusivity coefficient
k_L	Mass transfer coefficient
k_B^A	Selectivity coefficient
k'^A_B	Molar selectivity coefficient
$(k_c)_B^A$	Selectivity coefficient ionic equivalent fraction
${}^N k_B^A$	Rational selectivity coefficient

NOMENCLATURE
(Chapter Two)

m_i	Ionic concentration of *i* component in solution phase
$\overline{m}_i$	Ionic concentration of *i* component in resin phase
N_A	Flux of ion species A (particle diffusion rate)
Q	Liquid flow rate [m^3/l]
q_A	Concentration of ion species A at the resin bead surface
$\overline{q}_A$	Mean concentration of species A within the bead
R	Rate of exchange
r	resin bead adius
R^+	Resin of cation form medium
R^-	Resin of anion form medium
R-H	Resin in hydrogen form
R'-N	Weak base resin
R-Na	Sodium loaded cation resin
R-NHCl	Weak base resin (*R-N*) loaded with hydrochloric (*HCl*) molecule
Si^{--} (Si^{2+})	Silica
SO_4^{--} (SO_4^{2+})	Sulphate
S_R	Salinity to be absorbed by the resin [eq/m]
t	Operating time [h]
V_R	Resin volume [m^3]
x_i	Equivalent of ion fraction of *i* component in solution phase
$\overline{x}_i$	Equivalent of ion fraction of *i* component in resin phase
x_A	Equivalent of ion fraction of ion species A in solution phase
x_B	Equivalent of ion fraction of ion species B in solution phase
$\overline{x}_A$	Equivalent of ion fraction of ion species A in resin phase
$\overline{x}_B$	Equivalent of ion fraction of ion species B in resin phase

NOMENCLATURE

(Chapter Two)

X^-	A anion attached to a anion exchange medium
Y^-	A co-ion (anion) taking part in a anion exchange
y_A	Concentration in solution phase of counter ion A
y_B	Concentration in solution phase of counter ion B
Z_A	Valence of element A
Z_B	Valence of element B
$\lvert Z_A \rvert$	Absolute values of electrochemical valence of ion species of A
$\lvert Z_B \rvert$	Absolute values of electrochemical valence of ion species of B
z_l	Resin bead length parameter
α_B^A	Separation factor
ϕ_A	Overall coupled flux of counter ion A
ϕ_B	Overall coupled flux of counter ion B
λ_i	Distribution coefficient

NOMENCLATURE

(Chapter Three)

vA	Model matrix of l_n parameters
a_n	Experimental coefficient of the n^{th} stage
ax	Experimentally determined gradient
A^*	Matrix of known nonlinear function $y(t)$ and $u(t)$ at moments $t_1, t_2, \ldots, t_K$.
AW	Atomic weight
AW_H	Atomic weight for hydrogen
AW_{Na}	Atomic weight for sodium
ΔA^*	Correction errors of matrix A^*
B	Model matrix of $m_{n,n}$ parameters
B_1	Model matrix of k_n parameters
b_n	Experimental coefficient of nth stage
C	Model output matrix
$C_{H,f}$	Hydrogen concentration in liquid phase into first stage
$C_{H,t}$	Hydrogen concentration in liquid phase at the top stage
$C_{n,H}$	Concentration of hydrogen ions in liquid phase in the n^{th} stage
$C_{n,Na}$	Concentration of sodium ions in solution phase in the n^{th} stage
$C_{n,Na}$	Concentration of sodium ions in liquid phase in the n^{th} stage
$C_{Na,f}$	Sodium concentration in liquid phase getting into the stage 1
$C_{Na,t}$	Sodium concentration in liquid phase on the top stage
E	Residual error vector
e_i	Residual errors at moment i
F	Periodic control vector
F_j	m arbitrary independent functions of unknown parameter
$F_{L,f}$	Liquid flow rate entering the first stage (stage 1)
$F_{L,t}$	Liquid flow rate flowing out of the top stage (last stage)
$F_{R,f}$	Resin flow rate leaving the first stage

NOMENCLATURE
(Chapter Three)

F_R^s	Resin flow rate in steady state
$F_{R,t}$	Resin flow rate going in to the top stage
H^*	Scalar positively determined matrix of an error between the output of the model and real process model
H^+	Hydrogen ions
H_n	Liquid ionic holdup of liquid in the n^{th} stage
h_n	Resin ionic holdup of resin in the n^{th} stage
J	Criterion
j	Index of calculation
Jp	Optimum criterion for p-values
K	Number of steps in the sampling period (final value for the sampling points)
k_n	Parameters of the model connecting b_n, H and h in the n^{th} stage
l_n	Parameters of the model connecting F_L, an, H and h in the n^{th} stage
m	Number of parameters
M	Number of iterations
m	Number of parameters
$m_{n,n}$	Parameters of the model connecting a_n, H and h in the n^{th} stage
$m_{n,n+1}$	Parameters of the model connecting a_n, H and h of the n^{th} stage and of next stage (n+1) stage.
n	Number of stages starting from stage 1 until the last stage N
N	Last stage (top stage)
Na^+	Sodium ions
p	Unknown system parameter
$\hat{p}$	Estimated parameters
$\hat{p}_i$	Estimated parameter at moment i

NOMENCLATURE
(Chapter Three)

$p(t)$	Unknown system parameter
p_0	initial estimate value of the vector of parameters
P_c	Vector of current estimate parameters p_{ic}
p_{ic}	Current residuals corresponding to the current estimates
$\hat{P}$	Estimated vector
Δ_{pi}	Differential corrections
ΔP	Residual estimate correction errors
$\nabla \hat{p}$	Necessary conditions for optimality for approximates
$\nabla_p J$	Sufficient conditions for optimality of estimates
Q	Symmetric definite matrix
$q_{H,f}$	Hydrogen concentration in resin phase out of the first stage
$q_{H,t}$	Hydrogen concentration in resin phase at the top stage
$q_{n,H}$	Concentration of hydrogen ions in resin phase in the n^{th} stage
$q_{n,Na}$	Concentration of sodium ions in resin phase in the n^{th} stage
$q_{n,Na}$	Concentration of sodium ions in resin phase in the n^{th} stage
$q_{Na,f}$	Sodium concentration in resin phase flowing out of stage 1
$q_{Na,t}$	Sodium concentration in resin phase in the top stage
Superscript ^{T}	Transpose of a matrix
t	Moment of time
T	Period of time (cycle time)
t_i	Moment of time t for each parameter determination
t_j	Moment j of time
T^s	Periodic time operating at steady state
Δt	Sampling interval specified by the user
$u(t)$	Input signal
V	Measurement error vector

NOMENCLATURE

(Chapter Three)

$v(t)$	Noise measurement (state vector plus noise)
W	Symmetric, positive definite vector
$w(t)$	Vector of external disturbance (noise)
w_{je}	Weighting errors
x_i^*	Composition of i^{th} component in phase x
x_j^*	Composition of j^{th} component in phase x
x^*	Composition of phase x
x_f	Resing ionic fraction leaving the first stage
x_n	Resin composition on the n^{th} stage
$x_{n,H}$	Mole fraction of hydrogen ions in resin phase in the n^{th} stage
$x_{n,Na}$	Mole fraction of sodium ions in resin phase in the n^{th} stage
x_t	Resin ionic fraction at the top stage
Y	Matrix of input functions at different time moments
$y(k)$	State space vector in discrete moments of time
$y(t)$	State space vector
y^*	Composition of phase y
y_0	Initial values of state values
y_f	Liquid feed ionic fraction entering the first stage of the column
y_f	Input disturbance vector
y_k	Value of $y(t)$ at a discrete moment (k) Δt
y_{k+1}	Value of $y(t)$ at a discrete moment $(k+1)\Delta t$
y_{k-1}	Value of $y(t)$ at a discrete moment $(k-1)\Delta t$
y_n	Liquid composition on the n^{th} stage
$y_{n,H}$	Mole fraction of hydrogen ions in liquid phase in the n^{th} stage
$y_{n,Na}$	Mole fraction of sodium ions in liquid phase in the n^{th} stage
y_t	Liquid ionic fraction at the top stage

NOMENCLATURE

(Chapter Three)

y_i^*	Composition of i^{th} component in phase y
y_j^*	Composition of j^{th} component in phase y
$\hat{y}(t_k)$	Estimate of $y(t_k)$ calculated from the model
$\dot{y}(t)$	State vector determining the model of the process
$z(t)$	State variables
$\tilde{z}(t)$	Measured output variables
$\tilde{Z}$	Observed measured vector
ΔZ	Correction vector
ΔZ_c	Current residual of correction vector
Δz_{ep}	Error corrections
ΔZ_i	Current residual of estimate
ΔZ_{jc}	Current residuals corresponding to the current p-estimates
ΔZ_{jp}	Linearly predicted residuals after correction from Taylor series
Δz_{jp}	Value of linearly predicted residuals after correction
λ^*	Matrix function of sensitivity
$\dot{\lambda}^*(t)$	Matrix function of sensitivity output vector
$(\lambda^*)^T$	Transpose of matrix function of sensitivity output vector
μ_j^I	Chemical potential of the j^{th} component in phase I
μ_j^{II}	Chemical potential of the j^{th} component in phase II
α_{ij}^*	Relative volatility of component i to component j
α_H^{Na}	Separation factor connecting molar fraction of sodium
α	Step of gradient calculation
Δ	Value of deviation

NOMENCLATURE

(Chapter Three)

ε	Error for calculating estimated values
α^*	Relative volatility
δ_j	Measurement of variance

NOMENCLATURE
(Chapter Four)

α^j	Step of the gradient calculation procedure at iteration step j
λ_s	Vector of Lagrange multipliers
λ_s^j	Vector of Lagrange multipliers at iteration step j
α	Step of calculation procedure
μ	Penalty coefficient
φ	Smooth vector function in a neighbourhood of a set-point
β	Smooth vector function in a neighbourhood of a set-point
ε_λ	Small positive number for the conjugate variable
$\lambda(K)$	Conjugate variable of the final sampling moment
$\lambda(k)$	Conjugate variables
$\rho(k)$	Coordinating vector
$\nu(k)$	External reference signal for nonlinear control
$\nu(k)$	External reference signal for nonlinear control
$\mu(k)$	Penalty vector
$(K\text{-}1)$	Sampling interval before the final one
Δt	Sampling period
ε_v	Small number for end of calculation determination
A	Matrix of process model parameters
B	Matrix of parameters of the process model
B_1	Process model parameters matrix
C	Output parameter model matrix
$e_\lambda(k)$	Conjugate variable error
$e_{\lambda_s}^j$	Conjugate variable error at iteration step j
$e_{F_R}(k)$	Control variable error

NOMENCLATURE
(Chapter Four)

F_R^{sp}	Control set-point
F_R^s	Control vector in steady state
$F_R(k)$	Control vector of steady state control plus some error
$e_u(k)$	Error between optimal steady state control and the control to the process
$e_y(k)$	Error between the optimal steady state values and real process output
$e_y(k)$	Error of the state variable
$e_z(k)$	Error between the steady state output and the real output at moment k
$F_R(k)$	Control
$G(k)$	Symmetrical matrix
H	Matrix of the controller
I	Identity matrix
J	Minimum criterion
J_d	Criterion for dynamic model solution
J_s	Criterion for a steady state model solution
K	Number of steps in the sampling interval
k	Sampling moment
$K\Delta t$	Time required to reach steady state
L	Constant matrix given by desired (linear) dynamics
l	Index of coordination
L_a	Lagrange functional
L_s	Lagrange functional
M	Big number for stopping calculation procedure
$\overline{M}$	Nonlinear controller
$p(K)$	Conjugate variable at moment K
q	Current moment occurring at each noticeable disturbance
Q	Weighting variable

NOMENCLATURE
(Chapter Four)

Qi	Weight matrix
R	Positively defined weighting matrix
Ri	Weight matrix
S	Weight matrix
$Superscript\ ^{T}$	Transpose of a matrix
U	Nonlinear process model vector
$u(k)$	Control variables
V	Constant matrix
$v(k)$	Values of the state, control vectors and sampling interval
$v_{max}(k)$	Minimum values for the state, control vectors and for the sampling interval
$v_{min}(k)$	Minimum values for the state, control vectors and for the sampling interval
W	Positively defined weighting matrix
$y(0)$	Initial state
$y(K)$	Desired final state
$y(k)$	Variables of the state
$y(q)$	State variable at disturbance moment q
y_0	Initial state
y_f	Disturbance
$y_f(k)$	Disturbance vector
y_N	Output variable of the last stage N
$y_N(k)$	Output vector
$\bar{y}$	Desired steady state
y_q	Initial condition of the state, vector at moment q
y_q^s	Optimal solution at steady state at disturbance moment q

NOMENCLATURE

(Chapter Four)

y^s_{q-1}	Optimal solution at steady state at previous disturbance moment of q
y^s	Optimal solution in steady state
y^{sp}	Optimal solution setpoint
y^{sp}	State set-point
z	Output variable
$z(k)$	Output vector
$z(k)$	Output vector
z^s	Steady state output

NOMENCLATURE
(Chapter Six)

Δk_H^*	Average gradient of conductivity of hydrogen versus concentration curve
Δk_{Na}^*	Average gradient determined over two points in the K_H^+ and C_H^+ curve
(Δk_{Na}^*)	Gradient determined over two points in the K_H^+ and C_H^+ curve
$(\Delta k_H^*)i$	Gradient of number i determined over any two points in the K_H versus C_H curve.
b_{Na}	Experimental gradient for sodium used to determine concentration sodium
C_H	Solution concentration contributed by the H+ ions
Cl	Chlorine
C_{Na}	Solution concentration contributed sodium ions
C_{Na}	Concentration as determined by sodium
C_{pH}	Concentration as determined by pH
H	Hydrogen
H^+	Hydrogen ion
I*measured*	Measured current
Iprobe	Current output from Conductivity or pH probe
K_H	Solution conducivity contributed by the H+ ions
K_{Na}	Conductivity sodium in the solution
K_{Na}	Solution conductivity contributed by sodium ions
K_{NaCl}	Conductivity determined for overall salt (sodium chloride) concentration
K_{pH}	Conductivity as determined by conductivity
K_T	Total conductivity in solution
m_I	Current gradient determined by the ratio between current output from the probe and the real measured pH or Conductivity
m_{pH}	Gradient of the pH scale over the probe output current
m_V	Voltage gradient determined by the ratio between current output from the probe and the real measured pH or Conductivity

NOMENCLATURE

(Chapter Six)

Na	Sodium
Na^+	Sodium ion
$NaCl$	Sodium Chloride
pH	Acidity/alkalinity scale
$Vmeasured$	Measured voltage
x_i	x value of a difference between two points in the x-axis
y	Final measured concentration of the solution
y_i	y value of a difference between two points in the y-axis

TABLE OF CONTENTS

Chapter One

THE PROBLEM FOR COMPUTER ON-LINE OF ION EXCHANGE PROCESS

TABLE OF CONTENTS

Chapter Two

ION EXCHANGE AND FUNDAMENTAL PROPERTIES

TABLE OF CONTENTS

TABLE OF CONTENTS

TABLE OF CONTENTS

TABLE OF CONTENTS

TABLE OF CONTENTS

TABLE OF CONTENTS

Chapter Six

IMPLEMENTATION OF DATA ACQUISITION AND CONTROL ALGORITHMS AND PROGRAMS

TABLE OF CONTENTS

Chapter Seven

CONCLUSION

TABLE OF CONTENTS

APPENDICES

Chapter One

The Problem for Computer On-line Control of Ion Exchange Process

---

Chapter one deals with the identification of control requirements for the ion exchange process as used for desalination of water. Ion exchange control and disturbance variables are identified for optimal control strategy. The multilayer structure is proposed for solving the control strategy based on the global aim of the ion exchange process and is discussed. Latest developments in the control field are studied and discussed.

"A greater pitfall into which many young process engineers fall, particularly in recent years is to get so involved in the fancy computer control hardware that is now available that they loose sight of the process control objectives. Keep in mind your objectives:

- To come up with an effective control system." Willham L. Luyben (Luyben, 1990).

1.1 ION EXCHANGE PILOT PLANT FOR DESALINATION OF WATER

Water upgrading and recovery system can presently be divided into two major categories:

- Those that provide a desalination function and
- Those that provide a tertiary function.

Desalination of water for potable purpose varies between two extremes:

1) Situation where the only available water is extremely saline (e.g. sea water with total dissolved solids, TDS content of 35mg/l) and
2) Where water is abstracted from boreholes with TDS between 1mg/l and 10mg.

In general, plants for upgrading sea water are based on phase change process such as evaporation or freezing whilst those used for borehole water are membrane process, reverse osmosis (RO) or electro-dialysis and ion exchange. RO becomes more attractive as the salinity level increases and that under in certain cases, a combination of both the ion exchange and reverse osmosis is better. For upgrading low salinity borehole water usually only ion exchange and reverse osmosis are economical. Ion exchange suffers the disadvantage of requiring a chemical content input proportional to the salinity level.

The tertiary treatment of water includes both the unit operations of a conventional sewage plant and those associated with drinking water, pretreatment (flocculation, filtration, and chlorination). A certain degree of organic removal occurs when RO and ion exchange, desalination plants are operated. It has also been possible to use ion exchange and RO as a combined desalination process and tertiary treatment operation, this came about through the supposed development of RO membranes and ion exchange resins with the ability to desorb organics which they have removed from the water being treated. The Desal process has been identified as an economical tertiary treatment that uses a form of the weak electrolyte resin system. The modification in the process included a flocculation step which performed most of the organic removal.

The Problem for Computer On-line Control of Ion Exchange Process

---

The ion exchange process belongs to a more general class of unit operation which is sometimes called *percolation.* A percolation process is defined as any process in which a fluid is passed through a bed of material which has the capacity to alter the concentration of the fluid. This definition includes some classic unit operations such as ion exchange, adsorption, chromatography, drying, and washing. These operations are performed in order to obtain,

a) Purification of the diluents

b) Separation of products

c) Recovery of solutes

Ion exchange removes ions from aqueous phase by exchange with counter ions on the exchange medium (ion exchange resins). The exchange ions are cation, positively charged or anions, negatively charged between the contaminants (feed-water) and the exchange medium. Ion exchange materials may consist of resins made from *synthetic organic* material that contain ionic functional groups to which exchangeable ions are attached. They may also contain *inorganic* and *natural polymeric* material. Ion exchange can remove dissolved metals and radio nucleides from aqueous solutions. Other compounds that have been treated include nitrate, ammonia, nitrogen and silicates. Factors that may affect applicability and effectiveness of the process include oil and grease, suspended solids, pH of the influent (feed), oxidants and wastewater contents (which may require additional treatment and disposal).

Ion exchange process currently used for the removal of salts from the wastewater (desalination) employs a method that involves passing water through columns of cation-exchange and anion-exchange with resin beads in hydrogen for (H^+) and hydroxide (OH^-) forms respectively. After a period of use (exchange) the resin beads get saturated with exchanging ions and then it becomes necessary to regenerate the resin to their original

forms. A base solution is used for strong cation resins and an acid solution for weak anion resin (acid is for regeneration of basic resins and base is for regeneration of acidic resin).

The use of combined cation and anion unit has the following advantages:

- reduction of regenerant acid and base, reduction of residual salt concentration in the product water,
- reduction in the quantity of salts rejected down the drain as waste,
- doubling the time between columns regeneration and neutralization of the solutions rejected and
- neutralization of the solutions rejected down the drain by mixing the acid and the base streams (Dudas, 1995; Remco Engineering, 1998; PROJECT: vol. 1, 1982; Water Quality Association, 1995). The emphasis of this research work is on the reuse of domestic water supplies starting with a *secondary sewage effluent* as feed to the columns and producing water of potable quality. However the recovery and reuse of industrial effluents is a matter of equal importance and to which ion exchange is particularly suited, for a number of reasons.

From the previous investigation conducted at the University of Cape Town in 1980 – 1982, the aim of that work was to provide a desalination step for a major water recovery project for Cape Town (PROJECT, vol. 1, 1982). It soon became apparent that in addition to removing the salt from the water the ion exchange operation also removed the bulk of the organic material which constituted the *chemical oxygen demand* (COD) of water.

In removing the salts virtually all the phosphates, nitrates and ammonia were eliminated. The two major decisions in developing an ion exchange operation are the nature of the contacting device to be used and the type of regenerant chemicals to be used. Some sodium will leak from the cation exchange and this determines the purity of the product water being process. For the new ion exchange pilot plant built at Peninsula Technikon, Chemical Engineering Department the type of the contacting device used is a continuous

countercurrent ion exchange (CCIX) of four columns. This system has proven to be more efficient for desalination process as compared to other systems of contacting devices. The most appreciated advantage is the fact that it maximizes the regenerant contact efficiency which results in higher purification levels. The countercurrent system passes the resin in the opposite direction to that of the feed and this fully regenerates the bottom of the resin beads which minimizes the sodium leakage in the product. The regenerants used for the new plant are sulphuric acid and lime because of their easy handling capabilities in terms of waste disposal, relatively less expensive and are general purpose regenerants making them easily availability, this has been proven from the previous investigation. Ammonia and nitric acid as the alternative provides easiest regeneration system from operational point of view but also have the potential of giving the least disposal requirements but a need a fifth column which could prove uneconomical for this investigation.

Careful control operating conditions are necessary to ensure that for a given desalination level, maximum water output is balanced with minimum regenerant chemicals consumption. This can be achieved by designing an optimal control system with the aim of maximizing water output and minimizing regenerant chemicals used.

1.2 PROBLEM FOR OPTIMAL CONTROL OF ION EXCHANGE PROCESS

1.2.1 Ion Exchange Process as an Object of Control

Ion Exchange column as an object of control is characterized by the following variables

- Inputs:
  - * Resin flow rate (F_R) [mol/h]
  - * Liquid flow rate (F_L) [mol/h]
  - * Inlet resin concentration at the top (q_t) [mol/l]
  - * Inlet liquid concentration (C_f) [mol/l]
- Disturbances:
  - * Input liquid concentration (C_f) [mol/l]
- Outputs:
  - * Exit liquid concentration (C_t) [mol/l]
  - * Exit resin concentration (q_f) [mol/l]
- Controls:
  - * Fractional resin transfer (d) [fractional]
  - * Voidage (e_v) [fractional]
  - * Cycle Time (T) [h]
  - * Liquid feed rate (F_L)

Control requirements are that:

- Overall resin mass balance is maintained

$$F_R T = (1 - e_v) V_R d \tag{1.1}$$

or

$$F_R T = hd \tag{1.2}$$

where h – is resin holdup in each stage,

V_R – volume of resin moved from one stage to the other.

For the work covered the chosen variables are:

- Control action – cycle time, T,
- Measured output variable – exit liquid concentration, C_t,
- Main input disturbance – input liquid concentration, C_f and
- State Space Variables – Sodium (Na) concentration in every stage.

The values of the voidage, fractional resin transfer, liquid flow rate, inlet resin concentration are constant and are determined by the column design.

Difficulties in control of columns include such factors as

1) Columns with many stages have slow responses, often with large time delays.
2) The process of ion exchange is affected by many variables.
3) Control loops.
4) On-line measurement of the stage concentration is not always feasible.
5) The process is influenced by the input flow concentration.

The control strategy is based on the following:

- The goal of the process
  - * to obtain high purity of the product water
- The type of the process
  - * slow,
  - * with time delays,
  - * nonlinear dependence (between the control and the state) and
  - * interaction between stages.
- The technology of process variable measurement
  - * pH and conductivity measuring devices (sensors)

Control of separation systems is probably the greatest challenge faced by the control system designer. These processes are nonlinear and consists of many variables, feed and product flows, compositions and energy inventories, all of which interact (PROJECT: vol. 1 & vol. 4).

1.2.2 Optimal Control Problem Formulation in Economic Terms

Based on the requirements for the plant operation in economic terms the following problem for optimal control may be formulated:

- To maintain the operating conditions such that the required quality of water is produced at constant throughout for the minimum consumption of regenerant chemicals.

From the results of the proposed control strategy, the following advantages are expected:

- reduction of acid and base excesses required for regeneration
- reduction of residual salt concentration in the product water.

In addition to the treatment of domestic effluents, there are other applications which the ion exchange process is particularly well suited for, such as the recovery and reuse of industrial effluents. The proposed control system is developed in such a way that it caters for any desalination ion exchange process, it does not have to depend on certain kind of effluent.

1.2.3 Disadvantages of the Old Control Strategy

The pilot plant for the ion exchange for treatment of sewage effluents was built in 1980 in the Department of Chemical Engineering, University of Cape Town (PROJECT: 1982). The microprocessor system was developed to control the above process. The objectives of the microcontroller based control system of the previous system was:

- To provide a microcontroller based control system that would automate the flow of resin and feed water and
- Also to provide regulation of regenerants for every efficient system that could limit most of manual operation.
- The following aims were than formulated based on the above objectives:

- To develop a new or evaluate an existing ion exchange process to provide a simultaneous desalination and tertiary treatment of saline second sewage effluent.
- To test the process in a laboratory and in a pilot plant scale equipment for technical feasibility.
- To develop and test design criteria in order that a large scale plant may be designed and costed (PROJECT: vol. 1, 1982).

This system does not exist anymore. The problem for waste water treatment has become so important that a new pilot plant has been built in the Department of Chemical Engineering at Peninsula Technikon. A new control system has been designed and implemented based on a personal computer (PC) as a controlling platform.

The ion exchange process and the control system that was developed in 1980 had some disadvantages according to their proper and optimal work:

- As the up-flow cycle times are determined separately for each column on the basis of the feed concentration, resin capacity and the required stoichiometric ratio, the cycles come in and out of phase.
- The starting time of the columns is very long. The time taken from the process to reach a new steady state when the feed water changes its concentration is also long.
- The used model considers only steady-state operating conditions.
- The closed loop controls are not built into the previous system.

1.2.4 Necessity of the New Control Strategy Based on a Personal Computer

The control system for the ion exchange process as developed in 1982 (PROJECT: vol. 4, 1982) was based on the old microcomputer system. It is therefore necessary to develop and improve its ideas on the bases of a personal computer technique. Then the obtained results could be applicable for solving the wastewater problem in industry according to the present level of computing technique and knowledge.

The use of new computer techniques is connected to a deeper understanding of the ion exchange process, the proposal of new models and methods for its description and control. The new computer based control system gives solutions to the above disadvantages by providing the following:

- Better measurement techniques based on used data acquisition system.
- Improved calculations since they are done by the same software package as used for data acquisition, thus minimizing calculation time.
- Calculation and realization of the optimal control.
- New packages can be implemented with the current system since it is run from a personal computer. Software packages such as for data acquisition, optimization and control implementation can be used at any stage should changes be a necessity.
- Reliable and not expensive hardware for measurements and signal conditioning.

This creates a very robust and flexible system that can accommodate most computer based control packages, which also solves all the disadvantages of the previous microcontroller based control system.

Another benefit of the computer-based system is that in addition to the timing, complex control valves interlocking based on optimal control conditions is achieved. The control valves mechanism is performed by a timed sequence from a computer – which uses calculated optimal values based on pH and conductivity measurements acquired from the feed concentration.

1.3 COMPUTER OPTIMIZATION AND CONTROL OF INDUSTRIAL PROCESSES

1.3.1 Bridging Between Business Objectives and Control

Computer optimal control of the chemical processes offers several advantages over conventional control. Primarily, it brings the gap between a plant's true business objectives and its actual operation (Latour, 1979). The optimal computer control of these processes is needed due to:

- a lack of enforcement of action,
- a lack of accurate information,
- the time lag involved in making changes,
- the advent of less expensive and increasingly reliable equipment.

Process control instrumentation and computers are used to measure the state of the plant and regulate the operating conditions of specified targets. The operating objectives of industrial plants can be summarized in a global way in terms of three factors *safety*, *product quality* and *efficiency*.

- Safety. Do not operate the process in way which might harm people or damage equipment.
- Product quality. Operate the plants such that product quality, specifications and production rate targets (or inventories) are achieved.
- Efficiency. Operate process economically, to minimize long term operating costs.

There are numerical specifications associated with each of these objectives. For safety they come from the equipment manufacturer or past experience. For product quality, they come from specifications in contracts or from the market customers. Optimum operating efficiency includes utility costs, product values and maintenance costs.

Most process control systems indirectly contribute to the operating objects above by logging, alarming and regulatory control of objecting conditions. The control systems provide the interface for translating the mentioned general operating objectives into the specific command inputs to the process control system of the plant. They bridge the gap between business and the plant operation. It seems apparent that genuine progress can be measured by the success of bridging this business or control gap.

The dissertation proposes online computer control strategy for a separation process of ion exchange as a technique for bridging the business or control gap, in the field of separation processes.

1.3.2 Types of Optimization

The concept of optimization is very broad and complex and it has different meanings for people in different settings. There is difference between the two general control methods of optimization – dynamic regulation (steady state stabilization) and steady state optimization. The functions and objectives of each type of control are quite different. Also, the techniques used to implement these control methods are different.

Dynamic optimization can be divided into two parts:

- dynamic regulation and
- dynamic optimization control

Dynamic regulation uses the values of the control variables (also called manipulated variables) to hold the controlled variables near specified previously target values, which can be fixed or time varying. This control function is achieved by feedback control, adaptive control, nonlinear control, and modern optimal control. The optimization is often used in the context of dynamic optimization. The process is described by differential equations and the objective function is an integral over time. The results from optimization are the parameters of the controller which will give the best dynamic

regulation. The criterion usually connects the values of the desired and controlled variables and express indirect way the business objectives. Dynamic optimal control is considered when the trajectory of the controlled variables has to be optimal in some sense. Different problems for time, energy and deviation optimization can be solved. One of the problems very important in process control is this for the startup of the plants. This problem can be solved as a problem for minimum time control. It is necessary for energy and time minimization. This problem will be considered and solved in the dissertation for ion exchange process startup time.

Steady state optimization is applied to processes and objectives which are described by algebraic equations rather than differential equations. There are three different types of optimization problems that are commonly encountered in industrial process operations:

- operation conditions,
- allocation and
- scheduling.

Optimizing steady state operating conditions include process conditions. Allocating problems have to do with the best distribution of a fixed resource among several pieces of equipment. Scheduling problems refer to the proper timing or frequency of repeating a periodic operation. The steady state optimization determines the set-points for the dynamic regulation control system.

The problems for dynamic optimization and steady state optimization of the operating conditions will be considered in the dissertation in their connection in order to achieve one global aim for process control. The scheduling problem will be solved also on the basis of the obtained solutions of the above problems (Junkins, 1991; Kilkas & Hutchison, 1980; Pierre, 1969).

1.3.3 Optimization Techniques

There are a wide variety of mathematical techniques for optimization employed for steady state and dynamic computer design and process control. Very frequently used for steady state optimization are the techniques of linear programming, gradients, simplex evolutionary operation, Lagrange multipliers.

Very frequently used methods to solve dynamic optimization methods are these of Lagrange functionals, Pontriyagin maximum principle, dynamic programming and Green's theorem (Kirk, 1970).

The method of Lagrange functional is used in the dissertation because of its big capability to handle large scale, linear and nonlinear problems with different constraints.

1.3.4 Requirements for Online Optimization

There are a number of considerations in the design and implementation of successful online system for computer control and optimization of industrial processes. The very requirements are (Latour, 1979):

- Select proper control variables.
- Formulate model requirements.
- Formulate business objectives functions.
- Select or develop optimization method and algorithm (appropriate to the nature of the problem). Always to guarantee a feasible solution.
- Specify sensor inputs, manual inputs.
- Define human interface for objectives, defaults, economic parameters, physical limits, laboratory analyses. The system must inform people what it is doing and why.
- Specify the interface with regulatory controls, consider timing, move limits, output sequencing, process dynamics, stability and interactions.

- Specify computer hardware and software.
- Consider procedures to maintain model fidelity.
- Specify the type and measurability of disturbances.

Very important in development of the online control system is to consider steady state, dynamic and disturbance behaviour of the process in connection and dependence. This dependence leads to a special kind of structure of optimization that can be used. The multilayer structure which can be implemented by a computer and realize optimizing adaptive control allow to achieve all objective of the process control. They will be used as a basis for development of hardware, design methods for control, algorithms and software.

Another important consideration is that the control system has to work in collaboration and to depend on the operator. It has to allow full automatic control, automatic control with the operator, or fully manual control. This requirement is followed in the dissertation during development of all algorithms and programs.

1.4 PROCESS CONTROL IN SEPARATION PROCESSES

The role of process control in separation processes has expanded during the years with introduction of computers. There are different directions of applications of different methods and strategies of control.

In most instances the standard PID (proportional-integral-derivative) controller is still used for the control of single-input single-output (SISO) systems. However, individual control loops may be placed in the hierarchy of a distributed control system. By doing so, the control of many single-input single-output (SISO) systems can be orchestrated better, and several more sophisticated control methodologies can be considered including cascade control and split-range control. Distributed control also provides a framework for considering multiple-input multiple-output (MIMO) systems and the interactions that may be inherent in these situations.

Most of the more recent development in control are linked to improvements in computing at all levels. Digital computers have been developed to the point where the element of the process can be treated more realistically as the complex highly nonlinear, and multi-interactive systems that they are. This has led to a more sophisticated use of dynamic modelling and simulation in process control. As computing costs have come down and computing capabilities have expanded, control strategy is being developed in a more global sense using simulation to consider the interaction of the process elements and sometime different processes. As a result the focus in process control has expanded from the tuning and performance of controllers for individual unit operations to the broader context of controlling and coordinating the operation of an entire plant.

A distributed control system may involve the use of microcomputers at the local level and the use of more powerful computers to coordinate overall plant control objectives.

The advantage to distributed control is that it will make effective use of current technology and provides a framework within which control and optimization developments can be implemented. These developments include better simulation and

optimization routines that help to assess the current state of the process plant and to suggest improvements (Buckely, 1964; Fisher, Doherty & Douglas, 1984; Higgin, 1984; Fisher & Mankat, 1974).

1.4.1 The Role of Process Modelling and Simulation in Process Control

In the perspective of process synthesis, process control should be viewed not as a separate element in process design and optimization but rather as a component of a coordinated approach. Therefore, the design and sequencing of ion exchange processes must consider the relationship that process will have to the final process structure. This can be done only through process modelling and simulation.

The most important aspect in the control of the process is understanding of the process itself. Previously, without the availability of digital computers, overdesign and the neglect of the process dynamics were practical necessities. Today, however, through process modelling and simulation with computers a better understanding of the process is possible before any hardware is fabricated (Franks, 1972; Holland & Liapsis, 1983; Morbidella, Sewida, Storti, & Carras, 1982). From a control standpoint this enables the process engineer to investigate the characteristics of the system over a wide range operating conditions and to obtain a better parametric understanding of the process. This, of course provides better basis for selecting appropriate control strategies.

With the continued increase in sophistication of process modelling and simulation, it is becoming apparent that the control of chemical processes often requires a unique application of the foundations of control theory.

Early chemical engineers often looked deeper and deeper into sophisticated control theory for answers to problems that arose in process control. It now seems, after useful experience with process modelling and simulation, that less-sophisticated applications of control theory often are adequate for even complex problems. The key to successful

process control has been found in an increased understanding of the process rather than sophisticated application of control theory.

Thus, process modelling and simulation has become a key element in process control. Complex computer programs for these purposes have evolved and become well accepted tools for chemical engineers. These simulation packages can be used to design new processes as well as to modify and optimize existing ones. Usually they are comprised of sophisticated physiochemical estimation routines along with process models for a variety of unit operations, reactors and auxiliary equipment. The utility and accuracy of these simulation programs are a function of how well conceived the models of the components of the presses are as well as how effective the necessary convergence techniques are implemented (Rosen, 1980).

The first process simulators developed for general use were for steady state operation. Process dynamics usually were ignored to avoid excessive computation times and computation difficulties. While steady-state information is useful for control strategy evaluation, process control systems are designed best with some knowledge of the process dynamics. As nonlinearities and interactions become more pronounced, information from the time dependent behaviour of a process becomes crucial. There is currently a great deal of effort focused on upgrading process simulators so that they can handle unsteady state operation.

Because of the computational complexities associated with dynamic process simulation for multi-unit process, there is still much to be done before simulators of this type become available for general application. Another problem complicating their development is that the process models for even individual separation units are usually built for steady-state cases. It is the result of both the incomplete understanding of the chemical and physical principles involved and computational difficulties. This is one of the main reasons why process control considerations are difficult to incorporate into chemical process simulation and synthesis and why on-line optimization is still far away in most instances.

Process modelling and simulation are nevertheless extremely important tools in the design evaluation of process control strategies for separation processes. There is a strong need, however, for better process models for a variety of separations as well as process data with which to confirm these models. Confidence in complex process models, especially these that can be used to study process dynamics, can come only from experimental verification of the models. This requires more sophisticated process sensors these commonly used for temperature, pressure, pH and differential pressure. Direct, reliable measurement of stream composition, viscosity, turbidity, conductivity and so on is important not only for process model verification but also for actual control applications (IDA, 1997; Lior, 1995; Lovland, 1968; Luyben, 1990; 1973; Johnson, 1993).

There are still many obstacles to overcome before the use of the process modelling, simulation and control reaches the potential that many think it holds. One of the most interesting possibilities is the development of system capable of performing on-line optimization functions. Although on-line plant optimization is still in future, optimization of sub-processes is already a possibility. Process modelling and simulation enabled new process control strategy to be developed (Haskins, 1983; Roussea, 1995).

Dynamic operation becomes an important aspect to be considered in the synthesis of separation processes. For some continuously operated columns their feed as well as product may be frequently changed from one to another. Moreover, startup and shutdown become usual operations in industrial practices. These operations necessitate dynamic policies, with which an objective (e.g. minimum time, maximum productivity or maximum profit) can be gained (Li, Flender, Wonzy & Fieg, 1999; Fieg, Wanzy & Kruse, 1993; Serchsen, Machietto, Stuart & Skogestad, 1996; Vassiliadis, Pantelides & Sargent, 1994; Krivosheev & Torgshov, 1999).

To optimize the dynamic operation of such processes, a large scale optimization problem has to be formulated. Thus an efficient solution approach is required. The

decomposition methods are very effective in solving such type of problems (Vassiliadis et al, 1994). These methods are used in the considered dissertation.

Although there have been quite a few studies on dynamic optimization of separation processes (especially in distillation) no reports on implementation and verification of the developed policies have been found. The reliability of the developed policies depends on the model describing the process. It is desired to use rigorous models for a higher accuracy. But the optimization problem becomes complicated. A compromise between the model accuracy and the solution capability has to be taken in the model selection.

In Krivosheev and Torgashov (1999) economical criterion is used to achieve dynamic optimal control. The optimal control problem is decomposed into two sub-problems:

- Steady state and
- Dynamic optimization

The aim of the first sub-problem is a steady state optimization. The aim of the second is the shift of the plant into optimal steady state over minimum time interval of time.

1.4.2 Process Synthesis of Control Structures

The objective in the control of the ion exchange process has evolved from the control of components of a process that already has been designated to an integrated plant involving simulation, design and control.

There have been several attempts at control structure development within process synthesis methodology. Govind and Powers (1982) started with steady-state flow sheets and used cause-and-effect diagrams to represent control logic for a particular process. Dynamics were modelled by a first order differential equation with time delay in series.

Morari, Arkun & Stephanopoulos (1980), (Morari and Stephanopoulous, 1980a, 1980b) describe a procedure for control structure development based on the multi-layer multi-echelon approach of hierarchical control theory, which is free of heuristics. The key to

the procedure is the effective application of decomposition to produce manageable subsystems of the problem. Examples are provided to show how structure controllability and observability can lead to a control system that is consistent with processing objectives (Morari, 1980; Stephanopolous, 1984, 1982).

1.4.2.1 Operating Objectives

The starting point in development of the control structure for some process is a qualitative formulation of the control objectives. Many will be determined by the specific nature of the process involved. General rules can not, nor need be given. In the first category of control objectives are those related to the operational feasibility. They are always function of process variables, which are to be kept within certain specified bounds, in spite of uncontrolled influences on the process. The origin of these requirements may be product quality specifications, safety considerations, operational requirements, environmental regulations etc.

The second category is derived from economic considerations. These enter only if, after satisfying the first class of objectives, manipulated variables are left to adapt the operating conditions to stay at the most profitable point of operation.

In attempting to design a feedback optimizing control structure, the aim is to translate the economic objectives into process control objectives. In the considered case the operating conditions is necessary to be kept such that the maximum production of water with desired quality (low level of salt concentration) is produced with minimum use of chemicals for regeneration. This objective can be transformed into a mathematical quadratic criterion in which the desired level of salt in product water appears as a set-point.

The objectives of the considered investigation are:

1) To improve the consistency of control applied to ion exchange process and thereby improve performance of the plant.
2) To develop methods and programs for real time implementation of the control.

The questions which have to be considered at the beginning of development of design methods and control implementation programs, are:

1) Define and prioritize operating objectives,
2) Identify disturbances variables,
3) Select basic column controls,
4) Locate composition (concentration) sensitive control point.

Operating objectives normally consist of composition specification. The following additional information is needed:

- Product composition or concentration is most important to maintain in the event of disturbances,
- Composition variation acceptable for each product or some limits for the product.

1.4.2.2 Disturbances Variables

The disturbances expected to enter the process during operation have to be defined and their impact on the performance evaluated. Only then the need for a certain controller can be established. This makes process control different from control in other disciplines where the disturbances can generally only be described in a noise-like manner.

The purpose of the ion exchange control system is to minimize the effect of disturbance variables on product concentration. A host of disturbances including changes in

- feed rate,
- feed concentration,
- environmental conditions,

may be encountered in the plant. Two approaches can be followed in minimizing consequences of expected disturbances:

- Firstly, design the ion exchange column control so that column operation is insensitive to the disturbances, and
- Secondly, eliminate the disturbance by providing secondary controls for this purpose.

In the design phase, definition of expected disturbances – including magnitude and frequency of occurrence – is necessary to design a control system that will assure successful column operation. This information provides justification for addition of stabilizing equipment and controls and is necessary to evaluate alternative control strategies during design phase.

Secondary disturbances can be eliminated through selection of special-purpose control equipment. For the ion exchange column such type of disturbance is the feed rate. It is kept constant by using flow-meters.

Feed concentration is the major disturbance for which the ion exchange control must be designed. The control strategy is based on the frequency and magnitude of the disturbance. The frequency determines the type of the control structure and the magnitude determines the magnitude of the control action.

Disturbances can be classified according to their frequency and in this way according to their influence over the different problems for control of the process. That is why classifying the disturbance is one of the first steps in the development of the control structure.

The considered main disturbance for the ion exchange process – the concentration of salt into the feed water is with low frequency. It appears at moments when the source of the salt water is changed. This disturbance will influence the model parameters and the optimal steady state values of the process variables.

1.4.2.3 Basic Column Control

At this step the control variables have to be selected on the basis of collection of data for the operating conditions near the design point and calculation of relative changes in one variable when small step change in another is provided.

There are two possible control variables for the ion exchange column:

- the feed rate and
- the up-flow time of the feed water.

The first variable is not very convenient to be used because the changes in flow rate will lead to changes of the environmental conditions of the ion exchange process between the resin and water. And also the solution (water) and the resin will not be uniformly mixed. The change of the up-flow time period is convenient because it overcomes the above difficulties and the time necessary for achieving equilibrium conditions for every stage can be controlled.

The experiments with the columns of the same type showed (PROJECT: vol. 3, 1982) that better results according to the level of final concentration of NaCl into the water were obtained when the up-flow time is used as a control variable.

1.4.2.4 Location of Concentration Sensitive Monitoring Points

The main measurable variables in the ion exchange column are the concentration of the input and output flows of liquid. The information from measurement of the concentration is used to:

1) monitor the concentration of NaCl at some points in the column,
2) to estimate the model parameters in real time,
3) to optimize the process and to minimize the effect of the changes in the feed concentration.

Two sensors are used to measure and calculate the concentration of NaCl in the water. They are for measurement of pH and conductivity. The sensors are located in the input flow and in the output, product flow of the cation loading column (Fig. 2.1).

All considerations for the steps necessary to be considered before design of the control structure lead to the following algorithm for selecting of control strategy (Morari, et al, 1980).

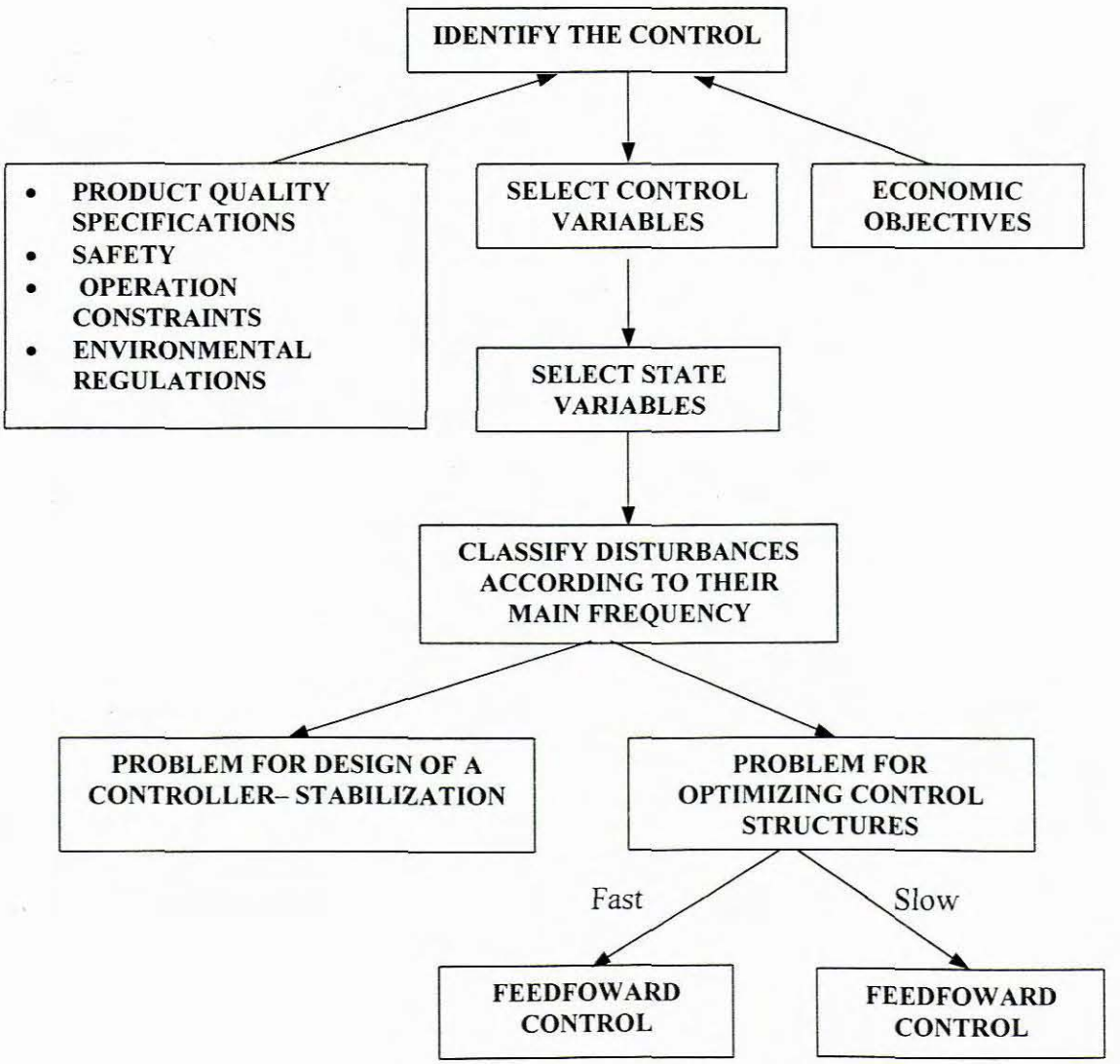


Figure 1.1. Steps to be Considered for Designing a Control Structure.

1.5 THE PROPOSED CONTROL STRATEGY

1.5.1 Synthesis and Implementation Process Control Structure within the Framework of Hierarchical Control

The optimal control strategy is formulated based on some disadvantages of the previous system and these disadvantages are mentioned. The strategy is implemented in the frameworks of the hierarchical control structures.

The theoretical development of the theory of hierarchical control system started with the books of Mesarovic (1870). These structures can be considered in two groups according to the way of decomposition of the common control problem, as

- multilayer structure, described by Lefkowitz (1966).
- multilevel structures, described by Mesarovic (1970).

The concept of multilayer, multilevel framework of control is found to be very meaningful, convenient, and having the potential for further development (Morari et al, 1980) in the field of chemical processes. Decomposition is an underlying, guiding principle, leading to the classification of the control objectives, adaptation, optimization, regulation, and the partitioning of the process for the practical implementation of the control structures.

1.5.2 The Proposed Control Strategy in Multilayer Structure. Problems Solved to Realize the Strategy

To achieve optimal control a person computer based system is used, whereby the column cycle times and regenerant feed rates are automatically optimized on the basis of salinity as determined by measurements of pH and conductivity at different points on the plant. These measurements are then processed and used for the calculation of the sodium (Na) concentration at the streams of interests. Based on the calculated of the up-flow time as a control variable, control actions are activated to achieve the calculated up-flow time. The calculation of the up-flow time is performed by the computer system, which is also responsible for achieving optimal control of the process.

The computer control also performs the online control function with a comprehensive monitoring and record keeping for the plant. Computer based monitoring and record keeping eliminates a need for a highly skilled operator.

The proposed control strategy consists of:

- *minimum startup time for the process to reach steady state,*
- feed-forward control to compensate changes in the feed salt concentration, and
- closed loop control to regulate the outputs of the columns due to small changes in the concentration of feed water (disturbances) and accurate timing of the control mechanism.
- Tuning sequence control of the flows in the process to combine all optimal implementations.

The start-up problem is formulated on the basis of criterion for minimum time for starting of the process from the given initial state to the desired one, and on the basis of derived mathematical model and constraints imposed by some technological requirements. This problem is characterized by non-quadratic criterion and linear control, which leads to difficulties in solving the two point boundary value problem.

The steady-state optimization problem is formulated on the basis criterion minimizing the norm of state and control errors between their nominal values and current values and on the basis of the steady-state version of the developed mathematical dynamic model. The problem is solved by a method of Lagrange's functional. The obtained solutions are the new set points for the closed loop control.

The dynamic problem for closed loop control synthesis on the basis of quadratic criterion of quality minimizes the norms of the errors between the new steady states (set-point) and the current values of states and controls and on the basis of the developed mathematical dynamic model. The problems are solved using methods for control synthesis from classic and modern control theory. Nonlinear controller is designed first to linearize the nonlinear model and linear optimal control is then solved to minimize the influence of the parameters' uncertainties.

The sequence control of the flows is based on the balance of resin in the process. It realizes the minimum time, steady state and dynamic controls switching on or off different solenoid valves in the frameworks of the process technology requirements.

1.6 AIM AND OBJECTIVES OF THE RESEARCH WORK

According to goals, the main purpose of the research project is to develop a strategy for optimal control and methods for identification and control synthesis and to use them for simulation and real time implementation with a new pilot plant. The proposed control strategy consists of:

- minimum time control to start up the process,
- feed forward control to compensate changes in the feed salt concentration,
- closed loop control to regulate the outputs of the columns according to small changes in feed water and
- accurate timing of the control mechanism to balance the resin flows in the process.

1.6.1 The Global Aim of the Process Optimal Control

The main aim of the ion exchange process control is to keep the desired quality of the product (purified water) in required throughput under minimum consumption of the regenerants. Technically this aim can be formulated using the concentration of the salt in the product water and the flow rate of the resin into the column. The salt concentration in the product stream can be used as the reference signal for the criterion expression which will be used as a base for control design. Flow rate of the resin inside the column is very important for the resin balance in the process. The final control system is designed according to the following:

The global aim is reached by the solution the achievement of the following objectives:

1. Development of the hardware structure for control of the process using a personal computer (PC) and signal conditioning and measurement instrumentation.
2. Development of data acquisition system for measurement of the important process variables important for the control purposes.
3. Development of the multilayer strategy for optimal control of the process.
4. Development of the method and algorithm and program for modeling and parameter estimation of the process dynamics.
5. Development of method and algorithm for minimum time problem for solving startup time optimal problem.
6. Development of method, algorithm and program for optimal control of the process in the presence of slowly varying disturbances.
7. Development of the sequence and program for flow control of the process.

1.7 CONCLUSION

Water upgrading and recovery system can be presently divided into two major categories those with desalination function and those with tertiary function. Ion exchange process has proved to be one of the convenient and attractive methods in desalination of water. The ion exchange process involves passing water through columns of cation exchange and anion exchange. The emphasis of the work herein is on the reuse of domestic water supply.

Analysis of the ion exchange process as an object of control is done. Attention is paid on the appropriate selection of the input, state space, output and control variables in order to realize the process technology and aims.

Latest developments in process control methods for design and real time implementation of the separation processes are proposed to be used for the considered ion exchange process. The applied control strategy aim to overcome some of the drawbacks of the previously built in the University of Cape Town control system and is based on application of multi-layer approach through which the system is optimized according to some criterion and the real control is realized by a computer. The on-line (optimization) control considers minimum time control, steady state and dynamic optimal control in the presence of disturbances.

The project aim and objectives are described at the end of the chapter.

Chapter Two

Ion Exchange Process and Fundamental Principles

In Chapter Two the ion exchange process is described including the operation and the physical structure of the pilot plant as built in Chemical Engineering Department at Peninsula Technikon. Process elements and their functionality are defined.

Fundamental principles behind the exchange of ions in the process equilibrium, kinetics and mass transfer relationships are also discussed.

2.1 ION EXCHANGE HISTORICAL BACKGROUND

There are suggestions found in the Bible and in writings by the ancient Greeks, that suggested knowledge of desalting blackish waters. The discovery of ion exchange dates back from the middle of the nineteenth century. The first official study happened in the nineteenth century in England where the studies of the ion exchange were documented.
In 1850 Harry Thompson and John Way, two agricultural chemists passed a liquid fertilizer through a soil sample. It was noted that the ammonia was retained by the soil and that calcium was thrown off. In their report to the Royal Agricultural Society they documented a number of important observations that form a foundation of the understanding of the ion exchange process (Keller, 1992; PROJECT: vol. 2, 1982; Water Quality Association, 1995; Sybron Chemicals Inc. 1992).

- The exchange of ions in soils involved the exchange of equivalent ions.
- Some ions were more easily changed than the others
- The aluminum silicates present in the soil gave it the exchange characteristics.
- The exchange of ions was different from true physical adsorption

The German chemist Eichorn in 1858 proved that the ion exchange reaction in soil was a reversible one. Lemberg in 1876 also confirmed the reversibility and calculated the stoichiometry of the process (Ullman's Encyclopedia, 1989).
The beginning of the 20^{th} century marked the era where the ion exchange was implemented for the practical purposes. In 1905 another German chemist Gans, developed a process of softening water on a commercial basis using cation exchange material. He traded magnesium and calcium ions for sodium ions in water. Gans called his cation exchangers *zeolites*. The term zeolite was first used by the Swedish geologist, Cronsteldt for naturally occurring silicateous minerals which became dehydrated when heated. The word zeolite is derived from the Greek words *zein* and *ethos* which literally mean the "*boilingrock*". The first synthetic resins (manufacturing as opposed to natural occurring) were manufactured in America by the Permutit Company in 1913.

Sulphonated coal referred to as carbonaceous zeolites was used as ion exchange material in 1935. That same year the two English chemists Adams and Holmes produce two new ion exchangers. The cation exchanger was a phenol-formaldehyde condensation product and the anion exchanger was a condensed product of polyamines and formaldehyde. These products made possible near complete removal of all ions from water.

In 1944 D'Alelio helped to develop cation exchange resins based on the copolymerization of styrene divinylbenzene. In 1946 McBurney produced polystyrene anion exchange resins by chloromethylation and amination of the matrix. The anion exchangers known till then were weakly basic and took up only strong mineral acids. The new resins produced by McBurney were stronger bases and could absorb weak acids such as carbon dioxide or silica. This process allowed complete demineralization of water with purity previously obtained only by multiple distillation in platinum. Two problems remained at the time, the fouling of resins by natural organic acids present in surface waters and mechanical stress imposed by plants operating at high flow rates. The ion exchange resins were then manufactured with high degree of crosslinking and artificial pores in the form of channels to absorb large molecules. Anion exchange resins based on the styrene-divinylbenzene copolymer were developed in 1948. These resins completely demineriliized water when run in the hydrogen-hydroxide form respectively. Many adaptations of the original products have been developed over years. Between 1970 and 1972 a new type of anion exchange resin known as polyacrylic anion exchangers, because of the polycrylic matrix structure appeared on the market. This type of resins processes exceptional resistance to organic fouling and very high mechanical stability due to elasticity of the polymer (Keller, 1992; Kirk-Othmer Encyclopedia, 1995; Sybron Chemicals Inc., 1992; PROJECT: vol. 2, 1982; Ullman's Encyclopedia, 1989; Water Quality Association, 1995).

2.2 ION EXCHANGE PROCESS

Ion exchange forms the basis of a large number of chemical processes, which can be divided into three main categories, substitution, separation and removal of ions. In substitution, a valuable ion (e.g. copper) can be recovered from solution and be replaced by a valueless one. This process can also be used for removal of toxic ion from a solution and be replaced by a non-toxic. In separation, a solution containing a number of different ions is passed through a column containing beads of exchanging ions resin resulting in ions being separated and emerging in order of their increasing affinity for the resin. Removal of ions is achieved by using a combination of a cation resin in the hydrogen ion (H^+) form and an anion resin in its free base form resulting in a demineralized solution (Streat & Cloete, 1995).

Natural water supplies contain dissolved salts which dissociates in water to form charged particle called ions. These ions are usually in relatively low concentrations and permit the water to conduct electricity. These ions are sometime referred to as electrolytes. The ionic impurities can lead to problems in cooling and heating systems, steam generators and manufacturing. Common ions encountered in most waters include the positively charged, cations calcium (Ca^{++}), magnesium (Mg^{++}), sodium (Na^+) and negatively charged, anions including, alkalinity, sulphates (SO_4^{--}), chloride (Cl^-) and silica (Si^{--}) (Streat, 1976). Ion exchange resins are well suited for removal of these ionic impurities for several reasons: (McGovan, 1988; Water Quality Associate, 1995)

- Ion exchange resins have high capacities for ions that are found in low concentration.
- Ion exchange resins are stable and readily regenerated (extended period of usage – years).
- Temperature effects are mostly negligible.
- The ion exchange process is excellent for both large and small installations.

In ion exchange process ions of a given charge (either cation, positively charged or anion, negatively charged) in a liquid solution are absorbed by a solid material (ion exchanger) and are replaced by equivalent quantities of other ions of the same charge as released by the solid material. Cations are interchanged with other cations and anions are interchanged with other anions. The ion exchange is a reversible process and this allows extended use of the sorbent resin before replacing them. During the ion exchange process, electroneutrality and equilibrium are maintained in both liquid and solid phases. The principle of ion exchange is accepted to be the reversible of ion interchange of cations or anions between a solution phase and a solid phase according to the following cation exchange and anion exchange reactions respectively: (PROJECT: vol. 2, 1982)

$$R^- - A^+ + BY \longleftrightarrow R^- - B^+ + AY, \tag{2.1}$$

$$R^+ - X^- + AY \longleftrightarrow R^+ - Y^- + AX, \tag{2.2}$$

where R – is a solid material (resin bead),

A^+ and B^+ – are positive ions taking part in the cation exchange.

X^- and Y^- – are negative ions taking part in the anion exchange.

Ions with opposite charge to that of the exchanger are known as the *counterions* (A and B) and ions with the same charge as that of the exchanger are called the *co-ions* Y in the case of equation (2.1) (Philbrick, Holmyard & Palmer, 1949; PROJECT: vol. 2, 1982).

A number of ion exchange (IX) systems have been developed for desalination and are found to be able to remove organic material. When desalination is for potable water, production contains either a strong and weak resin combination or two weak resins. A conventional ion exchange process for water desalination consists of cation resin and anion resins in series. Both these resins can be strong electrolyte resins but at least one needs to be strong to split natural salts or else the two weak resins have to be coupled in a special process (e.g. DESAL process) to perform this function. Ion exchange removes ions from aqueous phase by exchange of cation (positively charged ions) or anions (negatively charged ions) between the contaminants (feed) and the exchange medium (resin beads). Ion exchange material may consist of resin made from synthetic organic

materials that contain ionic functional groups to which the exchangeable ions are attached or inorganic and polymeric materials. Resins get exhausted after a period of use and it needs to be regenerated (Kirk-Othmer Encyclopedia. 1995). The duration of ion exchange process is typically midterm to long term depending on the application. The considered ion exchange process currently used for the removal of salts from water employ a method that involves passing the water through columns of cation exchange and anion exchange resin beads in H^+ (hydrogen) and OH^- (hydroxide) forms respectively. The sodium ions (Na^+) from the feed (water) are the cations and they exchange with hydrogen ions (H^+) from the resin beads which are also the cations (Korngold & Volsi, 1992; Korngold & Volsi, 1991). The acidic stream coming out of the cation column is then passed through the anion column. In this column the anions (Cl^-) from the acidic stream exchanges with the anions of the anion resin beads (OH^-) to form a neutral solution. The output stream from the anion load column is the product water of the purified stream. The total number and availability of the resin charged groups determine the capacity of the ion exchange. The capacity is affected by the accessibility of the charged group, effluent concentration and ionic strength, the nature of the counter ions, pH and temperature (Foutch, Kar & High, 1995; Glimshaw. & Harland, 1975; Kraaijeveld, 1991).

2.2.1 Ion Exchange Resins (Solid Phase)

Ion exchange resins are insoluble polymers in either basic or acidic form and are used for removal of acids and bases in solutions. Types of ion exchange resins are categorized by the nature of functional groups attached to the polymer matrix (chemistry of a particular polymer in the matrix) and by porosity of the polymer in a matrix. Most ion exchange materials (resin beads) are manufactured by a suspension polymerization process using styrene and divinylbenzene (DVB). The amount of divinylbenzene (DVB), the

crosslinking material determines relative strength of the bead. The polystyrene-DVB matrix that is formed during processing needs to be chemically activated to make it performs the exchanging function. Active groups are attached to the matrix to provide functional groups of either cation or anion resin characteristics of the beads. The basic material requirements of ion exchange resins are *insolubility*, *bead size* and *resistance to fracture*. The resin must be insoluble under normal operating conditions. The resin beads must be in the form of spheres of uniform dimensions, normal size range (16 – 50 US mesh).

Ion exchange resins can be manufactured into one of the two physical structures:

- gel (microporous) or
- macroporous.

Gel resins are homogeneous crosslinked polymers and are the most common types. They have exchange sites distributed evenly throughout the bead. Higher DVB content of the gel resins gives the bead additional strength but the additional crosslinking can hinder kinetics by making the bead too resistant to shrinking and swelling which are very important characteristics during the exchange. Gel resins usually have higher operating efficiencies and costs less. Gel resins have micropore structure. The micropore nature of gel resins gives better physical stability primarily because of its sponge like structure providing more stress relief (Marcel Dekker, Inc. 1992).

Macroporous also referred to as macrorecticular or fixed-pore resins are made from large pores that permit excess to the interior exchange sites. They are manufactured by a process that leaves a network of pathway through the bead. This produces a macroporous structure which combines the favourable characteristics of both high and low degrees of crosslinking. The sponge like structure allows the active portion of the bead to contain a high level of DVB without affecting the exchange kinetics. Unfortunately this also means that the resin will have lower capacity because the beads contain less exchange sites (pores take up 10 – 20% of the polymer). The higher the surface area in a macroporous anion resin gives better organic fouling resistance. In a cation macroporous

resin the higher crosslinking level gives better oxidation resistance. Macroporous resins have:

- high chemical and mechanical stability
- low swelling and volume change
- greater resistance to osmotic sock

The contraction and swelling of the resin bead operation must not cause the beads to burst. Ion exchange resins have the ability to remove *high selectivity* metals like nitrates and other trace metals from water. The resin structure is generally characterized by the pore-size distribution of spherical bead and the degree of crosslinking between polymer chains. Most commercial resins conform to one of three pore-size distribution *gel* and *macroporous* or *isoporous*. Smallest pore-size distribution occurs in the isoporous resins which is manufactured to obtain regular spacing of polymer crosslinking. Gel resins show a greater size distribution than isoporous resins but a more uniform structure than macroporous resins which has uneven distribution of solvent within the beads. Increasing the size of resin pore has a favourable effect on the *rate of the exchange* and *the capacity for ions with higher molecular weight*. Microporous resins usually do not work well in nonaqueous systems due to the disappearance of porosity. Macroporous resins however are more satisfactory in such systems since porosity is retained even if the resins are dried completely. The functional groups are classified into four categories strong acidic or basic and weak acidic or basic resins. Each active group has a fixed electrical charge which is balanced by an active equivalent number of oppositely charged ions which are free to exchange with other ions the same charge (de Graytez, 1991; Marcel Dekker, Inc. 1992; McGraw-Hill Encyclopedia, 1992; Kirk-Othmer Encyclopedia, 1995; Water Quality Association, 1995; Ullman's Encyclopedia, 1989; Kunin, 1958, 1972).

2.2.1.1 Cation Exchange Resins

Acid type resins (weak or strong) are used for removal of cations and are called cation exchangers. The strong acid-type cation exchange resins (sulphonic acid groups) are chemically inert polystyrene beads. When the functional groups are in the hydrogen form and the resin is in contact with a liquid containing other cations, hydrogen ions in the resin will leave the solid phase and enter the liquid phases. Cations from the liquid phase will then replace the hydrogen ion. A regeneration procedure restores the resin to the ionic form, its original form prior to the adsorption step.

The weak acid-type cation exchange resins (carboxylic acid groups) have carboxylic acid group (-COOH) attached to the polymeric matrix. Weak acid cation exchangers have no ability to split neutral salts such as sodium chloride (NaCl). For the same reason this type of resin is not used in water treatment applications because wastewater has sodium chloride as its main constituent.

The features of the cation exchange resins are shown in Table 2.1 (Water Quality Association, 1995).

Features of strong acidic cation resins	Features of weak acidic cation resins
▪ useful on all waters	▪ very high capacity
▪ complete cation removal	▪ very high operating efficiency
▪ variable capacity and quality	▪ only partial cation removal
▪ good physical stability	▪ useful only on specific waters
▪ good oxidation stability	▪ fixed operating stability
▪ low initial cost	▪ high initial costs
▪ low operating efficiency	▪ poor kinetics

Table 2.1. Features of Weak and Strong Acidic Cation Resins.

2.2.1.2 Anion Exchange Resins

Unlike cation resins anion resins cannot readily be divided into strong base and weak base instead they acquire their properties from the attachment of a nitrogen atom to the resin matrix to form groups of amine structures, primary amine, secondary amine, tertiary amine and quaternary amine. Resins whose active group is an amine are generally denoted as weakly basic but their basicity may vary considerably. Quaternary amine behaves analogously to a strong acid resin due to high degree of association. The tertiary amine ($-N(CH_3)_2$) is the most common type. Primary, secondary and tertiary amine resins constitute the weak base group. The strong base-type anion exchange resins have quaternary ammonium groups ($-NR_3OH^-$) where R is usually CH_3 as the function exchange sites. The strongly basic anion exchange resins are divided into two types, Type_1 and Type_2. Type_1 resins are used when total removal of anion is essential (e.g. silica removal). Type_2 resins are slightly basic but also basic enough to remove all anions but release anions more easily during regeneration with caustic soda. This results in a high exchange capacity and a better regeneration efficiency. Though they suffer a

disadvantage of producing greater silica leakage than Type_1 resins (PROJECT: vol. 2, 1982).

Weak base resins are frequently preferred over strong base resins for removal of strong acids to take the advantage of easy regeneration. Weakly basic resins also have slow kinetics and rate sensitive. In addition their capacity depends on the composition of the water which increases as the concentration of strong acids decrease. Only small amount of weak acids is removed.

The features of the anion exchange resins are shown in Table 2.2 (Water Quality Association, 1995).

Features of strong basic anion resins	Features of weak basic anion resins
▪ complete anion removal	▪ high operating capacity removal
▪ low initial costs	▪ high regeneration efficiency
▪ variable efficiency and quality	▪ excellent organic fouling resistance
▪ excellent kinetics	▪ good thermal stability
▪ shorter rinsing period	▪ can be regenerated with, left over caustic from strong base resin, alkaline byproducts, ammonia, soda ash and other weak bases and waste streams.
▪ less organic fouling resistance	▪ Only partially anion removal
▪ limited life	▪ does not remove silica or carbon dioxide
▪ thermodynamically unstable	▪ high initial costs
▪ efficiency versus quality	▪ long rinses
▪	▪ poor kinetics

Table 2.2. Features of Weak and Strong Basic Anion Resins.

Primary application for the ion exchange is the softening and de-ionization of water. Other applications include, waste treatment, catalysis, purification of chemicals, plating, hydrometallurgy, food processing and pharmaceutical uses (de Silva, 1995; Dorfner, 1972; Fleming & Nicol, 1983; Kirk-Othmer Encyclopedia, 1995; Ullman's Encyclopedia, 1989).

2.3 TYPES OF ION EXCHANGE COLUMNS

The various types of combination processes, criteria used in choosing resins type and the recommended operational conditions should be considered. Resin manufacturers provide standard charts and curves for each type of resin enabling calculations of the volume of each resin, amount of regenerant, operating capacity and leakage. Regeneration is carried out with an amount of acid calculated to be slightly in excess of the design capacity. In hydrogen exchange capacity depends on the type of acid used for regenaration. Hydrochloric acid is the most efficient because it dissociates completely in comparison to sulphuric acid. In addition if the resin has taken up a lot of calcium the sulphuric acid must be highly diluted to avoid precipitation of calcium sulphate during regeneration.

The two major decisions in developing an ion exchange operation are the nature of the contacting device to be used and the type of the regenerant chemicals to be employed. Contacting devices can be fixed beds or moving beds. In fixed beds, resin operation, loading and washing are all conducted in one vessel (e.g. batch process). In a batch operation the entire material is brought to contact all at once and the product then removed. The resin and the solution are all mixed in one tank and them allowed to settle to reach equilibrium. The solution is then filtered off and the resin is prepared for the next cycle. This method is limited by the selectivity of resins under equilibrium. For effective utilization of an ion-exchange resin, the equipment in which the resin is placed must be designed to take advantage of the special characteristics of that resin (Ford, 1984; Ford, & Kelly, 1985; Mutjaba, 1997; Funke & Coombs, 1973; Henry, 1982).

Moving beds were developed to improve the batch contact systems. Moving beds can be divided into two basic types:

1) packed beds
2) fluidized beds (also known as unpacked beds).

Packed beds essential features are closed loop column in which resin is periodically pushed around from one section to another. Fluidized beds (unpacked beds) are moving

beds systems that have essential advantage of being able to accommodate particulate matter in the feed (e.g. slime pulp in Uranium recovery systems). The fixed (packed) and fluidized (unpacked) beds can be operated in (co-current or countercurrent and or semi-continuous or continuous) columns. Most fluidized beds are based on countercurrent flow systems, thus the general term continuous countercurrent ion exchange (CCIX). The advantage of this type of operation is the greater efficiency (Marchello & Davis, 1963; Rao & David, 1964).

Fluidized (unpacked beds) of ion exchange resins are used where suspended solids are present in a liquid to be treated and when it is not practical or not desirable to remove these solids. The primary design concern of fluidized bed operation is the complete contact of the liquid and the ion-exchange resin. In the fluidized beds design resin is transferred from stage to stage down the column systematically without interrupting feed flow. Column systems are made of vertical cylindrical vessels with a resin support-liquid collection system at the bottom and a distribution system above the resin bed. Externally the unit is provided with valve manifold to permit operation and backwash, introducing chemicals and the rinsing of the resin (Martinola, 1974; RPI, 1994).

Co-current and countercurrent beds in standard operations, both regenerant solution and the liquid being processed are passed down-flow through a resin column. In a co-current case, both the resin and the liquid being treated flow in the same direction whereas in the countercurrent system the resin and liquid being treated, flow in opposite directions. To obtain the highest regeneration efficiencies and purest effluents, the regenerant solution and liquid to be treated are passed through an ion-exchange column in opposite directions. The countercurrent technique allows the liquid to contact the most highly regenerated resin last, so there is little or no leakage (concentration of salt in the effluent). Countercurrent ion exchange processes provide not only higher purities of treated liquids but also result in higher chemical efficiencies than the co-current operation. To maximize a countercurrent system the resin must not fluidize during the regeneration, if fluidization occurs, resin back mixing will result and this will eliminate realization of the

advantages of this technique. As regeneration levels increase, the chemical efficiencies of the two techniques will become equal, however, for practical ranges of regeneration, the countercurrent method will maintain its advantages of effluent purity (Martinola, 1974).

Semi-continuous and continuous systems are with few exceptions, practiced in columns. In semi-continuous or continuous ion-exchange columns, resins are the pulsed. The resins are pulsed in loop operations for load, regeneration and rinse. These operations are separated by valves that control the sequencing of resin and liquid flows. In the fluidized beds the transfer of resin continues from stage to stage separately and consecutively until the top stage is empty thus it is a continuous system. Continuous separation of ions from a mixture (acid say) is introduced by introducing the mixture of the acid and another solution into the fluid stream continuously circulating it in a closed loop of the system. Most column systems are semi-continuous since flow of the stream being processed must be interrupted for regeneration. Resin or liquid conversion within one column affects the performance of other columns within the plant because columns are interlinked by resin and liquid flows. There are many equipment designs in which ion exchange resins are used for processing operations. There are a few types of different designs used, the single columns, multiple columns, mixed beds, continuous ion exchange, resin pre-coat filter and simulated moving beds (Keller 1992; Remco Engineering, 1998; Sybron Chemicals Inc., 1992; PROJECT: vol. 2, 1982.

The performance of an ion exchange plant is limited by kinetics, equilibrium and mechanical factors of the ion-exchange resin. These effects are minimized when using ion-exchange resins in fixed (packed) beds (Kirk-Othmer Encyclopedia, 1995; McGraw-Hill Encyclopedia, 1992).

The amount of regenerant is chosen according to permitted ion leakage (i.e. it depends on the permissible level of electrical conductivity in the treated water). The greater the amount of regenerant the lower is the electrical conductivity and the greater is the operating capacity. The operating capacity of the resin also depends on the cation

composition of the water more in relation to proportions than to absolute values (George, Ross & Prater, 1968; Kuo, & David, 1963; Sybron Chemical Inc., 1992).

Service water is a second consideration. The operating cycle for water treatment includes backwashing, regeneration and rinsing stages. The salinity of the service water is thus added to ionic load of the production cycle and reduces the resin capacity. The volume of resin must therefore be increased in proportion to the amount of service water used.

Flow-rate and cycle duration are of great importance during the exchange. The specific flow rate should not be too low because a very even flow must always be obtained. Irregularities in flow may cause "*channeling*" in the resin bed, thereby impairing complete exchange. If the flow rate is too high an excessive "*head loss*" occurs. From the calculated operating capacities the volume of each type of resin can be determined as a function of the operating time chosen between two regeneration phases:

$$V_R = \frac{Q_R\, t\, S_R}{C^*}, \tag{2.3}$$

where V_R – is the resin volume [m^3],

Q_R – is the liquid flow rate [m^3/h],

t – is the operating time [h],

S_R – is salinity to be absorbed by the resin [eq/m],

C^* – is the operating capacity of the resin [eq/m^3].

The specific flow rate (Q_R/V_R) should be between 4 and 40 bed volume per hour. If this is not the case the volume V must be appropriately adjusted by shortening or lengthening the operating time (t) (Kelly, 1995; Kirman, 1995).

In most applications the choice of operation methods centres around two concerns:

- should the resin be in a packed or fluidized bed
- should the liquid to be treated and the regenerant solution contact the resin in co-current or in countercurrent fashion.

The performance of an ion exchange is limited by kinetic, equilibrium and mechanical factors. These effects are minimized when using ion-exchange resins in fixed (packed)

bed. The fixed bed provides an exchange zone and an actual exchange front to be setup in the column. The design of fixed bed columns is undertaken based on manufacture's data. Fluidized (unpacked) beds of ion exchange resins are used when suspended solids are present in a liquid to be treated and it is no desire or impractical to remove these solids. Fluidized beds can be operated continuously or semi-continuously. Although the operation is termed continuous, solution and resin flow do not occur simultaneously but periodically. Initially the solution enters the bottom of the column and flows upwards. This fluidizes the resin bed in each stage as created by the separator plates. Normally the feed solution flow would be upward in order to alleviate the pressure drop problems. The advantage of this type of operation is the greater regeneration efficiency. At the end of the solution flow period the resin within each stage is allowed to settle. The length of the solution flow period is determined by the desired operating conditions. On completion of the resin settle, solution flow through the column is allowed to reverse and the packed bed resin moves downwards one stage. When one stage volume has been transferred down the bottom stage leaves the column to the catchpot which can hold a volume of one stage of resin and the upward solution flow is resumed. Solution and resin contact each other under countercurrent but periodic flow conditions. *Fluidized models* are based on *film diffusion* control of the exchange reaction. These models rely on a mass transfer coefficient derived from the linear driving force relationship (Craig, Haines, Nicol, Olën, Douglas & Louw, 1978; Grünewald & Schmidt-Traub, 1999; Koh, Wang & Wanket, 1995, Peel & Benedek, 1981).

2.4 ION EXCHANGE PROCESS FOR WATER PURIFICATION

Natural water supplies contain dissolved salts which dissociates in water to form charged particles called ions. These ions are usually present in relatively low concentrations and permit the water to conduct electricity. These ions are sometimes referred to as electrolytes. To determine how much electricity is the water conducting a conductivity scale is used. These ionic impurities can lead to problems in most manufacturing processes. Common ions encountered in most waters include sulphate, chlorine and silica (Shelley, 1997). The ion exchange resins (solid particles) are particularly well suited for removal of these ionic impurities for several reasons:

- The resins have high affinities for ions that are found in low concentrations.
- The resins are stable and readily regenerated for reuse
- Temperature effects are the most negligible part
- The ion exchange process is excellent for both large and small installations (e.g. from water softeners to huge utilities, industrial plants)

In general, a conventional ion exchange desalination process for producing water of potable quality (purification) consists of cation resin column followed by an anion resin column (Fig.2.1). Cation resin column performs natural salts splitting function and the anion resin column removes the acid that was formed during salt splitting. The considered ion exchange process is a fluidized bed ion exchange column of continuous countercurrent (CCIX) contact device which can be operated at steady state. With the mass transfer rate being controlled either by film or pore diffusion depending on the column operating conditions. The system is modelled on the same basis as such other stage-wise contact devices, distillation and absorption columns. This approach should simplify the design of the CCIX columns and dispense calculation procedures adopted by (Arden, 1968; Dodds, Hudson, Kershenbaum & Streat, 1973; Katoka & Yoshida, 1975, Kirk-Othmer Encyclopedia, 1995; Marinsky, 1966; Shinsky, 1977; Slater, 1991).

2.4.1 Design Considerations of the CCIX Pilot Plant

Currently only the cation columns have been erected at Chemical Engineering Department of Peninsula Technikon with the layout of each column given below (Fig. 2.1). These columns are made of cylindrical Perspex material joined together to form one long cylinder. In this pilot plant, the stage height is 0.5m and the system has 8 stages, this gives the column height of 4m. Column diameters are 400mm for the load columns and 160mm for the regeneration column. Each stage has a multi-orifice stage separator plate with a number of holes for both columns. Load column diameters are determined by allowing for a 50% bed expansion of loaded resin. On this basis and choosing available Perspex tubing sizes which would give approximately the correct bed expansion. Below each of the column is a vessel called the catchpot. Its purpose is to collect and hold the volume of resin and liquid that leaves the bottom stage of the column during resin pull-down period. Top catchpots have also been provided for each column to hold resin just before it is introduced to the column. In practice, the liquid volume associated with the pull-down resin is 120% of the resin volume (Cloete & Streat, 1963; Giddey, 1979; Kelly, 1995; PROJECT: vol. 3: 1982).

The ion exchange pilot plant is built at the Department of Chemical Engineering at Peninsula Technikon is composed of two cation columns (loading and regeneration) based on the NIM CCIX (National Institute of Metallurgy continuous countercurrent ion exchange) design. The National Institute of Metallurgy is now known as the Council for Mineral Technology (MINTEK).

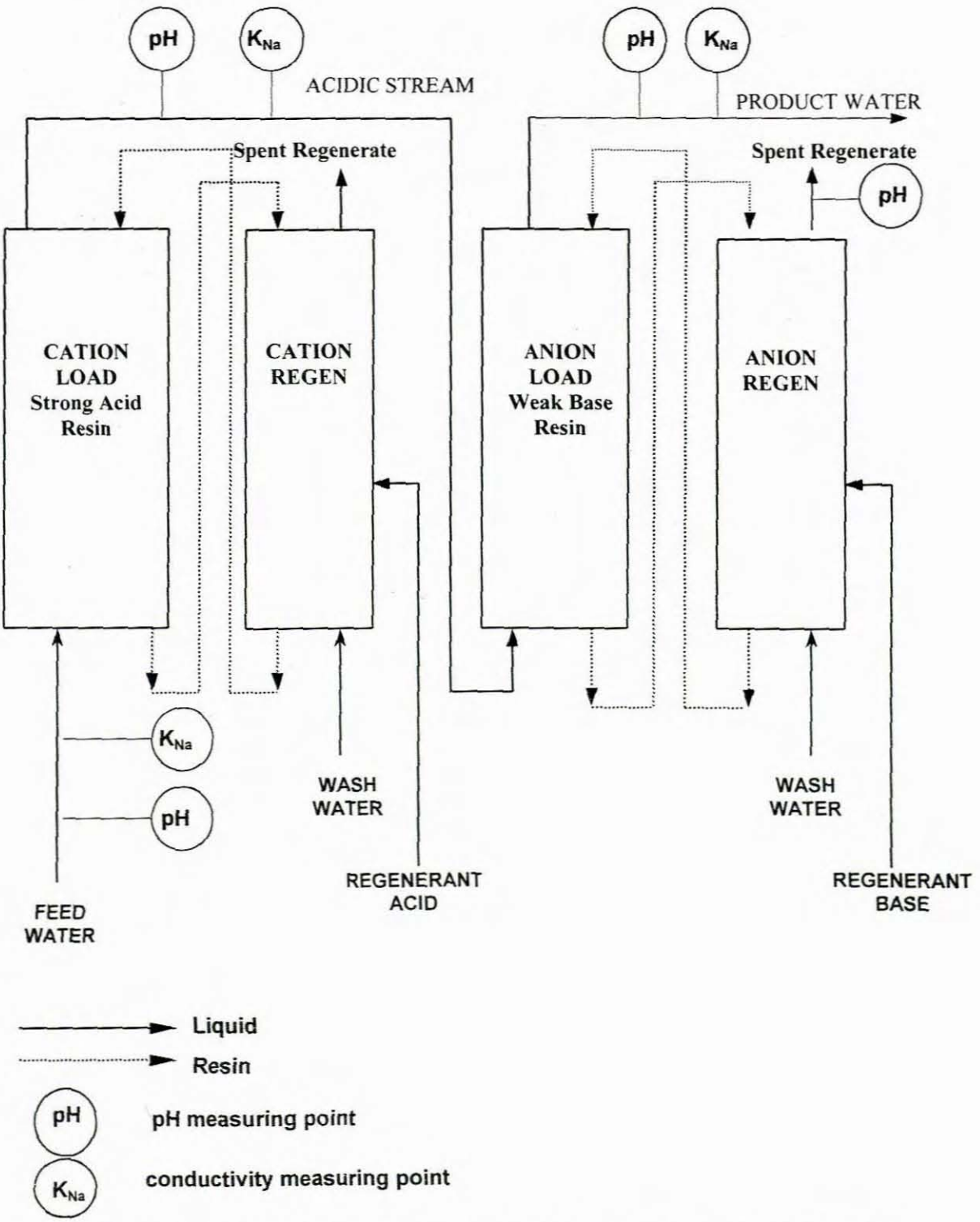


Figure 2.1. Basic Continuous Countercurrent Ion Exchange (CCIX) Configuration.

The method used for determining the size of a continuous countercurrent ion exchange (CCIX) column required performing a particular duty based on theory in the proposed model of the fluidized bed column. The design techniques were used in the following way for the purposes of cost evaluation:-for any particular column, the number of exchange stages were calculated on the basis of the total flow of both resin and solution in the column and stage efficiency. The number of holes is calculated according to the calculations as shown in equations (2.4) and (2.5) below. Each hole should be 2% of open area of total area of the plate. Holes can either be square pitched or triangular pitched. In the case of square pitched area, holes will form a square shape one next to each other, covering all the available area. In the triangular pitched area, holes are structured in a triangular form. From previous experience hole sizes were made 8mm and 10mm for the load column and regeneration column respectively.

$$Number\ of\ holes = \frac{Ap}{2\%Ap} = 50\ \ holes, \tag{2.4}$$

where $Ap = \pi r^2$. (2.5)

Ap – is the available apparent area.

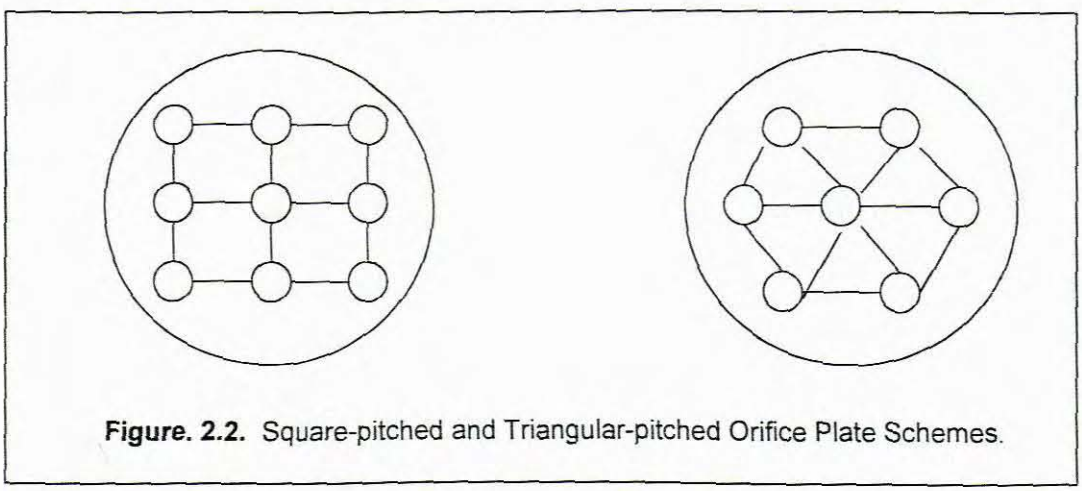


Figure. 2.2. Square-pitched and Triangular-pitched Orifice Plate Schemes.

In the case a square pitch of 50mm apart was used to fit these holes. Equation (4) shows that irrespective of the active area available, it is only the percentage that determines how many holes will be used. Table.2.3 below shows a few examples of different number of holes as calculated using different percentages.

Diameter	Number of Holes				
	2%	3%	4%	5%	6%
Load column (400mm)	50	33	25	20	17
Regeneration column (200mm)	50	33	25	20	17

Table 2.3. Number of Holes as Determined from Percentage of Active Area.

The column design computation can either be performed numerically or graphically depending upon the equilibrium relationship. For the previous investigation the calculation procedure of operation and evaluation of a CCIX column was computerized using equilibrium expressions and kinetic data for relevant exchangers evaluated. The stage efficiencies were evaluated from the particular column contact time via the particular bed expansion.

Together the flow rate and bed expansion determines the column diameter. Experience in the uranium CCIX plant has shown that the optimum stage height is 100cm. This allows the calculation of the volume of each stage. On the basis of stage volume and bed expansion, the quantity of resin in each stage can be calculated. The quantity of the stage, together with the actual number of stages gives the total volume of resin per column (Giddey, 1979, PROJECT: vol. 2,1982).

The pilot plant column dimensions are constrained to a certain extend by the floor area and height available for erection, the need to allow flexibility in the operation of the system under various conditions and the available sizes of material used for the plant

construction. After due considerations the construction material chosen of polyacrylic (Perspex) tubing and sheeting was based on:

- the chemical resistance of this material to inorganic salts,
- ammonia, hydrochloric and nitric acids at concentrations, which would occur within the plant,
- the ease to machine Perspex should the modification be required, the fact that Perspex could be glued if repairs had to be done,
- all constructed equipment would be clear and visual for observation of the phenomena within the column and
- the availability of the material.

2.4.2 Operation of the CCIX Plant

The basic ion exchange of four multiple columns system has been used, the columns are multistage contactors with liquid and resin flowing in opposite directions. It is paired into two main operations of two columns each, cation load and regeneration and anion load and regeneration columns. The continuous countercurrent ion-exchange process (CCIX) which consists of anion load, cation load, anion regeneration and cation regeneration steps must be performed simultaneously and continuously in separate but interconnected columns. The columns contain two resin types (cation and anion) resins, each type with individual hydraulic and ionic exchange properties. The columns operate at a very different solution flow rates and solution concentrations. Each of the operation cycles consists of two principal steps, load (adsorption) and regeneration (Belter & Speaker, 1967; RPI, 1994; Osborn, 1961; Tetra, 1991a,c).

2.4.2.1 Cation Resin Load

In the cation load column salts are converted into their corresponding strong acids using strong type cation resin in the hydrogen form. During cation loading a strong acid resin R-H is used to convert the saline (natural salts of water) into acid. In this column impurities are removed from the feed stream during the up-flow period and valuable constituents with HCl are collected at the output stream. The performance of the load column is rated primarily on meeting the objectives for complete removal impurities and also on the operating conditions and equipment design.

Cation load is widely used to remove undesirable ions from a solution without changing the total ionic concentration or pH of the solution. The cation load reaction can be used in many ionic forms, but the sodium form is usually preferred because the resin has a relatively low affinity for sodium. The ion affinity facilitates the adsorption of other metals. The following reaction takes place at this column:

$$R-H+NaCl \leftrightarrow R-Na+HCl \qquad (2.6)$$

The HCl acid that is formed from the cation load reaction is taken up by weak anion resin in the anion load column in its free base form. This leaves the resin in the R-Na form. This resin must be converted back to the original form R-H and this is done at the cation regeneration operation. Cation load column can either be of strong acidic (hydrogen) cation resin or weak acidic (carboxylic acids) cation resin. In case of strong cation load metallic ions are replaced with hydrogen ions. This leads to a reduction of the total dissolved solids (TDS) in a solution and production of free acid, HCl in this case of the pilot plant being used.

2.4.2.2 Cation Resin Regeneration

Resins are seldom used once and discarded because they can be regenerated for reuse and they can be reused for a very long time. Whether the system is run batchwise or in columns, the resin must be periodically removed from service and be regenerated. The choice of regenerants is very important to ion exchange operation, it determines the cost of the plant which is made of two major costs, the capital costs of the plant and costs of regenerant chemicals. A combination sulphuric acid (H_2SO_4) for cation regeneration and lime ($Ca(OH)_2$) for anion regeneration can be used or ammonia and nitric acid. Though ammonia and nitric acid are easy regenerants to work with from operational point of view and they have the least waste disposal problem, they need additional column, which means extra costs. Sulphuric acid and lime are much less expensive, most likely general-purpose regenerants and are easily available. The problems related with them are low solubility of lime as an anion regenerant and the formation of calcium sulphate scale (precipitation) when sulphuric acid is used as a cation regenerant. These problems can easily be overcome and thus optimizing the usage of sulphuric acid (Sybron Chemicals Inc, 1992).

After the load exchange reaction loaded resin must be converted from *R-Na* to its original form, *R-H* form. If a strong acid is used at the load stage a weak acid is used at the regeneration stage. For example, a sodium-loaded resin is converted back to its original hydrogen form using a weaker acid resin. In contrast, strong acid resin regeneration will only reach equilibrium and will not proceed to completion. Regenerant acid solution has to be successively renewed in order to achieve complete resin conversion. Regeneration of the loaded cation resin can be carried out with approximately 0,5N sulphuric acid solution. This results in a reaction of the form:

$$2R - Na + H_2SO_4 \leftrightarrow 2R - H + Na_2SO_4 . \qquad (2.7)$$

Because of ion exchange limitations, it is usually uneconomical to regenerate the strong resin completely since large excess of regenerant are required to achieve this. Thus after

regeneration a significant fraction of the resins' capacity may still be occupied by counter ions. Certain operating conditions will lead to precipitation and scaling of equipment surfaces during regeneration, ultimately causing blockage of stage separator orifices and failure of the column. Precipitation must then be controlled to have the beneficial effect of promoting the regeneration reaction by removing one of the reaction products. Control of precipitation would be aimed at avoiding supersaturation conditions and at producing fine crystals of $NaSO_4$ which would be washed out of the fluidized bed. The precipitation control could be done using by-processes like seeding (Giddey, 1979; PROJECT: vol.2, 1982).

2.4.2.3 Anion Resin Load

The anion load which uses weak base resin R'-N to remove the acid from the solution from the cation load column. In the anion load HCl acid from the cation load reacts with the resin in its free-base form and the following reaction takes place:

$$\begin{aligned} &HCl \leftrightarrow H^+ + Cl^-, \\ &R' - N + HCl \leftrightarrow R' - N + H^+ + Cl^- \rightarrow R - NHCl. \end{aligned} \tag{2.8}$$

Firstly to enable the functional group to attract an ion it must be protonated. In this way the entire acid is removed from the solution. The tertiary amine functional groups on the resin are unchanged in their free-base form.

2.4.2.4 Anion Resin Regeneration

The loaded anion resin is regenerated using slurry of lime and the following reaction takes place:

$$2R-NHCl+Ca(OH)_2 \leftrightarrow 2R-N+CaCl_2+H_2O. \qquad (2.9)$$

Only a small excess over the stoichiometric amount is needed to maintain a pH greater than 12,0 throughout the regeneration column. The calcium salts of the organic acids stripped from the resin form an insoluble flocculated sludge in the presence of the abundant calcium ions in solution and the free lime. Calcium usually remains on the regenerated resin even though most of other cations are removed. That portion of the resin capacity which is in the hydrogen form after regeneration in the effective process will result in a greater effective working capacity, allowing less resin to be recycled for a given *desalination duty*.

In the countercurrent ion exchange the columns are multistage contactors with liquid and resin flowing in opposite directions. The operation of this system is cyclic for both loading and regeneration of resins with three distinct phases, liquid up-flow, resin settle and resin pull-down. These phases collectively form the primary cycle of the plant flow. The first flows, primary cycles are regulated on the basis of time. The secondary cycles include resin transport, de-watering and rinse phases. The CCIX column operates as a periodic process with liquid and resin flows occurring sequentially rather than simultaneously. The flow sequence is then repeated on a continuous basis. The time lapse between the repeated occurrence of an event is termed a cycle. In each cycle, the following operations take place:

- for the major portion of the cycle the liquid enters the bottom of the column at a set concentration and flow rate. During this period the resin within each stage of the column is fluidized and the product solution overflows at the top of the column

- following liquid flow period is a resin settle in which resin in each stage is allowed to settle at the bottom of each stage forming a packed bed.
- after the settle period, a downward flow through the column pulls the resin down from each stage to the next with the resin of the last stage collected by the catchpot.

The three steps constitute one cycle of a CCIX column. The column operating conditions are set on the basis of the resin capacity, the feed concentration and flowrate and the required degree of either resin or liquid conversion (Kirk-Othmer Encyclopedia, 1995; PROJECT: vol. 2, 1982).

2.4.2.5 Cyclic Operation of the CCIX Column

- **Step1: Liquid Up-flow**

Water to be treated (feed) gets into the process through the bottom of the load column. The sodium chloride salt which is the main constituent of feed water is split during this period. During the up-flow period, the solution flow rate into the column is controlled through an automatic sensoring device based on the position of a float within a rotameter tube. Resin is loaded or regenerated during an up-flow cycle which typically lasts for about 30 minutes to 1h 30 minutes depending on the flow rate being used. During this up-flow cycle, the resin on each stage of the contactor is fluidized as the feed passes through each stage. On completion of the up-flow period, the feed flow is switched off through a pneumatic actuation of a valve and the resin is allowed to settle.

- **Step2: Resin Settle**

On completion of the up-flow period, the feed flow is switched off through a pneumatic actuation of a valve and the resin is allowed to settle (settle period). The length of this period is set to allow complete settling of the fluidized column with each stage of the column. It is typically 30 seconds but varies from column to column with the plant design.

- **Step3: Resin Pull-down**

Each resin settle cycle is followed by a much shorter downflow cycle to transport resin from one stage to the stage below. This period is only operational in the control of the column and it is not based on a time cycle. Instead a sensor placed on top of the column causes switching to occur when the liquid level in the column has dropped by a certain preset volume. In this manner, the volume leaving the bottom of the column can be accurately controlled. The resin at the bottom stage is first moved to a catchpot below the column. From the catchpot it is pumped to the top stage of the next column, regeneration if the resin is from the load column and to the load column if the resin is a regenerated resin. After the pull-down period the resin occupies about half the stage height, this means that each stage is fluidized at 50%. When the liquid level sensor terminates pull-down, the up-flow commences.

Various periods in a column cycle may be summarized as follows, primary cycles (up-flow, settling, pull-down) and secondary cycles (resin transport, rinse). The first flows, primary cycles are regulated on the basis of time. On commencement of the next up-flow period, resin that was pulled down to both the cation and anion regeneration catchpots is immediately transferred to the load columns. At the entry of each catchpot is a slotted resin screen which allows solution but not resin to pass through the screen opening.

The resin stage volume is the volume which when expanded by the set flow rate fluidizes to fill the particular stage under consideration. If the pull-down volume is less than the stage volume, then a degree of backmixing is introduced. Alternatively if the pull-down volume is greater than the stage volume resin bypass occurs (efficient utilization of resin). The pull-down volume and resin capacity together determine the resin equivalent flow per cycle. The stoichiometric ratio and the resin equivalent rate dictate the number of liquid equivalents required by cycle. Therefore, for any given feed concentration with set feed rate and stoichiometric ratio the duration of the feed flow time (up-flow time) is altered to achieve the required liquid equivalents per cycle (Giddey, 1979; PROJECT: vol. 3, 1982).

2.4.2.6 Control Operation Design Requirements

- **Operating Objectives**

The starting point in development of the control structure for some process is a qualitative formulation of the control objectives. Many will be determined by the specific nature of the process involved. General rules can not, nor need be given. In the first category of control objectives are those related to the operational feasibility. They are always function of process variables, which are to be kept within certain specified bounds, in spite of uncontrolled influences on the process. The origin of these requirements may be product quality specifications, safety considerations, operational requirements, environmental regulations etc. In the second category economic considerations are considered.

In attempting to design a feedback optimizing control structure, the aim is to translate the economic objectives into process control objectives. The objectives of the considered investigation are:

1) To improve the consistency of control applied to ion exchange process and thereby improve performance of the plant.
2) To develop methods and programs for real time implementation of the control.

The questions which have to be considered at the beginning of development of design methods and control implementation programs, are:

1) Define and prioritize operating objectives,
2) Identify disturbances variables,
3) Select basic column controls,
4) Locate composition (concentration) sensitive control point.

Operating objectives normally consist of composition specification.

- **Disturbances Variables**

The disturbances expected to enter the process during operation have to be defined and their impact on the performance evaluated. Only then the need for a certain controller can be established. This makes process control different from control in other disciplines where the disturbances can generally only be described in a noise-like manner.

The purpose of the ion exchange control system is to minimize the effect of disturbances variables on product concentration. A host of disturbances including changes in

- feed rate,
- feed concentration,
- environmental conditions,

may be encountered in the plant. Two approaches can be followed in minimizing consequences of expected disturbances:

- Firstly, design the ion exchange column control so that column operation is insensitive to the disturbances, and

- Secondly, eliminate the disturbance by providing secondary controls for this purpose.

Feed concentration is the major disturbance for which the ion exchange control must be designed. The considered main disturbance for the ion exchange process – the concentration of salt into the feed water is with low frequency. It appears at moments when the source of the salt water is changed. This disturbance will influence the model parameters and the optimal steady state values of the process variables.

- **Basic Column Control**

At this step the control variables have to be selected on the basis of collection of data for the operating conditions near the design point and calculation of relative changes in one variable when small step change in another is provided.

There are two possible control variables for the ion exchange column:

- the feed rate and
- the up-flow time of the feed water.

- **Location of Concentration Sensitive Monitoring Points**

The main measurable variables in the ion exchange column are the concentration of the input and output flows of liquid. Two sensors are used to measure and calculate the concentration of NaCl in the water. They are for measurement of pH and conductivity. The sensors are located in the input flow and in the output, product flow of the cation loading column (Fig. 2.1).

All considerations for the steps necessary to be considered before design of the control structure lead to the algorithm (Fig. 1.1) for selecting of control strategy (Morari, Arkun, & Stephanopoulos, 1980, Morari, & Stephanopoulos, 1980).

2.5 THE MASS TRANSFER OPERATIONS IN ION EXCHANGE PROCESS

Ion exchange as many other unit operations of chemical engineering are concerned with the problem of changing composition of solutions and mixtures. Usually these operations are directed toward separating a substance into its component parts. Some mixtures are entirely mechanical and some involve changes in composition of solutions. The latter are known as the *mass transfer operations* and the ion exchange forms part of these operations. Most chemical processes require preliminary purification of raw materials or final separation of products from by-products. Mass transfer operations are usually used for these processes. The mass-transfer operations are characterized by transfer of a substance through another on a molecular scale (Treybal, 1986).

Development of unit operation usually begins with an analysis of the physical behaviour of a system and the establishment of a simplified physical model. Unit operations can be classified to groups based upon similar principles, such as stage operations, rate operations etc. The stage operations are considered by using a generalized model which is applied to all of the mass-transfer operations. The rate operations are introduced with a thorough coverage of the principles of molecular and turbular transport. Stage operations are simply based on stoichiometric and equilibrium concepts. Fundamental principles of rate operations include the operations in which a particle of a phase diffuses or is transferred under the influence of a potential gradient between phases. Basic concepts that comprise a chemical process include, equilibrium, kinetic force, separations, flow patterns and type of the process. Equilibrium is a condition where no interchange of properties takes place between phases of the process (usually mass or energy in chemical processes). When flow rates and equilibrium are known, they are modelled mathematically into operation calculations. All the unit operations are based upon principles of science that are translated into industrial applications in various fields of engineering (Coulson & Richardson, Backhurst & Harker, 1997; Coulson & Richardson, Backhurst & Harker, 1978; Foust, Wenzel, Clump, Maus & Andersen, 1980; Kirman,

1995; McGraw-Hill Encyclopedia, 1992; Kunin, 1971). The unit of operations may be analyzed and grouped using one of the following methods:

- analysis using a simple physical model that reproduces the action of the operation
- analyses considering the equipment used for the operation
- analyses by a mathematical expression that describes the action and testing using experimental data

The physical model is established by careful study of basic physical mechanism. The model is then applied to a real situation either through a mathematical expression or by physical description. Mathematical model of the considered ion exchange process will be used for its analysis and control design.

2.5.1 Ion Exchange Kinetics

Ordinary diffusion can be defined as the transport of a particular species relative to an appropriate reference plane, owing to the random motion of molecules in a region where a composition gradient exists. Although the mechanism by which the molecular motion occurs in different phases involved, essential features of a random molecular motion in a composition gradient are the same. A general theory of transport phenomena suggests that the diffusive flux would be made up of terms associated with gradient in composition, temperature, pressure and other potential fields. The assumed linear relationship between diffusive flux and concentration gradient is traditionally known as *Fick's First Law of Diffusion*, given by (Carslow & Jolger, 1959; Giddey, 1979; Kuo. & David, 1963; Ullman's Encyclopedia, 1989; PROJECT: vol. 2, 1982):

$$J_{d,z} = -D\frac{dC_d}{dz_l}, \tag{2.10}$$

where J_i – is the flux of the diffusing species i of concentration C_i [mol/sec/cm^2],

D – is the diffusion coefficient [cm^2/sec],

C_d – is the concentration of the given d species [mol/cm^3],

z_l – is the length parameter [cm].

Usually it is assumed that all ion exchange particles are spherical and uniform in size. In this case diffusion processes are fundamentally described by Fick's First Law. Equation (2.10) can be rewritten as:

$$J_i = -D\, grad C_i, \tag{2.11}$$

where *grad C* – is the ionic concentration gradient [mol/cm^3].

Equation (2.11) generally relates the diffusive flux vector to the gradient of composition in the three coordinate system. Fick's First Law defines mass-transfer flux through the film for a given ion. Measurement of the mass-transfer coefficient of new and used resins can provide useful information about their ability to operate under critical conditions. Diffusion in the resin phase is always slower than in the solution, due to obstruction created by the resin matrix. Highly crosslinked materials (macroporous) resins have smaller diffusion coefficients. In actual ion exchange the counter diffusion ions possess different mobilities. If the chemical reaction occurs between the counter ions and the fixed exchange sites the rate controlling step is no longer a conventional diffusion process, but chemical reaction kinetics. The rate of ion exchange is then governed by the rate of the corresponding chemical reaction (Boyd, Ademson, & Myers, 1974; Glaski & Dranoff, 1963; Glueckauf, 1955; Helfferich & Plesset, 1958; Montgomery, 1985; Plesset, Helfferich & Franklin, 1958).

2.5.1.1 Film Theory

The earliest model for turbulent mass transport to a phase boundary which is generally attributed to Nernst, suggests, that the entire resistance to mass transfer resides in a film of fixed thickness near the surface to which the transfer occurs. Transport through the film is assumed to be governed by steady state unidirectional molecular diffusion. Composition gradients are assumed to exist in the film only in the direction of transport. The film theory model can be used to predict the effect of high mass transfer rates on the mass transfer coefficients.

For the design of an ion exchange plant the rate of the exchange and sorption reactions involved will influence the size of equipment required to achieve desalination (PROJECT: vol. 3, 1982). Kinetics of ion exchange are governed by diffusion or mass action mechanism, depending on which is lowest step. Diffusion through the film and in the phase occurs at different rates and two steps maybe rate-determining:

Step 1: Diffusion in the bead within the resin, the *particle diffusion.*

Step 2: Diffusion in the Nernst film, the *film diffusion.*

In general the diffusion of ions in the external solution is termed *film diffusion* control. The diffusion or transport of ions within the exchanger phase is commonly termed *particle diffusion* control. A static layer of solution, known as the *Nernst film* surrounds the bead. This film is defined such that it is unaffected by conventional flow around the bead. Ion exchange takes place by diffusion only (no chemical reaction). High flow rate decreases film thickness. The ion concentration is practically constant outside the Nernst film and a concentration gradient occurs within it. Chemical reaction at the exchanger sites can be rate controlling in some case (Kataoka, Yoshida & Ozasa, 1977; Schloge & Helfferich, 1957; Turner, Church, Johson & Snowdon, 1966). Kinetics of ion exchange maybe divided into five steps (Fig. 2.3), (Kirk-Othmer Encyclopedia, 1995; Ullman's Encyclopedia, 1989; PROJECT: vol.2, 1982):

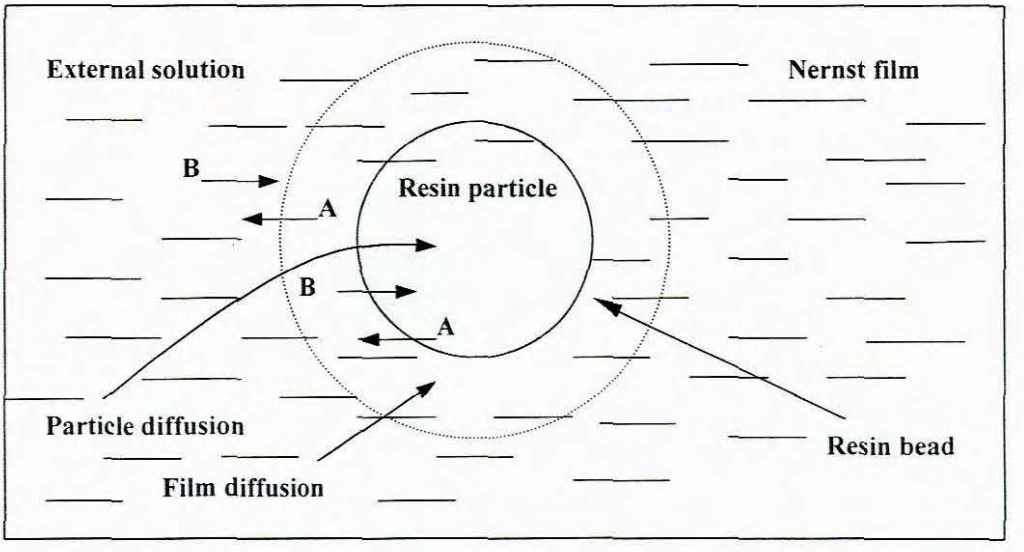


Figure 2.3. Kinetics of the Ion Exchange Reaction.

1. diffusion of the counter ions through the bulk solution to the surface of the ion exchanger
2. diffusion of the counter ions within the solid phase (resin bead)
3. chemical reaction between the counter ions and the ion exchanger
4. diffusion of the displaces ions out of the ion exchanger
5. diffusion of the displaced ions from the exchanger surface into the bulk solution

The rate of exchange or kinetics of the ion exchange reaction is governed by several factors:

- the solution being treated (the *contact time* and the *ionic concentration*),
- the *amount of crosslinking* (divinylbenzene, DVB content),
- the *size of the ions* being exchanged,
- *temperature* and
- also the *strength of the exchange site*.

Any cations diffusing out of the resin into the solution create a net negative charge in the solution and the resulting potential difference is called the DONNAN POTENTIAL. The DONNAN POTENTIAL prevents anions from penetrating the negatively charged resin. The process is called DONNAN EXCLUSION. For the cation resins the fractional attainment of equilibrium is faster in electrolyte desorption process than it is for an anion exchange reaction. This is due to the DONNAN EXCLUSION principle which effectively changes the co-ion diffusivity by creating a potential gradient within the resin bead which helps in the desorption step. Anion resin has slower electrolyte desorption rate when compared with the cation resin. At high solution concentration there is a tendency for the bead radius to decrease which leads to rate increase. Due to this process the co-ion, anion in this case, would not participate in the exchange process. Firstly the exchanger must have a balance of charges, it must be in equilibrium and interaction of ions must be accountable. In addition selectivity of each ion is the basis for development of the exchanger. The DONNAN EXCLUSION allows for acceptance of one ion ever another due to the ability of the resin to exclude the other ion (Crank, 1956; Smith & Dranoff, 1964; Wierzchowski, 1995).

Principles of the considered counter current ion exchange are illustrated in (Fig. 2.4, Ullman's Encyclopedia, 1989) below. The figure illustrates the uptake of Na^+ ions by a resin bead of H^+ form. The bulk solution contains a large excess of available Na^+ ions with an *effect constant concentration* (Levenspiel, 1962).

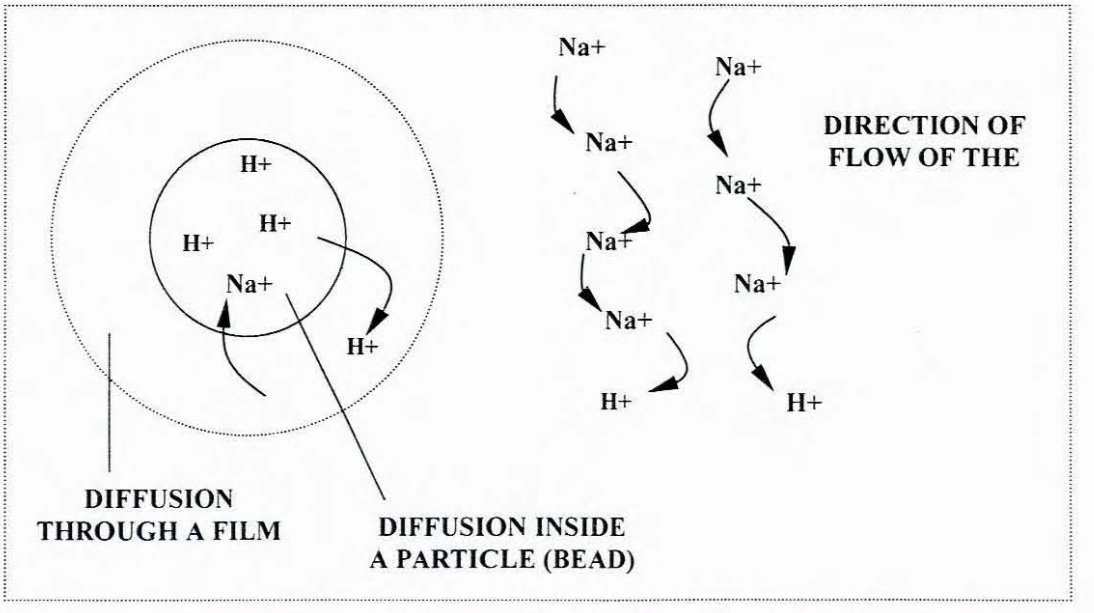


Figure 2.4. Film and Particle Diffusion of the CCIX Process.

The slower step controls the overall ion-exchange rate. A criterion has been established by Helfferich (1962), to determine which process is rate-determining. The Helfferich constant is calculated:

$$H = \frac{\underline{C}\,\underline{D}\delta}{CD\gamma}(5 + 2\alpha_B^A), \qquad (2.\ 13)$$

where $\underline{C}$ – total ion concentration in solution,

C – total ion concentration in the solid phase (total capacity),

D, $\underline{D}$ – diffusion constant,

H – Helfferich constant,

α_B^A – is separation factor between ions of species A and B

δ – thickness of Nernst film,

γ – radius of ion-exchange bead.

When H is far greater than 1 the film diffusion is rate limiting and when H is far less than 1 the particle diffusion is rate limiting. The definition of H shows that film diffusion is favoured by the following high resin capacity, thick Nernst film (low flow rate), low concentration C in the solution, small resin beads and high selectivity (high separation) (Bunzl, 1995; Giddey, 1979; Helfferich, 1962; Kirk-Othmer Encyclopedia, 1995; Ullman's Encyclopedia, 1989; PROJECT: vol. 2, 1982).

2.5.1.2 Film diffusion

When the rate controlling step is film diffusion in ion exchange, it is usually the solution properties which dictate the control. Two major solution properties generally responsible are the film thickness and the concentration. The film thickness is the function of flow of solution around resin bead and a degree of shear that exists between the solid and liquid (Fig.2.4) above. The film thickness decreases due to increase in shear forces, this increases the rate of transfer through the film. The solution concentration has a direct effect on the rate of exchange. The gradient across the film is the difference in concentration between the bulk solution and the equilibrium at the bead surface. The gradient is directly proportional to the concentration of the bulk solution. Film diffusion is based on assumption that equilibrium at each solution interface is determined by the composition of the bead at any point in time. As the bulk solution concentration increases the concentration also increases. At concentration higher than 0.1N the film diffusion is no longer rate controlling. For the high concentration the high gradient makes film diffusion faster than particle diffusion. Two properties of – resin bead diameter and resin capacity – also have an effect on film diffusion rate. The following rate expressions for linear concentration gradient across the film is valid (PROJECT: vol. 2, 1982; Water Quality Association, 1995).

$$R = k_L a(C_A - C_A^*), \tag{2.14}$$

where R – rate of exchange,

k_L – mass transfer coefficient,

a – area where exchange can occur,

C_A – A ion concentration in bulk solution,

C_A^* – A ion concentration at the resin surface in equilibrium.

The assumptions made were that the bulk solution is perfectly mixed and equilibrium exists at the resin surface.

At concentrations up to 10 *meq/l* and flow rates up to 120m^3/h used in water treatment diffusion rates through the resin mass are much greater than through the surrounding film. The film thus controls the rate of the exchange and the process exhibits film controlled kinetics. If the flow rate is slow enough, equilibrium is established as the solution reaches a new layer of the resin. In the slow flow wavefront is only slightly diffuse. Each successive layer of resin being almost completely exhausted before leakages occur. As flow rate increases equilibrium is no longer reached because the Nernst film thickness is an inverse function of function rate the film becomes thinner as the flow rate increase (PROJECT: vol. 2, 1982).

2.5.1.3 Particle Diffusion

As the concentration of ions in solution increases the mass-transfer rate through the film rises until it exceeds the diffusion rate through the resin beads. The diffusion through the resin then because the controlling factor of the ion exchange and the system is said to exhibit particle-controlled kinetics. This condition occurs mainly during regeneration of resins with solutions having concentrations between 1N and 3N.

For loading and regeneration of resins an attempt is made to maximize the rate of exchange if the resins are performing a desalination function. Eventually rate limiting step when the flow past the resin is high or the solution concentration is high likely to be

pore diffusion controlled. Factors affecting particle diffusion are counter ions within the solution, the degree of crosslinking and the resin pore structure. Particle diffusion occurs when counter-ions move within the solution contained in resin pores. Increasing the degree of crosslinking of a resin decreases the ability of a resin bead to swell and this contributes to smaller pores size distribution which is a limiting factor to the particle diffusion. It can then be said that the rate of exchange is directly dependent upon the counter ion diffusitivities which in turn are dependent upon the resin pore structure. External solution properties have no effect on pore diffusion and the rate is inversely proportional to the bead radius (Vermeulen, 1955). The particle diffusion rate models are linear driving force and quadratic driving force expressions (2.15), (2.16) (Giddey, 1979; PROJECT: vol. 2, 1982):

$$N_A = \frac{kD}{r^2}(q_A - \bar{q}_A), \tag{2.15}$$

$$N_A = \frac{kD}{r^2}\left(\frac{q_A^2 - \bar{q}_A^2}{2q_A}\right), \tag{2.16}$$

where N_A – flux of ion species A,

kD – diffusivity coeffitient (constant),

r – resin bead radius,

q_A – concentration of species A at the bead surface,

$\bar{q}_A$ – mean concentration of species A within the bead.

2.5.1.4 Kinetic Leakage

The concentration of ions that is not taken up by the particle but passes into the treated solution is called *ion leakage*. Kinetic ion leakage is the ration of the flow rate of the solution to be treated to the volume of the resin. The specific flow rate is expressed as cubic meters of solution per hour per cubic meters of resin or as bed volume (BV) per hour.

Leakage curves are used to show the effect of ionic concentration and flow rate. If the flow rate is too high to allow the migration of ions to exchange sites in the resin particles the leakage curves show that less bed volumes are treated at lower leakage percentages of influent concentration. Shrinkage has a negative effect on the kinetics whereas swelling opens up and improves migration of the constituents to be absorbed. Once ions appear in the effluent, they increase in concentration as more solution passes through. Since the curves show the effluent concentration versus the volume of the solution treated they are called *leakage curves* (the amount of leaked ions). This determines how much target ions pass through without being removed during the exchange (Keller, 1992; Kirk-Othmer Encyclopedia, 1995; McCabe & Thiele, 1925; Sybron Chemicals Inc., 1992; Ullman's Encyclopedia, 1989; PROJECT: vol. 2, 1982).

The selectivity of strongly acidic resins for the cations increases in the order $Na^{+}<Mg^{2+}<Ca^{2+}$. The sodium ion (Na^{+}) is the first to emerge in the treated solution, the other cations are more strongly held by the resin. This explains the high leakage obtained for water having only sodium as a cation. The quality of treated (demineralized) water is generally expressed in terms of electrical conductivity, sodium leakage from the cation exchange column is the main contributor to the conductivity of the treated water. Weakly acidic and weakly basic resins are almost 100% regenerated at the start of the load operation but their exchange rate is relatively low so that some ions are not taken up and pass through the bed, this is known as *kinetic leakage*. Strongly basic or strongly acidic resins are always returned to service after regeneration but are partially regenerated. In

an exchange where salts from the solution in the load are retained by the regenerated resin layers, each cation that is taken up by the resin ejects a counter cation ion from the resin. The counter ions move down the column and reach a region where the resin is only partially regenerated. Reverse exchange takes place (self-regeneration) this leads to leakage of the exchanging ions from the solution back to the treated water (Sybron Chemicals Inc., 1992).

2.5.2 Ion Exchange (Resin) Equilibrium

For the process in equilibrium, there exists a potential difference (force) that tends to keep the system at equilibrium. When the phases are not at equilibrium and are in contact there is the tendency for a change to bring the phases to equilibrium. The counter flow principle is used mostly in chemical engineering operations in order to allow greater transfer of a property that would be indicated by the attainment of a single equilibrium between the contacting streams. Important properties of ion exchange materials are

1. to preserve electroneutrality, *ion exchange is stoichiometric, capacity is independent of the nature of the counter ion* (exchanging ion)
2. ion exchange is nearly always a reversible process
3. ion exchange is a rate controlled process, usually governed by diffusion in the bead or the surrounding stagnant liquid film.

Preservation of electroneutrality through the bead dictates that fluxes of A and B should be equal but opposite

$$Z_A\phi_A + Z_B\phi_B = 0, \tag{2.16}$$

where Z_A, Z_B – are the valances of elements A and B respectively,

ϕ_A, ϕ_B – are the overall coupled flux of counter ion A and B respcetively

The total counter-ion concentration is fixed by,

$$Z_A \overline{C}_A + Z_B \overline{C}_B = \overline{C}, \tag{2.17}$$

where $\overline{C}$ – is the resin capacity,

$\overline{C}_A, \overline{C}_B$ – are resin phase concentration of ions A and B.

When a particular form of either strong or weak cation and anion resin is placed into an electrolyte solution containing a given concentration of counter ions, a number of mass transfer processes commence. The extent to which the swelling solute sorption and ion exchange proceed depends on the structure and the functionality of the resin and properties of the solution in which it is placed. Together the resin and the solution govern the equilibrium point at which the respective mass transfer operations stops, as the forces involved in the mass transfer are balanced (Dekker, 1966; Denbigh,1955)

Ion exchange equilibrium can be characterized by the ion exchange isotherm. Isotherm is a graphical representation covering all experimental conditions at a given temperature. Equilibrium can also be described in terms of separation factor, selectivity coefficient or distributed coefficient. These quantities vary with experimental conditions. Thus any specific value of one of these quantities corresponds to only one point of isotherm surface. Ion exchange isotherm shows the ionic composition of the ion exchanger as a function of experimental conditions. In equilibrium, absence of one counter ion in the solution requires the presence of this ion in the exchanger (Katoka. & Yoshida, 1975; McGraw-Hill Encyclopedia, 1992; PROJECT: vol. 2 & vol. 3, 1982).

2.5.2.1 Separation Factor

The presence of the ion exchange for one of the two counter ions is often expressed by the separation factor. This quantity is particularly convenient for practical applications, for example, calculations of column performance. The separation factor of two exchanging ions A and B (α_B^A) is defined by:

$$\alpha_B^A = \frac{\overline{m}_A m_B}{\overline{m}_B m_A} = \frac{\overline{C}_A C_B}{\overline{C}_B C_A} = \frac{\overline{x}_A x_B}{\overline{x}_B x_A}, \tag{2.18}$$

where m_A and m_B – ionic concentration of ions species of A and B in solution phase respectively,

$\overline{m_A}$ and $\overline{m_B}$ – ionic concentration of ions species of A and B in resin phase respectively,

C_A and C_B – concentration of components A and B in the solution phase,

$\overline{C_A}$ and $\overline{C_B}$ – concentration of components A and B in the resin phase,

x_A and x_B – equivalent ion fraction of A and B in the solution phase,

$\overline{x_A}$ and $\overline{x_B}$ – equivalent of ion fraction of A and B in the resin phase.

The separation factor is described as the quotient of the concentration ratios of the two counter ions in the ion exchanger and the solution. If ion A is preferred over ion B the factor will be larger than unity and if B is preferred the factor is smaller than unity. The factor is not affected by the choice of the concentration units because it is dimensionless. There is also a simple relationship between the separation factor and the ion exchange isotherm. The separation factor usually depends on the total concentration of the solution, temperature and equivalent fraction x_A (Boyd, Ademson & Myers, 1974; Kirk-Othmer Encyclopedia, 1995; Ullman's Enclopedia, 1989; PROJECT: vol.2, 1982).

2.5.2.2 Selectivity Coefficient

Instead of the separation factor the selectivity coefficient can be used for describing ion exchange equilibrium. The selectivity coefficient (molal) is defined by:

$$k_B^A = \frac{\overline{m}_A^{|Z_B|} m_B^{|Z_A|}}{\overline{m}_B^{|Z_A|} m_A^{|Z_B|}}. \tag{2.19}$$

The reason for the use of absolute values of the ionic valence is so that they will correspond to preference for A ion. If molarities or equivalent ionic fractions are used instead of molalities the equation becomes (PROJECT: vol. 2, vol. 3, 1982):

$$k'^A_B \equiv \frac{\overline{C}_A^{|Z_B|} C_B^{|Z_A|}}{\overline{C}_B^{|Z_A|} C_A^{|Z_B|}}, \tag{2.20}$$

$${}^N k_B^A \equiv \frac{\overline{x}_A^{|Z_B|} x_B^{|Z_A|}}{\overline{x}_B^{|Z_A|} x_A^{|Z_B|}}, \tag{2.21}$$

where k'^A_B and ${}^N k_B^A$ – are the molar and the rational selectivity coefficients respectively.

Selectivity coefficients are not constants but depend on experimental conditions. For counter ions of different valences the numerical value of the selectivity coefficient depends on the concentration scale. The general equation is given by:

$$\left(\alpha_B^A\right)^{|Z_A|} = k_B^A \left(\frac{\overline{m}_A}{m_A}\right)^{|Z_A|-|Z_B|} = k'^A_B \left(\frac{\overline{C}_A}{C_A}\right)^{|Z_A|-|Z_B|} = {}^N k_B^A \left(\frac{\overline{x}_A}{x_A}\right)^{|Z_A|-|Z_B|}. \tag{2.22}$$

The essential difference between separation factor and the selectivity coefficient is that the selectivity coefficient contains the ionic valence as exponents. Thus the separation factor is usually quite different from the selectivity factor if the valences of the competing counter ions are not equal. In such cases the selectivity coefficient remains nearly constant when varying experimental conditions. For single-monovalent exchange the separation factor α_B^A and the selectivity coefficient (k_B^A) are identical and practically

independent of the total salt concentration. For single-divalent exchange α_B^A is a function of the term $k\frac{\overline{C}}{C}$.

2.5.2.3 Distribution Coefficient

In certain practical applications equilibrium is conveniently expressed in terms of distribution coefficient of the counter ions. The molal and molar distribution coefficients are defined by equations (2.24) and (2.25) respectively (Kirk-Othmer Encyclopedia, 1995):

$$\lambda_i = \frac{\overline{m}_i}{m_i} = \frac{x_i \overline{m}}{x_i m}, \tag{2.24}$$

$$\lambda_i = \frac{\overline{C}_i}{C_i} = \frac{x_i \overline{C}}{x_i C}, \tag{2.25}$$

where C_i – the concentration of ion species i in solution phase,

$\overline{C}_i$ – the concentration of ion species i in resin phase,

m_i – the ionic concentration of ion species i in solution phase,

$\overline{m}_i$ – the ionic concentration of ion species i in resin phase,

x_i – the equivalent of ion fraction of ion species i in solution phase,

$\overline{x}_i$ – the equivalent of ion fraction of ion species i in solution phase.

The coefficients increase with dilution of the solution, the decrease in m and C. For any given conditions the distribution coefficient can be calculated from selectivity coefficient. However, there is no simple explicit relationship between these quantities. The use of the distribution coefficient is particularly advantageous if the species is only a trace element. In contrast to the separation factor, the selectivity coefficient and the distribution the equilibrium constant is an integral quantity characteristic of the whole isotherm surface

and is a true constant depending on the temperature only. The equivalent constant gives no information about the exact counterion distribution under any experimental conditions. The relationship between the equilibrium constant and the activities of counter ions in the ion exchanger and the solution depends inherently on the definition of the activities. A simple relation exists only if electrolyte sorption and changes in swelling can be neglected.

$$k_B^A = \frac{\bar{a}_A^{|z_B|} a_B^{|z_A|}}{\bar{a}_B^{|z_A|} a_A^{|z_B|}}, \tag{2.26}$$

where a_A, a_B – are the solution phase concentration of ion species A and B respectively,

$\bar{a}_A$, $\bar{a}_B$ – are the resin phase concentration of ion species A and B.

The above relation shows that the numerical value of the equilibrium constant depends on the choice of the *standard states*. The reference state, active coefficients equal to unity is a solution or a fictitious pore liquid of infinite dilution. The standard state activity coefficients equal to unity is and approximately one molal solution or pore liquid.

2.5.2.4 Ion Exchange Selectivity

The selectivity or affinity of ion exchange resin is influenced by properties of the resin bead, ions being exchanged and the solution in which the ions are present. Water is an essential component of ion exchange resins. The amount of crosslinking of the bead has an impact on the moisture content of the bead. The moisture in turn has an impact on selectivity. A resin bead with high moisture content has a high porosity and the active groups are spaced further apart from each other. Ion exchange resins generally have a greater selectivities for ions with increased a higher atomic weight.

Ion exchange equilibria in cation exchange and anion exchange resins depends largely on the type of the *functional group* and the *degree of crosslinking*. The functional group is

introduced to a resin matrix during manufacturing to formulate the resin as either a cation or an anion resin. The degree of crosslinking is a property that specifies how much can a resin bead swell or shrink, i.e. the degree of crosslinking determines the tightness of the matrix structure and thus its porosity. Crosslinking varies from the outer shell to the centre of the bead and is usually quoted as nominal (in name only) DVB content. The most widely employed method for expression of ion exchange equilibria has been developed from the law of mass action or *DONNAN MEMBRANE* theory (PROJECT: vol. 2, 1982).

For the exchange of cations A and B between a cation exchange resin and a solution containing no other cations, assuming that the ion exchanger is initially in the B form and that the solution contains A ions, the mass action expression for the cation exchange is written as:

$$Z_A \overline{B}^{Z_B^+} + Z_B A^{Z_A^+} \leftrightarrow Z_B \overline{A}^{Z_A^+} + Z_A B^{Z_B^+} \tag{2.27}$$

where $A^{Z_A^+}, B^{Z_B^+}$ – are the ionic species of counter-ions A and B in solution phase,

$\overline{A}^{Z_A^+}, \overline{B}^{Z_B^+}$ – the overbar, denotes the ionic species of counter ions A and B in resin phase,

Z_A^+, Z_B^+ – denote the valency and change of counter-ions A and B.

Thermodynamic equilibrium constant k_a can be defined using the equation:

$$k_a = \frac{(\overline{a}_A)^{Z_B}(a_B)^{Z_A}}{(\overline{a}_B)^{Z_A}(a_A)^{Z_B}}. \tag{2.28}$$

The resin-phase activity coefficients are usually combined into equilibrium constant k_a to provide a new pseudo constant. For most practical applications it is usually satisfactory to assume that the solution phase activity coefficients are almost unity

It is often desirable to use equivalent ionic fractions to represent concentrations in the solution and resin phases.

$$(k_c)_B^A \left(\frac{\overline{C}}{C}\right)^{Z_A - Z_B} = \frac{(y_A)^{Z_B}(x_B)^{Z_A}}{(y_B)^{Z_A}(x_A)^{Z_B}}, \tag{2.29}$$

where $\frac{\overline{C}_A}{C_A} = m_A^* = \frac{y_A \overline{C}}{x_A C}$ – is defined as the distribution coefficient,

$\frac{\frac{y_A}{x_A}}{\frac{x_B}{y_B}} = \alpha_B^A$ – the separation factor,

y_A – the concentration of counterion A in solution phase,

y_B – the concentration of counterion B in solution phase,

If the exchange of equivalent ions is $Z_A - Z_B = 1$, then selectivity coefficient becomes:

$$(k_c)_B^A = \frac{y_A x_B}{y_B x_A} = \frac{y_A(1-x_A)}{x_A(1-y_A)} = \alpha_B^A \tag{2.30}$$

If $k_c > 1$, species A is preferred by the exchanger and for $k_c < 1$ species A is less preferred and the ion exchange sorbs B species. The selectivity of the ion exchange is affected greatly by the degree of crosslinking. Slight crosslinked and highly swollen resin exhibit reduced selectivity for one small in over another. With increase in crosslinking, selectivity is increased. Swelling of resin during the exchange process is a result of an osmotic process during which the osmotic pressure of the resin gel is opposed by tension in the resin structure. Increased selectivity with increasing crosslinking is observed and can be predicted from the theory (Weatherly & Turner, 1976; Ruchenstein, Vaidyanathan & Youngquist, 1971).

When selectivity of a resin of a dissolved ion is high a sharp wavefront is formed this wavefront moves forward the column outlet thus making the maximum use of the resin. In contrast when selectivity is low a diffuse wavefront is formed which limits the use of the resin.

2.6 APPLICATIONS OF ION EXCHANGE

The industrial applications of ion exchange are extremely widespread and range from the purification of low-cost commodities such as water to the purification and treatment of high-cost pharmaceutical derivatives as well as precious metals (e.g. gold and platinum). The largest application, measured in terms of ion exchange resin is water treatment, that is, *water softening*, *water demineralization* for high pressure boilers and *dealkinization*. Indeed, enormous advances in ion exchange technology have occurred because of the relentless requirement for pure and ultrapure water. Other major industrial applications are the processing and decolouring of sugar solutions and the recovery of uranium from relatively low-grade mineral acid leach solutions. The fields of water treatment, effluent and pollution control are predominant (Dyer, Hudson & Williams 1995; Kirk-Othmer Encyclopedia, 1995; Osmonics Inc., 1992; Owens, 1985; Streat, 1995; Tetra, 1991b). Other applications include medicine, pharmacology, chemical processing, catalysis and analytical techniques (Foutch, Kar & High, 1995; Greig, 1996; McGraw-Hill Encyclopedia, 1992; Nachodi, 1956).

2.6.1 Water Treatment

Two primary applications of ion exchange in water treatment are softening and deionization. Water softening is the major use of ion exchangers in water in water treatment. Other applications would include dealkalization, softening produced water, desilicizing and nitrate removal. Removal of hardness from water by ion exchange represents the first major application of ion exchange in water industry. Water softening has been used in some countries in some countries in domestic water supplies. The hardness in water is removed by passing the water over a cation-exchange column in sodium form. Hard water contains mainly the calcium (Ca^{++}) and magnesium (Mg^{++})

ions which cause scale formulation in power plants, boilers, pipe lines, and cooking. In water softening the hard ions Ca^{++} and Mg are exchanged for soft ions like sodium (Na^{+}) from the exchanger. Hard water is passed through a bed of strong acid cation exchanger in the sodium form and is softened.

Because of the change in selectivity with concentration the efficiency of the cation-exchange softening process with increasing salinity of the water. The removal of salts and other ion impurities from water by means of ion-exchange is based primarily on the exchange of cations (Na^{+}, K^{+}, Ca^{2+}, Mg^{2+} etc.) with hydrogen form of a cation-exchange resin, and the exchange of the anions (Cl^{-}, SO_4^{-}, NO_3^{-}, etc.) with hydroxide form of an anion-exchange resin. Softened water prevents such problems as scaling of pipes and heat exchangers. It is quite difficult to obtain complete regeneration of each resin and therefore some cation and anion contamination (ion leakage) of the treated water will occur. Deionization, refers to removal of cations and anions from water and replacing them with hydrogen and hydroxide (Schmuckler, Nativ & Goldstsein, 1976).

An ion exchange process that lowers the bicarbonate concentration in is called dealkalinization. It is used for water supplies having a relatively high alkalinity or ratio of bicarbonate to sulphate and chloride present in water. In secondary oil recovery projects involving stream injection to heat oil remaining in strata and to make it more fluid, the steam condenses and the water becomes contaminated with calcium, magnesium and other salts. This water is cycled back to steam generators after it is separated from the oil. A problem common to produced water applications is the tendency for oil fouling of the resin. In those areas where nitrate concentrates in the water supply have been close to or above allowable limits for potable water, nitrate removal is practiced. Strong base anion-exchange resins are used in the chlorine form. Processing water beyond an acceptable nitrate can result in the concentrate exceeding the influent concentration. The advantage of macrorecticular and isoporous anion resins are relatively easy to regenerate (PROJECT: vol. 3, 1982).

In many industrial it is necessary to remove the hardness and alkalinity from raw water before the water is used in a process. Several process involving ion exchangers are for softening and dealkylizing water. Ion exchangers frequently replace acids, alkalis, and metal catalysts in hydrolysis, inversions, estirifications, hydrations or dehydrations, polymerizations, hydroxylations, and epoxidation reations as acid free catalysts. Another use of ion exchangers is in catalytic distillation in which the reaction and extractive distillation is carried out in a single unit (Osborn, 1961, van Winkle, 1967).

Demineralization is used for treatment of feed-water to high pressure boilers, for treating processing cooling water, for treating process water for various chemicals, pharmaceutical and food applications and for producing high purity water for the electronics and other industries. Demineralization is a process for removing dissolved solids (ionic impurities) from water. This can be accomplished in a high flow deep-bed filter demineralizer.

Ion exchange is also widely used for condensate polishing. In condensate polishing, steam is recovered as condensate after passing through turbines, commonly used in electrical power industry. During the process, small amounts of soluble impurities appear in the condensate. Recycling the hot water after removing these impurities with resins is far more economical then treating cold water. The system is made of columns (deep beds) and thin layers of ground resin deposited on a precoat filter (powdered system). Removal of particulate matter and dissolved solid from utility power plant condensate.

Boron removal, since boron is occasionally present in water supply at an unacceptable level and it cannot be removed with the standard anion exchange resins unless water is deionized. The borate form of conventional strong base anion exchangers is used in some nuclear reactors to adjust the concentration of boron in water used as a moderator.

Application of mixed-bed ion exchange columns in high pressure boilers is now widespread. Ion exchange resins are also widely used in effluent treatment and pollution control. Process strategy depends entirely on the waste to be treated, concentration of pollutants, flow rate, etc., and applications are extremely diverse. Typical present day

processes include treatment of drainage water from mines, removal of ammonia and nitrates from ground water and treatment of nuclear waste solutions. The efficiency of waste treatment is strongly dependent on the regenerant consumption and thus it is usual to attempt treatment processes with either weak acid or weak base exchanger. Success is likely if the process solutions are either acidic or alkaline. Ion-exchange resins can be further used for the removal of noxious gases from gas streams.

2.6.2 Sugar Processing

The sugar and corn sweetener industries have the largest volume of installed ion-exchange resin in food processing industry. Lesser quantities are used to process wine, whey, fruit juices and gelatin.

All sugar that is milled and refined is produced from either sugar cane or sugar beet. Sugar and sucrose is obtained from sugar cane as a juice by pressing cut canes, and from sugar beets by slicing the beets and extracting the sucrose with hot water. Organic and inorganic impurities must be removed from these extracts to obtain a white crystalline product. The principal applications of ion exchange in purification and treatment of sugar solutions, juices and syrups are as follows:

- softening and demineralization of sugar juices to remove scale-forming,
- de-colouring using anion exchange resins,
- catalytic inversion of sucrose to fructose and glucose and
- glucose-fructose separation.

In general , the largest field of application for ion exchange in sugar processing is in the sugar beet industry. Sugar syrup contains significant amounts of calcium and magnesium salts and these can be exchanged with conventional cation-exchange resin in the sodium form. Resins are rarely used for de-colouring due to irreversible fouling that occurs. In recent years, macrorecticular and isoporous strong base anion resins have been used

without prefilters. Resins have the following advantages over granular carbonaceous adsorbents as colouring agents:

- high capacity for colour retention,
- rapid equilibration,
- low rinse water requirements and
- in situ regeneration.

The inversion of sucrose can be catalyzed by a cation-exchange resin in hydrogen form.

2.6.3 Pharmaceutical and Medical Applications

Ion Exchange is used extensively in the fields of pharmaceutical manufacturing and medicine. Some of more important applications are:

- processing of pharmaceuticals,
- use of ion exchangers in pharmaceutical formulations,
- use of ion exchangers and related materials in artificial organs and
- analytical use in medicine.

The separation of antibiotics fermentation broths is an interesting application for ion exchanger. Vitamin B12 is produced by microbial fermentation and can also be separated from broth using a carboxylic acid exchanger. A related antibiotic, neomycin, is also recovered with a carboxylic acid ion exchanger. Vitamin B12 is sorbed by the acid form of the ion-exchanger after being treated with HCl to remove impurities, its product is eluted with an acid-acetone water solution.

In medicine, ion exchange materials have found use as preparative media and for various clinical treatments. In the collection of blood, citrate and dextrose are added to prevent coagulation by passing over the sodium form cation-exchange resin to remove calcium and magnesium, for long term stability.

2.6.4 Hydrometallurgy

Quite a number of metals have been recovered and purified commercially by ion exchange. Ion exchange is particularly suitable for high-cost, low-throughput purification processes. Separately, the recovery of trace amounts of metal from effluent and waste streams accounts for many applications, for example chromium from spent metal effluent and waste streams accounts waste arising in the rayon and synthetic fiber industry. However, the largest single application in hydrometallurgy is the recovery of uranium for uranium in the nuclear industry, although more recently liquid extraction using liquid ion-exchange reagents has found acceptance in some mining locations. Ion exchange is particularly advantageous for the treatment of low-grade uranium deposits (*Rossing uranium mine – Namibia, Africa*).

The removal of zinc from the acid stream is achieved by ion exchange and this can be recovered subsequently by liquid extraction using a cationic extractant. It has become common practice to recover gold from low-concentration side-streams with activated carbon. Conventional multistage fluidization-bed columns are used for the sorption process, although the regeneration step is more difficult and requires high-temperature elution and thermal reactivation of the absorber. Recently there has been renewed interest in the application of exchange resins for gold recovery from cyanide liquors.

Resins are less susceptible to poisoning by calcium organic impurities and possess higher sorption capacity and selectivity for gold. This is achieved with both weak and strong base resins although the former will probably prove easier to elute in practice thus providing a cheaper recovery process.

Ion exchangers are used to recover small amounts of desired solute from large quantities of contaminants by selecting and concentrating the solute. For example, recovering uranium from leach solution by using a strong base anion exchanger, and recovery of copper (Cu) with a strong acid cation exchanger in the hydrogen (H^+) form. Nickle can be recovered from rinse water by using a strong acid cation exchanger. Precious metals,

gold, platinum, and silver can be recovered in the form of metal complexes an anion exchangers.

2.6.5 Waste Treatment

Ion exchangers have been used to remove trace amounts of radioactive materials from water before discharging it to the environment. Synthetic organic cation and anion exchangers based on the styrene-DVB matrix are in a mixed bed system. Strong base anion exchangers in the chloride form are used for the selective removal of nitrates from nitrate polluted waters.

Ion exclusion is a process permitting the separation of ionized substances from nonionized or slightly ionized organic or inorganic substances when both are present in aqueous or semi-aqueous solutions. An advantage is the ability to purify contaminated substances inexpensively using water as the regeneration medium, e.g. glycerin-salt separation, sucrose-salt separation, separation of minerals from acetic acid, separation of salts from acetic acid, separation of salts from alcohols and separation of salts from high molecular weight amines. Ion exchangers are used in processes for removing contaminating acids, alkalis, salts or mixtures from non-ionized or slightly ionized substances.

Ion exchangers are applicable to organic or inorganic compounds (e.g. removal of amines from methanol, removal of iron from steel pickling operation, purification of aluminium, phosphoric acid and removal of ion chloride complex with an anion exchanger).

Ion retardation is a separation process that utilizes an amphoteric ion exchanger (i.e. having both cation and anion groups within the matrix). Absorbed ion pairs are weakly held and may be displaced by water as the regeneration. Ion retardation is used to separation salts from organic solutes and may be used to separate mixtures of inorganics and highly ionized electrolytes (Rousseau, 1995).

Strong acid cation exchanger in the dry state are useful as desiccants. The sodium or potassium form of exchangers is most satisfactory for reusable applications. Ion exchangers show their greatest capabilities in drying solvents such as hydrocarbons and chlorinated hydrocarbons.

2.7 CONCLUSION

Application of ion exchange for water desalination is described. Different operational methods and types of ion exchange are discussed. Design considerations for use of the continuous countercurrent (CCIX) column are considered. Resin loading and regeneration cycles are discussed.

The major disturbance variable are identified as – the liquid-feed salt concentration with low frequency of occurrence. The up-flow time is identified as the column control variable. The used technology for concentration monitoring is pH and conductivity sensors.

Mass transfer operations of the ion exchange process which concerns the problem of the changing compositions is described. This involves ion exchange kinetics, film and particle diffusion, equilibrium, ion leakage and selectivity.

Different applications of ion exchange process are considered – water treatment, waste treatment, metallurgy, medical and pharmaceutical applications and food processing industries.

The knowledge from the Chapter 2 is used in Chapter 3 for development of the ion exchange process model.

Chapter Three

Modelling and Parameter Estimation

The aim of this chapter is to analyze the ion exchange process. It refers to the application of scientific methods to the recognition and definition of the problem of modelling and the development of procedures for its solution. This means that:

- mathematical specification of the modelling problem for the given physical situation,
- detailed analysis to obtain the mathematical model and
- synthesis and presentation of results for the purpose of control design have to be done.

Here the process denotes an actual series of operations or treatment of materials. The model represents a mathematical description of the real process. The processes and models are characterized by variables and parameters. A parameter is a property of the process or its environment, that can be assigned arbitrary numerical values; also a constant or coefficient in an equation.

Development of mathematical model of ion exchange process is for the purpose of optimal control design and implementation.

Development of theoretical and empirical (experimental) models is discussed. General principles of modelling, types of models and their uses are defined. Steps for mathematical modelling of the ion exchange process are detailed. Validation of the

Chapter Three

Modelling and Parameter Estimation

---

theoretical model, estimation of model parameters, if the model is a true reflection of the process is covered

"Mathematical modeling is very much of an art, it takes experience, practice and brain power to be a good mathematical modeler. Remember to always go back to the basics *mass, energy* and *momentum*" Luyben (1990).

3.1 MATHEMATICAL MODELS

Modelling finds applications in areas as diverse as engineering, science, ecology, medicine and agriculture. Different models are built for different needs, a few known models will be models built out of curiosity, models for prediction and control, models for state estimation, models for diagnosis of faults and inadequacies and models for simulation and training. In general, the model will be composed of the basic material and energy balance equations, design relations and physical property equations that describe physical phenomena taking place in the system. Because the dynamics of energetic systems are due to the energy dissipation and storage of energy, it is natural to construct a system model in terms of variables that describe these actions. The variables that describe a system's condition or state are its state variables. The quantity and flux variables are common choices for this role since they describe a system's potential and kinetic energy storage (other are also possible). In general the number of state variables required equals the number of energy storage compartments in the system. The dynamic behaviour of physical systems results from the interchange of energy between potential and kinetic forms and from loss of energy through. What might be essential characteristic for your purpose might not be essential for an investigator with different goals. For example, omitting time-varying aspects in a model rarely results in dramatic failures of engineering design (Brogan, 1974; Sinnot, 1997).

In engineering most processes deal with time-dependent effects such as time dependent behaviour of fluid-flow and heat-transfer processes significantly affects the quality of the product of a chemical process. In order to deal in a systematic and efficient way with problems involving time-dependent behaviour of the objects or processes involved, a ***model*** must be described. The model is used as a tool for process analysis and for the design of control systems. The type of a model that describes the system in mathematical relationship is called the ***mathematical model***. A mathematical model represents a process as a set of equations (relationships) used to optimize process design (Palm III,

1983; Dorf, 1998). These relations will consist of differential or difference equations. Only a suitable model is selected or constructed to meet the requirements of the study. Mathematical models are developed to extract essential information from complicated evidence and to quantify the implications. Mathematical modelling of engineering systems is extension of traditional chemical and physical laws to include time dependent terms in the process. Mathematical models are used to get the insight into understanding the activities that make the process work. This enables the design engineer to identify many confusion factors and get to understand the core of the system. The process of constructing a mathematical model of a dynamic system from observations and prior knowledge about the system forms part of broader field of engineering known as identification. The most vital role an engineer plays in the modelling exercise is the engineers judgement as to what assumptions can be validly made about the process. Questions that arise from the description are: (Himmelblau & Bischoff, 1968; Norton, 1986; Scharzenbach, 1996)

- Why is a model needed.
- Why the dynamic system and what does it signify.
- What sort of mathematical model.
- What sort of prior knowledge and observations.
- How is the model constructed.
- How to reach a conclusion that the model is a good model.

One way to develop a model of a system is to identify the flow paths and storage compartment of mass or energy and to identify and describe quantitative links between these compartments. The dynamics of a physical system result from the transfer, loss and storage of mass or energy. The developed model makes it possible to study the interactions of various parts of the process and to evaluate process control structures and strategies. Models can also be used for simulation of operations like start-up, shutdown and emergency procedures. Models can also be used in troubleshooting control and processing problems in startup and during training of the operator, optimization and

studying the effects of expansions if needed and removal of bottlenecks. A model will perform the above functions in a very cheap, faster and safer method as compared to using the actual operating unit. This does not rule out the use of plant tests. Plant tests are very important in confirming the validity of the model and verification of important ideas and recommendations that evolve from the model.

Any model of a system must contain the minimum amount of information necessary to achieve its purpose at no more for it to be a useful model. This requirement is most immediately reflected in the choice static versus dynamic-element models. These elements whose behaviour is slow relative to other elements are often modeled as static elements (in order to reduce the complexity of the model). For example switching time of a thermostat is fast as compared to the time required for the room temperature to change appreciably. One set of dynamical models commonly used in identification is that of linear, lumped, time-invariant and finite order models. Properties of these models are said to be dynamical as opposed to being static. The feature that distinguishes a dynamic system from a static one is that the output of a dynamic at any instant in time depends on its history inputs, not just the present input. A dynamic system is then said to have memory and it is attributed to some stored energy. Any dynamic model must describe how each of its energy storages contributes to the output. The order of the model is determined by the number of model variables. Some variables may be well known in advance not to influence the output (being zero) and these parameters may be ignored and this will in turn reduce the size of the model (Himmelblau & Bischoff, 1968; Roy, 1981).

The mathematical models can be discussed as experimental plots or using different type of equations – algebraic, partial differential equations. The equations can be linear, or nonlinear, or stationary, or non-stationary.

A question that often arises is why are differential equations commonly used in mathematical description of a physical process. The final objective is normally to develop an algebraic, transcendental or graphical relationship between the input and

output of the process. Why not start directly with algebraic expression? The answer to this question is based on the objectives of the analysis. If there is a process currently in operation and it must be described mathematically for some purpose an empirical algebraic equation can be fitted to the operating data by statistical methods. This empirical model is fundamentally restricted to the given process and limited to the large range of variables for which data were available. One does not pretend that the differential equation itself represents the basic truth of the process of interest. But it closely resembles the process than an approximation by an arbitrarily selected equation. Thus by starting with the general differential laws and by choosing proper boundary conditions models that are applicable to each subsystem can be found. By proper manipulation of the differential equations and boundary conditions, it is hoped to find a suitable mathematical expression for the subsystem. In cases where theory is not complementary some additional experiments should be run in order to qualify (check) the prediction but in cases where basic theory has proved to be correct, experiments won't be necessary (Himmelblau & Bischoff, 1968).

The most fundamental description of process would be based on molecular considerations. Because molecular description treats an arbitrary system as if it were composed of individual entities each obeying its own rules. Equally important in formulating the differential equations or difference equations when developing a model is the selection of appropriate boundary conditions and or initial conditions. Appropriate boundary conditions arise from the actual process or the problem statement either given or most often deduced from physical principles. The boundary conditions are usually mathematical statements that show that the dependent variable at the boundary is at equilibrium or if some transport is taking place that the flux of mass, momentum and energy is conserved at the boundary (Palm III, 1983; Himmelblau & Bischoff, 1968).

3.2 COMMON PRINCIPLES IN DEVELOPING A MATHEMATICAL MODEL

3.2.1 Types of Mathematical Models

The main objective in modelling can be reduced to that of obtaining a functional relationship between the various process variables, which explains the observed process behaviour. The underlying belief is that a true – but unknown – fundamental relationship dictated by natural laws, exists between the process variables, and our modelling exercise is directed towards discovering this relationship. Theoretical modelling seeks to arrive at a fundamental modelling relationship through systematic application of the laws of nature to the most important phenomena assumed to determine the behaviour of the process. The end result is a *theoretical process model.* On the other hand, empirical modelling seeks to approximate this unknown functional relationship by some (usually simple) mathematical functions, using information gathered experimentally from the system. Because they depend entirely on experimental information and experience, the obtained approximating mathematical functions are called *empirical models*.

It is typical to adopt the theoretical modelling approach when the underlying mechanisms by which the process operates are reasonably well understood. When the process is too complicated or the model equations are enormously complex, the empirical approach is the appropriate choice. Clearly it would be wasteful to begin the study with the most complex model if the simpler model could serve to indicate that there is less potential for improvement. The bases for mathematical models are the fundamental physical and chemical laws of conservation of mass and momentum. Development of a model that incorporates basic phenomena occurring in the system requires a lot of skill, ingenuity (invention of new methods) and practice. The model building involves going through three following stages: model formulation, model parameters estimation and model validation (Fig.3.1).

3.2.2 Procedures for Theoretical Model Development

3.2.2.1 Problem Definition

There are several factors one needs to define very clearly the scope of the problem one wishes to solve. Some of these factors are:

- It is impossible to represent all aspect of the physical process one can only hope to capture aspects that are most relevant to the problem at hand.
- The behaviour of a dynamic process can be interpreted mathematically in several different ways, the various phenomena explained to their varying degree of the details. The result is that several different models for any given process all which might attempt to capture the same aspects of the process but from various angles and to varying degrees of complexity can be developed.
- A process model is as useful as the tools available for obtaining solutions to the process equations. Some modelling equations can be solved analytically while some can be solved by numerical methods using a computer.

As a result, before embarking on the actual developing a mathematical model for a physical process, a number of questions that are by no means independent of on another should first be answered.

1) What does one use the model for?
2) How simple or complex will the model has to be?
3) Which aspects of the process are considered the most relevant (and therefore should be contained in such a process model)?
4) To what extent are the fundamental principles underlying the operation of the process known?
5) How can one test the adequacy of the model?
6) How much time one has for the whole modelling exercise?

3.2.2.2 Model Formulation

The following are the principles of formulation:

- assumptions,
- physical laws – conservation principle,
- mathematical consistency,
- parameter estimation and
- verification.

- **Assumptions**

In this area, creativity and innovativeness of the engineer becomes the element of success. When formulating a model some assumptions have to be made to keep the model within feasible boundaries otherwise the problem becomes too large to be solved or impossible to keep under control. Assumptions made should be carefully considered and listed. Assumptions impose limitations on the model and should be kept in mind when evaluating their results. Assumptions should be as simple as possible and reasonable without losing any important information. This process is called *optimum sloppiness*. In practice this optimum corresponds to a model which is more complex as the available computing facilities will permit.

- **Physical Laws – Conservation Principle**

There are basic laws used for mathematical model development:

- continuity equations, and energy equations
- equations of motion (momentum) transport equations and

equations of state.

The models are built on the basis of basic chemical or physical laws applying the *conservation principle*. The general conservation principle in essence, states that:

"What remains accumulated within the boundaries of a system is the difference between what was added to the system and what was taken out of the system, plus what was generated by the internal production."

$$Accumulation = Input - Output + Internal\ Production \tag{3.1}$$

It is often more appropriate to present the conservation principle on terms of rates. The rate of accumulation of a conserved quantity q within the boundaries of a system is the difference between the rate at which the quantity is coming in and the rate at which it is taken out plus the rate of internal production. Like the equation

$$\begin{aligned} Rate\ of\ Accumulation\ of\ q = Rate\ of\ input\ of\ q - \\ - Rate\ of\ output\ of\ q + \\ + Rate\ of\ Production\ of\ q \end{aligned} \tag{3.2}$$

The fundamental quantities that are being conserved in chemical reactions in all cases are either mass, or momentum, or energy or the combination of these. Such balances could be made over the entire system, to give overall, or macroscopic balances, or they could be applied to portions of the system of different size giving "differential" or microscopic balances.

The balances are expressed by explicit equations for the rates. The most widely used are:

- Basic definitions in terms of physical properties as density
- Transport rate equations - Fick's law of diffusion

- Chemical kinetic rate expressions - based on the law of mass action, Arrenius expression of temperature dependence in reaction rate constants.
- Thermodynamic relations - equations of state (e.g. ideal gas law, van der Waal's equation) and equations of chemical and phase equilibra.

Continuity equations are classified into total continuity equation (mass balance equation) and component continuity equation (component balance equation). The mass balance equations are used to model the process under consideration. These equations may be with respect to the mass of individual components in a mixture or a component mass balance, or with respect to total mass. The general form of equation is:

$$\begin{aligned} Rate\ of\ Accumulation\ of\ mass = Rate\ of\ Input\ of\ mass + \\ + Rate\ of\ Generation\ of\ mass - \\ - Rate\ of\ Output\ of\ mass + \\ + Rate\ of\ Depletion\ of\ mass \end{aligned} \tag{3.3}$$

If there are n components in a mixture, then n component mass balances of this will give rise to n equations, one for each component, i.e. if one also formulates a total mass balance, only *n* equations are independent. The total mass balance equation will not have only generation or depletion terms; these will always be zero, as the total mass within the system will be constant.

- **Total Continuity Equations (Mass Balance)**

Development of the theoretical models is based on the fundamental physical and chemical laws. We will use the laws of conservation of mass, which are principles of chemistry since ion exchange is a chemical process.

The principles of conservation of mass when applied to a dynamic systems says

$$\begin{aligned} &\textit{Mass flow into system} - \textit{Mass flow out of system} \\ &\qquad = \textit{Time rate change of mass inside the system} \end{aligned} \tag{3.4}$$

The units of this equation are [mass per time]. Only one total continuity equation can be written for one system.

- **Component Continuity Equation (Component Balance)**

Unlike mass, chemical components are not conserved. If a reaction occurs inside a system the number of moles of an individual component will increase if it a product of the reaction or decrease if it is a reactant. Therefore the component continuity equation of the i^{th} chemical species of the system is,

$$\begin{aligned} &\textit{Flow of moles of jth component into the system} - \\ &- \textit{Flow of moles of jth component out of the system} + \\ &+ \textit{Rate of formation of jth component from chemical reaction} = \\ &\qquad = \textit{Rate of change of moles of the component inside the system} \end{aligned} \tag{3.5}$$

The units of this equation are moles of j^{th} component per unit time.

The flows in and out can be both convective due to bulk flow and molecular due to diffusion. One component continuity equation can be written for each component in the system. If there are n component in the system, there are n component continuity equations for any one system. However, the one total mass balance and these n components balances are not all independent since the sum of all the moles times their respective molecular weights equal to the total mass. Therefore a given system has only n independent continuity equations and usually the total mass balance and n-1 component balances are used if n is the number of components. The ion exchange process theoretical models are based on macroscopic descriptions, using lumped variables.

- **Mathematical Consistency**

Once the equations for a mathematical model has been written, it is usually a good idea in complex systems to make sure that the number of variables equals the number of equations. The next step is to check to see if the units of all terms are in consistence. This is a very trivial and obvious step that is most often forgotten. If the system is under-specified or over- specified, something is wrong with the formulation. This is achieved by what is so called *degree of freedom* made zero. The available solution techniques and tools must be kept in mind as the mathematical model is being developed.

- **Parameter Estimation**

In developing a model, whether by theoretical or empirical means certaion paremeters appear whose values must be specified before the model can be used to predict process behaviour. The parameters may be known a priori, available in the literature or estimated from independent experiment performed on a pilot plant, or in a laboratory. It is customary to obtain such parameter estimation using experimental data obtained directly from the physical process.

- **Verification**

An important but often neglected part of developing a mathematical model is to prove that the model describes the real process situation. Sometimes this cannot be done at the design stage because the plant (unit) has not been built. Fortunately there are either similar existing plants or a pilot plan from which experimental data can be obtained.

3.2.2.3 Procedure for Model Development

The physical laws governing the process are analyzed to produce modelling equations, which are usually complex and non-linear and may involve partial differential or integral equations. This model is usually simplified to a form which is easily handled, but that still retains the essential features of the process. At this point the equation structure of the model is fixed. Next those model parameters that are completely unknown or not known previously enough are estimated using process data taken from dynamic input/output of the experiment performed on the actual process. It is important to be mentioned that various types of errors are introduced at each stage of the model development.

The stages in development of the theoretical process model are shown on fig.3.1. These steps will be followed for ion exchange process model development.

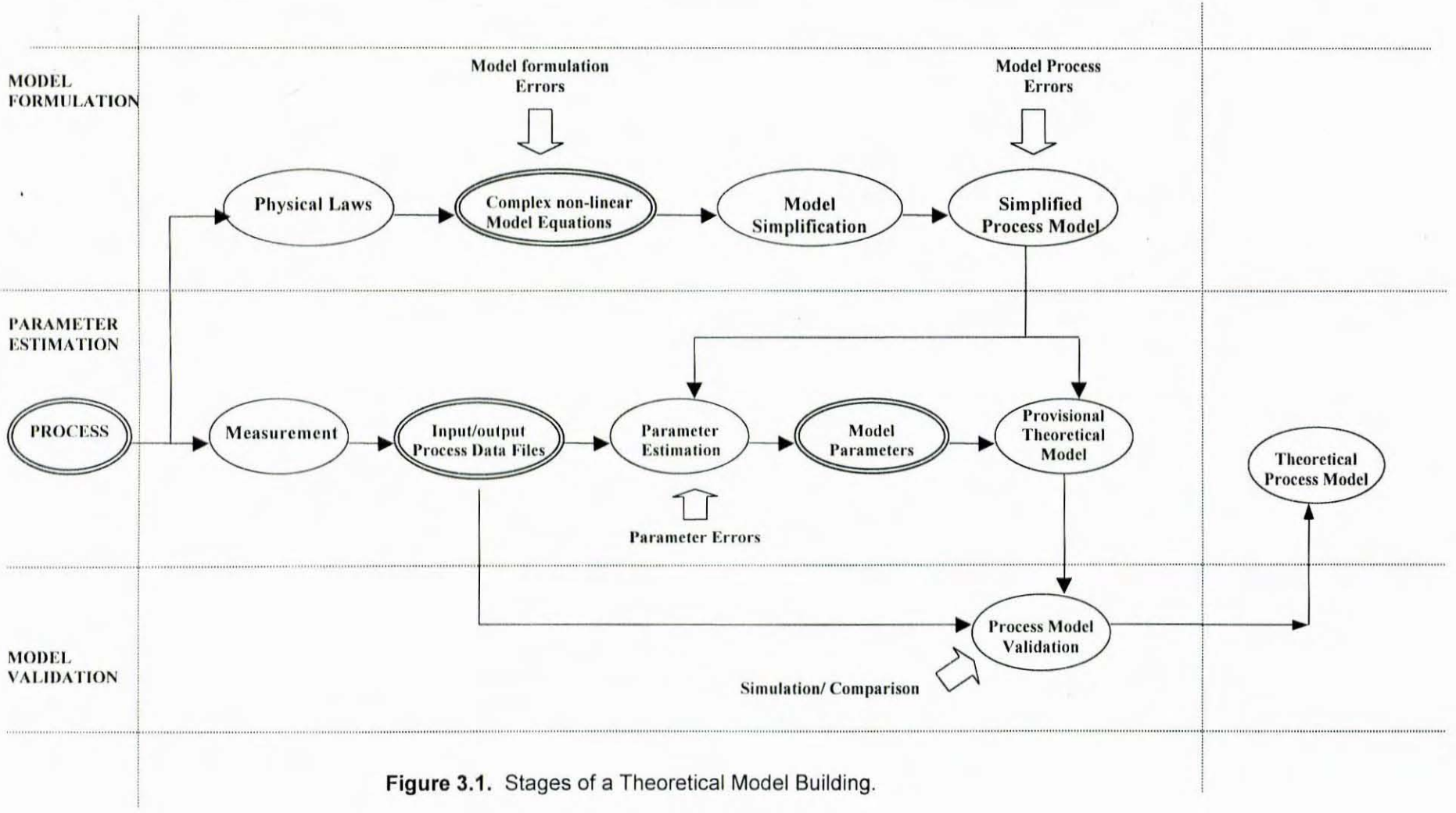


Figure 3.1. Stages of a Theoretical Model Building.

3.3 PARAMETER ESTIMATION

The process of confirming a model using experimental data is called *parameter estimation.* Those model parameters that are completely unknown or not known previously enough are estimated using process data taken from dynamic input/output experiments performed on the actual process (Norton, 1986).

In principle, experimental data can be used to check how well does it agrees with predictions from the theoretical model. There are two sets of data available:

1) real process and
2) model data

If the output of the model compares favourably with the experimental output the mathematical description is then said to be appropriate. If the output does not agree perhaps a revision of the model or another approach should be considered. When the process being evaluated cannot be tested in a fully operational manner because of some reasons, tests can then be done on some approximation of the system or pilot plant or under modified operation conditions. Some limitations might occur such as the availability of data and accuracy of data. The success of the process analysis depends heavily on the basic information available. After setting up the model the major task is to evaluate the parameters in the model on the basis of experimental data. The accuracy with which parameters must be known depends to some extent on their influence in the overall process (Davis, 1984; Himmelblau & Bischoff, 1968; Johansson, 1993; Norton, 1986).

3.3.1 Experimental Model Building

In modelling the behaviour of a dynamic process the main objective is that of obtaining a functional relationship between various variables of the process. The underlying belief is that a true but unknown fundamental relationship dictated by natural laws exists between the process variables. Modelling exercise is directed towards discovering this relationship. It is impossible to portray perfectly all the details of the actual process behaviour in mathematical form and therefore application of the laws of nature are assumed to determine the behaviour of the process. Development of rigorous theoretical models may not be practical for complex processes if the model requires a large number of differential equations with a significant number of unknown parameters (e.g. physical or chemical properties). An alternative approach is to develop an empirical model directly from the experimental data. One common approach is to use optimization theory to derive *least-squares estimates* for the model parameters (Box and Draper, 1987).

In the process of modelling, empirical model are used to approximate the unknown functional relationship between the process variables by some mathematical functions (usually simple functions) using information gathered experimentally from the process. Because these models depend entirely on experimental information and experience the approximating functions obtained are known as empirical (based on experiments) models. Parameter estimation deals with comparison the theoretical model with empirical model to conclude if the theoretical model is correct or incorrect. There are several stages one has to go through in developing a process model starting from a physical system (process) and ultimately abstract a model that can be used as a surrogate (depute) to the process.

In control engineering, model–building from measurements on a dynamical system is known as *identification*. Identification is the process of constructing a mathematical model of a dynamic system from observation and prior knowledge (Johansson, 1993).

There are three main groups of methods for identification of dynamic systems:

- Classical methods of identification using impulse, step and sine wave testing,
- Identification based on correlation functions and
- Least squares model fitting.

In the first two groups of methods for identification the influence of the noise on the parameter estimate is done by time averaging. The justification is essentially statistical. The method relies on the zero–mean noise–dependent terms affecting the estimates becoming negligible if averaged over a long enough interval. The least square model fitting uses a different approach. It is necessary to find a model of specified structure, which fits the observations best according to a deterministic measure of error between model output and observed output, totaled over all the observations. This method has two possibilities not real time estimates and real time estimates (Carnahan, Luther & Wilkes, 1969; Coleman, Branch & Grace, 1999; Unbahanen & Rao, 1997, 1990). In the dissertation only not real time is discussed.

3.3.2 Identification Problem Formulation

Important problem, connected to the implementation of the control in real time is the problem for *identification*. The problem for identification is formulated as follows:

- Built an optimal according to some criterion model of the process from the real data and its input and output variables.

There are two main groups of problems for identification. They depend on the apriori information we have about the process. They are:

- Identification in narrow meaning
- Identification in wide meaning

Identification in narrow meaning is connected with parameter estimation from data of input and variables obtained in the conditions of real action of the process. Here the

structure of the (system) process is known and the structure of the model is known. The apriori information about the process is sufficiently big.

Identification in wide meaning give answers to the following questions

1. selection of the structure and class of the model,
2. estimation of the linearity and time variety of the process and acting variables and
3. selection of state variables.

Under classical approach to the modelling, identification is done usually only once and used for a long time. In modern control systems the control strategies need to be continuous in real time modelling and identification of the process, in order to give possibility of optimal adaptive control in the conditions of uncertainty. This is called *identification on-line* (Ramasamy, Deshpande, Tambe & Kulkarni, 1995; Sung, Han & Rhee, 1979).

Characteristics of the considered process for identification are:

- The model is represented using differential equations of first order, or the process is described in state space.
- The models of the ion exchange are nonlinear in accordance with the state and control variables and linear according to the parameters.

3.3.2.1 Problem Formulation

Given, measured vector of state of variables $z(t)$ and the input signal $u(t)$. $z(t)$ is subjected to noises of measurement $v(t)$, and

$$z(t) = h\left[y(t), u(t), v(t), p(t), t\right], \tag{3.6}$$

where p – is the vector of the unknown system parameters.

Given equation for description of the state space vector

$$\frac{dy(t)}{dt} = f\left[y(t), u(t), w(t), p(t), t\right], \tag{3.7}$$

where $w(t)$ – is a vector of external disturbances (noise).

Find, on the bases of data of measurement $z(t)$, (3.6) such parameters of the model (3.7), that some criterion for the exactness between $z(t)$ and $y(t)$ is minimized.

$$J = \int_0^t \left\| z(t) - y(t) \right\|_Q^2 dt \to min\,. \tag{3.8}$$

The common scheme of the identification process is shown on the figure below (Fig. 3.2).

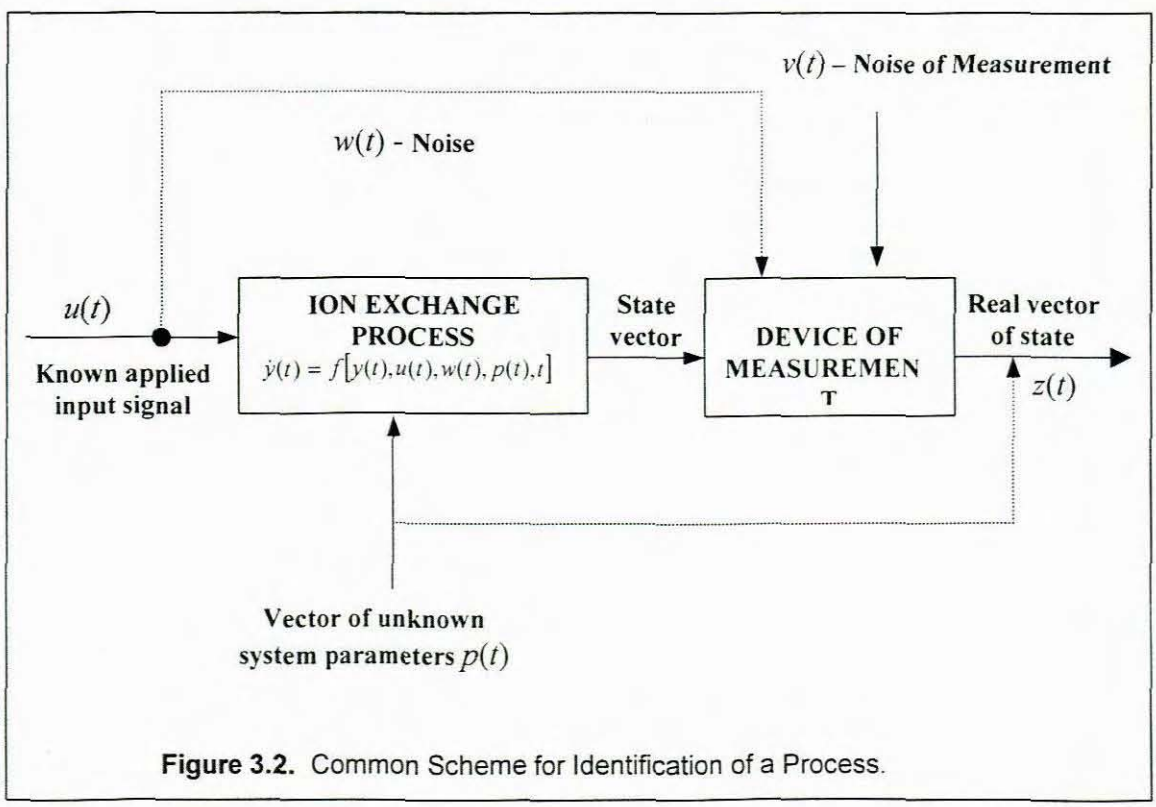


Figure 3.2. Common Scheme for Identification of a Process.

3.3.2.2 Identification Formulation

The problem for identification can be solved in different ways:

- In Real Time – where parameters of the model are calculated at every sampling interval during real work of the process.
- Not In Real Time – the problem for identification is solved once and the calculated parameters are used for a long time during the real work of the process.

The latter is considered in the dissertation.

Different methods are used for solving problems for identification differential approximation, gradient methods, search methods, method of quasilinearization and method using the sensitivity functions (Davis, 1984, Sinnot, 1997; Yu & Liu, 1999).

- **Differential approximation**

The differential approximation method is often used for model development. It is simplest when the process is linear, described by linear differential equation or by equation with linear parameters.

The approach is based on the calculation of the derivatives of the state vector in different moment of time t_1, t_2 , …, t_K and on representation of the model as a linear function according to the vector of parameters p.

$$\frac{dy(t)}{dt} = f_1\left[y(t), u(t), w(t), t\right]p \,. \tag{3.9}$$

Then the values of $u(t)$ and $y(t)$ can be determined – measured at the points t_1, t_2,…, t_K. The equation can be written in the following way

$$\begin{bmatrix} \hat{\dot{y}}(t_1) \\ \hat{\dot{y}}(t_2) \\ \hat{\dot{y}}(t_3) \\ \vdots \\ \hat{\dot{y}}(t_K) \end{bmatrix} = \begin{bmatrix} Matrix\ A^*, containing \\ known\ nonlinear \\ function\ y\ and\ u \\ at\ the\ moments \\ t_1, t_2, \ldots, t_K \end{bmatrix} \cdot \begin{bmatrix} p_1 \\ p_2 \\ p_3 \\ \vdots \\ p_m \end{bmatrix}, \tag{3.10}$$

where m – is the number of parameters,

$\hat{\dot{y}}(t_K)$ – is the estimate of $x(t_i)$, calculated from the model, $k = \overline{0, K}$.

If the vector of parameters p is selected in such a way, that the integral quadratic error between the measured values of $\dot{y}(t_k)$ and calculated from the model $\hat{\dot{y}}(t_k)$ is minimal, then the equation for $\hat{p}_1$ determination can be written in the following way

$$\hat{p} = \left[A^{*T} A^*\right]^{-1} A^{*T} \dot{Y}. \tag{3.11}$$

The method of differential approximation is very simple, but it has some drawbacks:

1) It needs finding derivatives of the state vector (measured) according to the time. Derivatives determination is difficult because of the non-stability of the numerical differentiating.
2) When there is noise after the procedure the result is close to the differential of the state, not to the state.
3) The method is not applicable when we can not measure all components of the vector of state in the process.

Good example for application of this method is the identification of the process in.

- **Gradient Methods**

There are many variants of the gradient methods. Common problem of identification is based on these methods:

- The measured data is supposed to be given by the nonlinear equation

$$z(t) = h\left[y(t), u(t), v(t), p_2(t), t\right], \tag{3.12}$$

where p_2 – is not known parameters of the system,

$z(t)$ – measurement vector – it is the state vector plus the noise,

$v(t)$ – is the error of the measurement,

$\dot{y}(t) = f\left[(y(t), u(t), w(t), p_1(t), t\right] \rightarrow$ the state differential equation,

$w(t)$ – vector of external disturbances.

Let these equations be given in the discrete format

$$z(k) = h\left[y(k), u(k), v(k), p_2(k), k\right], \tag{3.13}$$

$$y(k+1) = y(k) + \Delta t f\left[y(k), u(k), w(k), p_1(k), k\right], \tag{3.14}$$

$$\dot{y}(t) = \frac{y(k+1) - x(k)}{\Delta t}. \tag{3.15}$$

It is necessary to estimate the numeric values of the parameters, p in such way that $y(t)$ is equal to $z(t)$ with some error under the influence of the control $u(t)$ for the period $t_0 < t_i > t_K$. Comparison of $y(t)$ with $z(t)$ is possible to be done, introducing scalar criterion of the error J. It is equal of the sum of the differences between the output of the model and the real process under the given $u(t_i)$ in points t_i $t_0 < t_i < t_K$

$$J = \sum_{i=0}^{K} H^* \left[y(t_i) - z(t_i)\right] \tag{3.16}$$

where H^* – is the scalar positively determined matrix of the error which usually is determined as a sum of square power of the components of the vector of the error.

The structure of the method on the bases of the gradient calculation is shown in Fig. 3.4. Different search algorithms can also be used. The simplest iterative gradient procedure for improving of the initial estimations of the vector p – is the method of the fastest slope. Direction of the fastest slope is opposite to the direction of the gradient and at the initial

point coincides with the direction in which the error criterion reduces fastest under an infinitely small variation of the vector of parameters.

Practical rule: if the necessary exactness can not be reached for the number of steps equal to the number of parameters, than it is necessary to do for only one step the method of the fastest slope, and to continue with the method of conjugate gradients.

The most effective is the gradient method of David-Fletcher-Paul. The best quality of convergence has the method of conjugate gradients. This method can find minimum of positive definite quadratic function for number of steps smaller or equal to the number of parameters.

- **Methods of Direct Search**

The direct method is useful for the cases where the gradient methods are not useful. For these methods it is not necessary to know the value of the derivatives. The direction of minimum is determined on the bases of (consequently) step by step calculation of the criterion. As usual the gradient methods, and methods using the second derivatives have faster convergence than direct methods, but the calculation of the first and second derivatives require too much time.

The algorithms of the direct search are not fast but can be used for simple problems. For minimization of the nonlinear functions, the following methods are used: (Rosenbrock & Story, 1966)

- method of Houck-Jivs,
- simplex method,
- method of Rosenbrock, and
- method of Paul.

- **Methods of Quasilinearization**

The method of quasilinearization together with the method of least squares is a commonly used strategy for more exact estimation of the parameter, in the cases when their approximate values are known previously. The model of the process be described by

$$\dot{y}(t) = f[x(t), u(t), p(t), t], \quad y(0) = y_0 \tag{3.17}$$

If the right-side of the equation is represented by the Taylor series with the linearized expression, than for finding of p, – the method of successive approximation can be used as the right side of the equation is linear for every iteration. Such method has the quadratic convergence.

The problem for parameter estimation in the vector differential equation can be transformed into a problem for estimation of the initial conditions – if the constant parameter of the model is considered as function of the time, then an additional system of differential equation is established. This system is added to the system equations of the process. Then the initial model is transformed into another form with these equations included.

The method requires very good initial point. It is possible first the method of a differential approximation to be used to reach a good initial point and then the method of quasilinearization. The drawback of this method is the requirement of a lot of memory.

- **Methods using the Sensitivity Functions**

They are evristic methods (not fully analytical, some results used are from experiments), used for identification and for analysis of the models. Let the process be given by

$$\frac{dy(t)}{dt} = \dot{y}(t) = f[y(t), u(t), t, p], \quad y(0) = y_0 . \tag{3.18}$$

The matrix function of sensitivity of the output to the parameters is determined by

$$\lambda^* = \frac{\partial y}{\partial p}. \tag{3.19}$$

Using both equations it can be written as

$$\dot{\lambda}^*(t)\frac{\partial^2 y}{\partial p \partial t} = \frac{\partial f}{\partial y}\frac{\partial y}{\partial p} + \frac{\partial f}{\partial p}, \tag{3.20}$$

$$\dot{\lambda}^* = \frac{\partial f}{\partial y}(\lambda^*)^T + \frac{\partial f}{\partial p}, \quad \lambda(0) = \frac{\partial y_0}{\partial p}. \tag{3.21}$$

After integrating the last equation, the matrix function of the sensitivity is obtained. This function can be used for identification of the models of the process.

3.3.3 Least Squares Estimation Formulation

It is necessary to find the values of the coefficients in a given model which minimizes the sum of the errors between the model output and the observations of the output:

- Least squares estimates of the coefficients.

It is possible to consider other measures of fit than output error squared, but this measure has two big advantages:

- Large errors are heavily penalized: an error twice as large is four times penalized and
- Mathematical tractability: the formula giving the least-squares estimates is obtained by quite simple matrix algebra, and the estimates are computed as a solution to a set of linear equations.

In the dissertation the unknown parameters are estimated using least squares methods, when the measurements are linear in the parameters to be estimated, the least squares estimates of constant unknown parameters can be obtained using a one step procedure. No a priori estimates of the unknown parameters are required. For dynamic systems with

measurements nonlinear in the parameters, iterative methods are required as well as initial estimates of the unknown parameters.

3.3.4 The Basic Method of Least Squares

The Gauss's principle of least squares is applied. The consideration is restricted to problem with constant parameters. The method of least squares is found central to the solution of a large family of estimation problems (Junkins, 1991).

3.3.4.1 Linear Estimation

Suppose there is a set of measured values of the $\tilde{z}_j$ process $z(t)$, taken at known discrete instant of time t_j

$$\{\tilde{z}_1, t_1; \tilde{z}_2, t_2; \ldots; \tilde{z}_K, t_K\}, \tag{3.22}$$

and a proposed mathematical model

$$z(t) = p_1 y_1(t) + p_2 y_2(t) + \ldots + p_m y_m(t) = \sum_{i=1}^{m} p_i y_i(t), \quad K \geq m, \tag{3.23}$$

where $\{y_1(t), y_2(t), \ldots, y_m(t)\}$ – are a set of independent specified basis functions,

p_i – are constants whose numerical values are unknown.

It seems altogether reasonable to select the optimum p–values based upon a measure of "how well" the proposed model (3.23) predicts the measurements (3.22). Any particular choice of p–values $\{\hat{p}_1, \hat{p}_2, \ldots, \hat{p}_m\}$ when substituted into (3.23) will generally not yield y–values in exact agreement with measurements $\tilde{z}$. The disagreement between observed and computed z–values can stem from one or more of three possible sources:

- Incorrect choice of p–values,
- Observation errors and
- Modelling errors, i.e., the actual process being observed may not be accurately modelled by equation (3.23).

Introducing $\{e_1, e_2, \ldots, e_K\}$ as symbols for residual errors after the solution associated with any choice for $\{\hat{p}_i\}$, then $\{\tilde{z}_i\}$, $\{\hat{p}_i\}$ and $\{e_i\}$ must satisfy the equation

$$z_j = \sum_{i=1}^{m} \hat{p}_i y_i(t_j) + e_j, \qquad j = 1,2,\ldots,K \tag{3.24}$$

for every moment of time t_j, $j = 1,2,\ldots,K$

This equation can be written in matrix form.

$$\tilde{Z} = Y\hat{P} + E \tag{3.25}$$

where $\tilde{Z} = [\tilde{z}_1, \tilde{z}_2, \ldots, \tilde{z}_K]^T$ is the observed (measured) vector

$E = [e_1, e_2, \ldots, e_K]^T$ is the residual error vector

$\hat{P} = [\hat{p}_1, \hat{p}_2, \ldots, \hat{p}_m]^T$ is the estimated vector

$$Y = \begin{bmatrix} y_1(t_1) & y_2(t_1) & \ldots & y_m(t_1) \\ y_1(t_2) & y_2(t_2) & \ldots & y_m(t_2) \\ \vdots & \vdots & \vdots & \vdots \\ y_1(t_K) & y_2(t_K) & \ldots & y_m(t_K) \end{bmatrix}$$

– is the matrix of the input functions at different moments of time.

In the absence of model errors, the true z-values and true p–values satisfy equation (3.23) exactly. The measured z–values $\tilde{z}_j$, the measurement errors v_j (unknown), and the true p–values (also unknown) satisfy.

$$\tilde{z}_j = \sum_{i=1}^{m} y_i(t_j) + v_j, \qquad j = 1,2,\ldots,K, \tag{3.26}$$

or a matrix form

$$\tilde{Z} = YP + V \tag{3.27}$$

where $Y = [y_1, \ldots, y_m]^T$ is the true *Y*–value vector

$V = [v_1, \ldots, v_m]^T$ is the measurement error vector

Equations (3.25) and (3.27) are identical if $\hat{P} = P$, and if the assumption of zero model errors is valid. Both of these equations are commonly referred to as the "observation equations".

Gauss's principle of least squares selects, as an optimum choice for the unknown parameters, the particular $\hat{p}_i$ which minimizes the sum squares of the residual errors.

$$J = \sum_{j=1}^{K} e_j^2 = E^T E \tag{3.28}$$

From equation (3.24) $E = \tilde{Z} - Y\hat{P}$, and then equation (3.28) is written as

$$J = E^T E = \left(\tilde{Z} - Y\hat{P}\right)^T \left(\tilde{Z} - Y\hat{P}\right) = \tilde{Z}^T Z - \tilde{Z}^T Y\hat{P} - \hat{P}^T Y^T \tilde{Z} + \hat{P}^T Y^T Y\hat{P} \tag{3.29}$$

Observe that each term of equation (3.29) is a scalar equals its transpose.

$$J = \tilde{Z}^T Z - 2Z^T Y\hat{P} + \hat{P}^T Y^T \hat{P} \tag{3.30}$$

It is necessary to find the $\hat{P}$ which minimizes *Y*. To find it, first the necessary conditions for optimality have to be found and satisfied,

$$\nabla_{\hat{P}} J = \begin{bmatrix} \dfrac{\partial J}{\partial \hat{p}_1} \\ \vdots \\ \dfrac{\partial J}{\partial \hat{p}_r} \end{bmatrix} = -2Y^T \tilde{Z} + 2Y^T \hat{P} = 0 . \tag{3.31}$$

The sufficient conditions are

$$\nabla_p^2 J = \left\{ \frac{\partial^2 J}{\partial p_i \partial p_j} \right\} = 2Y^T Y . \tag{3.32}$$

$2Y^T Y$ must be positive definite in order *Y* to have minimum.

From equation (3.31) this equation can be written

$$2Y^T Z = 2Y^T \hat{P} . \tag{3.33}$$

From here

$$\hat{P} = (Y^T Y)^{-1} Y^T \tilde{Z} . \tag{3.34}$$

The used least square criterion implicitly places equal emphasis upon the model agreeing with each of the observed z-values. For the common event that the measurements are made with unequal precision, weights can be selected for each measurement. These weights are inversely proportional to the measurements estimated precision. One satisfy choice for weights is the reciprocal of the measurement variance, as

$$J = \sum_{j=1}^{K} w_j e_j^2 , \tag{3.35}$$

where $w_j = \dfrac{1}{\delta_j^2}$,

δ_j – is the measurement variance.

More generally (3.33) can be written in the form

$$J = \sum_{j=1}^{K} \sum_{i=1}^{K} w_{ij} e_i e_j = E^T W E , \tag{3.36}$$

where W – is a symmetric, positive definite weighting matrix.

For the criterion (3.36) the solution is

$$\hat{P} = \left(Y^T W Y\right)^{-1} Y^T W \tilde{Z} . \tag{3.37}$$

3.3.4.2 Nonlinear Estimation

Most real world estimation problems are nonlinear. They can be solved by successive approximation procedure. The most widely used is the least square differential correction.

The model in this case is represented by

$$z_j = F_j(p_1, p_2, \ldots, p_m), \quad j = 1,2,\ldots,K, \quad K > m , \tag{3.38}$$

where F_j – are K arbitrary independent functions of the unknown parameters p_i. It is supposed that $F_j(p_i)$ at their first partial derivatives are single-valued and continuous.

It is necessary to find the particular vector of p–values

$$\hat{P} = [\hat{p}_1, \hat{p}_2, ..., \hat{p}_m]^T, \tag{3.39}$$

which minimizes the weighted sum of squares of squares of the residuals

$$J = \sum_{j=1}^{K} \sum_{e=1}^{K} w_{je} \Delta z_j \Delta z_e = \Delta Z^T W^* \Delta Z, \tag{3.40}$$

$$\text{where } \Delta z_j = \tilde{z} - F_j(p_1, p_2, ..., p_m), \quad j = 1,2,...K, \tag{3.41}$$

$$\Delta z = [\Delta z_1, \Delta z_2, ..., \Delta z_K]^T.$$

In the most practical problems J cannot be directly minimized by application of ordinary calculus. Explicit closed form solution could not be obtained. For this reason, attention is directed to construction of a successive approximation procedure which is designed to converge to accurate least square estimate, given approximate starting values. Assume that the current estimates

$$P_c = [p_{1c}, p_{2c}, ..., p_{mc}]^T,$$

of the unknown parameter vectors are available. Whatever the unknown objective P–values $\hat{P}_i$ are, they are related to their respective current estimates P_{ic} by also unknown set of corrections Δp_i, as

$$\hat{p}_i = p_{ic} + \Delta p_i, \quad i = 1,2,..., K, \tag{3.42}$$

If Δp are sufficiently small, it may be possible to solve for approximation to them and thereby obtain improved estimate of p_i from (3.42). The current residuals corresponding to the current p–estimates p_c are calculated from (3.41) as

$$\Delta Z_{jc} = \tilde{Z}_j - F_j(P_{1c}, P_{2c}, ..., P_{mc}), \quad j = 1,2,..., K. \tag{3.43}$$

For small changes (corrections), the linearly predicted residuals ΔZ_{jP} after the correction follow from Taylor series of (3.25) about P_c as

$$\Delta z_{jp} = \Delta y - \left(\sum_{i=1}^{m} \left. \frac{\partial F_j}{\partial p_i} \right|_c \right) \Delta p_i, \quad j = 1,2,\ldots,K. \tag{3.44}$$

In the matrix notation

$$\Delta Z_p = \Delta Z_c - Y \Delta P, \tag{3.45}$$

where $\Delta Z_c = \left[\Delta Z_{1c} \quad \Delta Z_{2c} \quad \ldots \quad \Delta Z_{mc} \right]^T$, is the current the residual.

$\Delta Z_p = \left[\Delta Z_{1p} \quad \Delta Z_{2p} \quad \ldots \quad \Delta Z_{mp} \right]^T$, is linearly predicted residual after the correction ΔP.

$Y = \left(\left. \frac{\partial F_j}{\partial P_i} \right|_c \right) = K \times m$ matrix of partial derivatives, evaluated with current P–estimates P_c.

The objective is to minimize the criterion (2.41). The local strategy for determining the approximate corrections (differential corrections) Δ_{pi} is to select the particular corrections which lead to the minimum sum of squares of the linearly predicted residuals

$$J_P = \sum_{j=1}^{K} \sum_{e=1}^{K} w_{je} \Delta z_{jp} \Delta z_{ep} = \Delta z_P^T w \Delta z_P . \tag{3.46}$$

It is supposed that minimization of (2.46) is equivalent for small residuals to minimization of (2.41).

Substitution of equation (3.29) into equation (2.45) gives

$$J_P = \Delta Z_P^T W \Delta Z_P = \left(\Delta Z_c - Y \Delta P \right)^T W \left(\Delta Z - Y \Delta A^* \right), \tag{3.47}$$

Equation (2.44) is analogous to equation (3.29). Thus any algorithm for solving the weighted least squares linear problem directly applies for solving ΔP in (3.47) and

$$\Delta P = \left(Y^T W Y \right)^{-1} \left(Y^T W \Delta Y_c \right). \tag{3.48}$$

The Gauss's least square differential correction algorithm can then be summarized in the following way:

1) Input the measurements, and weights W.
2) Input the starting estimates of parameters P_c.
3) Compute current value of the measurement function from the model
   $Z_c = F(P_c)$.
4) Compute matrix of partial derivatives
   $Y = \left(\frac{\partial F_j}{\partial p_i} \right)\Big|_c$ in point c (current).
5) Form measured minus derivatives residuals
   $\Delta Z = \tilde{Z} - Z_c$ and their weighted sum square
   $J_c = \Delta Z^T W \Delta Z$
6) Determine the correction vector which minimizes the predicted residuals sum squares (2.47).
7) Apply the corrections from step (6) replacing previous current parameter estimates according to
   $P_c = P_c + \Delta P$. Return to step (3).
8) Check the convergence of the algorithm.
   Set $\hat{P} = P_c$ if the criterion for stop of the iterations is fulfilled.

The drawbacks are:

- The convergence is guaranteed only under rather strict requirements on the functions and their two partial derivatives,
- The starting solution is necessary to be close to the optimal one and
- Numerical difficulties can appear in solution for the correction Δp.

Other mathematical programming methods can be used in minimizing the sum of square residuals, as the methods of gradients or methods of steepest descent. The essential

difference between the approaches is that the gradient corrections are based upon linearizing the prediction of the sum square of residuals, whereas, the Gaussian *differential correction algorithm is based upon linearization of the equations which model* measurements.

The correction in the gradient algorithm is calculated as

$$\Delta P = -\alpha G \tag{3.49}$$

where $G = \begin{bmatrix} \frac{\partial J}{\partial p_{1c}} \\ \vdots \\ \frac{\partial J}{\partial p_{mc}} \end{bmatrix}$ is the gradient of the criterion according to the parameters.

and α – is the step of the gradient procedure.

Usually the gradient algorithms need more iterations, but they are numerically more stable.

The gradient type of algorithm is used to solve the problem for parameter estimation of the ion exchange process model, which is a dynamical model.

3.3.4.3 Dynamic Models

The mathematical models of the physical processes embodies one or more differential or difference equations. The ordinary differential equations will be considered here. In a significant part of the real applications, it is possible to obtain explicit algebraic solutions of the system of differential equations. Then this results in linear or nonlinear parameter estimation of the steady state problems. When analytical solutions can not be obtained some approximative methods could be developed, like the gradient methods (Smith, 1965).

3.4 MODEL VALIDATION

Before proceeding to use the process model for control, it is essential to evaluate how closely the model predicts the behaviour of the physical system it is supposed to represent. This is usually accomplished by testing the model against additional process data in the context of the process experience. Analysis cannot be completely isolated from modelling. A mathematical model represents a concise statement of the hypothesis. The model can be verified in two ways, experimentally or testing. Once the validity of chosen component models is satisfied, these models can be used to predict the performance of the system in question. Predicting the performance of the system from a model is called ***analysis***. A model is validated by answering the following questions:

1) Is the model credible?
2) Does the model work?

The first question supposes some background information and wants the model to confirm this data. A direct and revealing test of whether a model works is to try it on records different from those it was estimated from. However, there could be a well noticeable difference between the performance of the model on two sets of records, even if the model were optimal in some statistical sense and well structured. One might consider testing the significance of the difference in performance by comparing statistics from the new records to the previous records. The possible tests for model validation are:

1) the records of data before analysis.
2) estimation of parameters in light of background knowledge.
3) fitting the model (to be validated) to the records of data (using residuals).
4) the behaviour of the model as a whole, if it does fit in the data in an accepted way.

Checks on records of data – the flow of data has to be smooth which means that the sampling interval is selected such as to prevent aliasing. Checks on parameter estimates, - values of estimates are estimated again according to the knowledge gained previously about the process or according to some of theoretical calculations of the parameters.

In postulating the physical phenomena and applying their laws, approximations are to be made to the model equations and this introduces errors. These are known as model formulation errors. Also in simplifying the model to be numerically and mathematically tractable approximations that are made introduce errors. These are known as model process errors. Experimental measured errors are errors introduced during collection of process data which are both systematic calibration errors and random experimental errors. In addition to the effects of experimental measurement errors on estimated parameter, there are other sources of parameter estimation errors. For example, inadequate experimental design may produce data sets that intricacy looks for reasonable parameter estimates. Choosing a parameter estimation algorithm that is inappropriate for the data set may also result in poor parameter estimates, all such errors are known as parameter estimation errors (Junkins, 1991; Westphal, 1995).

- **Pre-Estimation Checks**

Valuable information can be gained by looking at a plot of the records as soon as they are received. A time-invariant nonlinear model might be preferable if enough in information is known to chose its form. Breaks in records, particularly due to oversight are sometimes repaired by interpolation without informing the recipient. Quantities affected by all parameters such as steady state gain can be calculated easily from parameter estimates if necessary and checked against background knowledge.

Before comparing residuals and innovations, model deficiencies can be detected online or offline by noting large and time-structured residuals or innovations. The obvious way to asses the reliability of parameter estimates is to inspect their estimated covariance in algorithms which calculate it (e.g. extended least squares).

- **Post-Estimation Checks**

The nature of the time variation in a simple model is a good pointer to the extra or modified features of the final model. The estimation of a time-varying model is not only interest when the final model will be time-varying but it is also valuable as a bridge between a very first attempt model and a refined and extended time-invariant final model. The detailed variation of parameter estimates or desired quantities such as (unit impulse response and steady state gain) can be checked against qualitative knowledge of physical underlying dynamics. In a constant parameter model, deficiency in a model structure may show up clearly in unlikely parameter value, although it is not always obvious (Norton, 1986).

A straight forward plot of the residuals or innovations can say a great deal about adequacy of the model. That is, a good model predicts all the systematic part of the output, leaving an unstructured innovation sequence.

A severe and informative test of a model is to simulate it on records. Deficiencies in model structure and poor estimates or dead time give rise to obvious systematic errors in the simulation-mode output sequence. Raising model order may improve the simulation (Tomich, 1970). The following are worthwhile checks after an estimate run:

- isolate large residuals which may reveal instrument errors
- short periods of large and highly structured or anomalously (deviation) small residuals, which may point to show up doctored records
- abrupt and unexpected changes in the parameter estimates or residuals which may point to unrecorded, incidents such as feed change, control action.
- input features with no apparent output consequence or output features with no apparent cause, as shown by large residuals over short periods suggesting that mote extensive or better measurements or a higher sampling rate may be required
- periodicity, specialized models for periodic phenomena may be a necessity.

Such features are effectively brought out when using simulation runs.

3.5 MATHEMATICAL MODELLING OF AN ION EXCHANGE PROCESS

Modelling an ion exchange process for water purification has been achieved using both theoretical and empirical models. The theoretical model was built based on the mass laws (conservation of mass), the mass balance and the component balance, equations (3.4), (3.5). The mass balance deals with the total mass transfer during the reaction in the process. The component balance deals with each species involved in the process reactions, i.e. the sodium ion (Na^+) from liquid phase exchanging with hydrogen ion (H^+) in the resin phase. From these laws of conservation of mass, a set of equations that deal with mass balance and another set that deals with component balance are developed to form a model that identifies the process. A model cannot be assumed to be working perfectly if it there is no way of validating the model. The developed theoretical model in this case is validated using experimental tests (empirical model). The process of validating the model is based on parameter estimation by comparing experimental data with theoretical data and then noting the difference between the two using the error generated during the comparison. The aim is to minimize the error such that the difference between the two models is almost non-existence. There are different methods used to achieve this, for this process the least-squares method has been suggested.

The purpose of a model are scientific research, prediction and control of systems, state estimations, diagnostic and inadequacies and simulation of systems. Prediction and control model is used for system design and prediction by a model is more directly involved in two control techniques – feed-forward and self-tuning control. State estimation model is used for tracking of variables which characterize some dynamical behaviour of a system (Höegfeld, 1991).

How the problem is solved. The model is developed for the purpose of control and optimization of the process. In order to reach this aim the following must be done:

- Characterization of the CCIX as an object of control. Input and output variables, control and state variables are determined.

- Mathematical model describing stage-wise process is developed and applied to the CCIX process.
- Calculation of mathematical model parameters on the basis of characteristics of columns, flows and resin is done.
- Experiments for obtaining real process data are performed, input variables be determined and then develop a method for measurement of stage concentration.
- Software programs are written using LabVIEW for analysis and calculation of model parameters.
- Fully mathematical model based on the experimental data be simulated and compared to the theoretical model.
- Software for parameter estimation is incorporated in LabVIEW for real time process control and optimization to perform the actual physical control of the plant (by providing relevant control action).

3.5.1 Characterization of Ion Exchange Process as an Object of Control

3.5.1.1 Pseudo-Steady State Mode of Operation

Ion exchange is based on mass transfer from one place to another due to the existence of a driving force arising from differences in chemical potentials. In practice, this requires the intimate contact of the phases for some period of time, during which equilibrium is approached as mass transfer progresses.

A stage is a unit in which the contacting occurs and where the phases are separated physically. A single stage process is one in which the contacting is done once and, if equilibrium actually is achieved, further classification as an ideal or theoretical stage is appropriate. Because of economic considerations, the contacting time in a particular

stage may not be long enough for equilibrium to be reached; the fractional approach to equilibrium is a measure of the mass transfer efficiency of the process.

When multiple stages are used, some consideration of the pattern of contacting of the two phases can be done. It can be:

- Cocurrent contacting, where the inlet stream from one phase is mixed with the inlet stream of another phase. It is used to promote better mixing.
- Countercurrent contacting, the inlet stream for one phase is mixed with the outlet stream of the other phase. It is used to maximize driving forces throughout the system. The highest mass transfer efficiency can be achieved by this system.

The considered process is a countercurrent one and a periodic one. It exhibits properties which may be of great practical value. The theory of periodic processes includes the theory of steady state and batch processes, Since these two types can be regarded as special cases of periodic processes. Formally, periodic processes are similar to some types of steady state recycle processes, because a cycle in time and a cycle in space lead to very similar mathematical representations (Horn and Lin, 1967).

The dynamical behaviour of many chemical systems can be represented by a set of differential equations.

$$\frac{dy_1}{dt} = f_i(y_1 \dots y_N,\ F_1 \dots F_m), \quad i = \overline{1,N}, \tag{3.50}$$

$$y_i(0) = y_{0i}$$

where y – is the state, $y \in R^N$ and

$F \in R^m$ – is the control vector.

If y_{i0} are known and the control variables are given functions of time, the state of the system can be calculated as a function of time. Of special interest to industry are steady state solutions – that is, solutions of the equations which correspond to time – independent state and control variables

$$f_i(y_1 \dots y_N,\ F_1 \dots F_m) = 0, \quad i = \overline{1,N}. \tag{3.51}$$

There may be none, one or more than one steady state solutions of (3.51) belonging to one constant control. The steady state may be unstable or stable. In the latter case this steady state is approached by the system from appropriately chosen initial states under the constant control.

Another mode of operation is to use periodic control functions F(t), that is functions satisfying the relations

$$F_j(t+T) = F_j(t), \tag{3.52}$$

for any t, $j = \overline{1,m}$.

T is the period for which the control is constant. Obviously, the constant control is a trivial case of a periodic control, because it satisfies equation (3.51) for any T. If the system is subjected to a periodic control, possibly, the state variables, also become periodic functions of time.

If the state variables are periodic functions, and the period is the same as for the control variables, the process is called a *periodic process*. In this case, the following relations are satisfied

$$y_i(t+T) = y_i(t) \text{ for any } t,\ i = \overline{1,N}. \tag{3.53}$$

The initial condition in this case is

$$y_i(0) = y_i(T), \quad i = \overline{1,N}. \tag{3.54}$$

As in the case of steady state operation, there may be more that one periodic control, and each of the corresponding processes may either be stable or unstable. Such mode of operation is distinguished from others because, in a periodic process, both control sequence and output sequences are repeated again and again, and this of practical advantage.

Horn (1967) proved for countercurrent continuous processes that the steady state can be reached using periodic control for long enough time.

A necessary condition for pseudo-steady state is that the volume of resin transferred from each stage to the stage below must be the same for all stages (Dodds, Hudson,

Kershenbaum, & Streat, 1973). Furthermore, the continuous resin feed rate F_R to the top stage must satisfy an overall material balance for the column over the cycle to ensure no buildup or depletion of solids in the column. From equation (1.1)

$$(1-e_v)V_j = const,\ j = 1,2,\ldots,N,$$

$$(1-e_v)V_R d = F_R T. \qquad (1.1)$$

where d – is the fraction of solid holdup transferred per stage per cycle,

e_v – is the voidage of every stage (fractional liquid holdup),

F_R – is the resin flow rate,

V_R – is the resin volume,

V_j – is the volume of the stage,

T – is the cycle time.

3.5.1.2 Phase Equilibria

Most of the common separation methods used in the chemical industry rely on a well-known observation:

- when a multi-component two phase system is given sufficient time to attain a stationery state called equilibrium, the composition of one phase is different from that of the other. It is this property of nature which enables separation of fluid mixtures by ion exchange distillation, extraction and other diffusional operations.

For rational design of such operations it is necessary to have a quantitative description of how a component distributes itself between two contacting phases.

Since separation processes are based on the creation of composition differences within and between phases, a consideration of mass transfer principles is necessary to the analysis, design and control of such processes. It is combination of mass transfer principles with phase equilibrium relationships, energy considerations and system

geometry or configuration that permits proper description of the separation processes (Buttler, 1964; Davis, 1984) discussed in the dissertation.

It is necessary to provide quantitative description rate of species transport within phases under the influence of composition differences.

Three basic concepts are used for development of the model:

- conservation laws,
- rate expressions and
- equilibrium thermodynamics.

They express the mass transfer principles

1) Mass transfer occurs owing to a concentration gradient or difference within a phase.
2) The mass transfer rate between two phases is proportional to their interface area and not the volumes of the phases present.
3) Transfer owing to microscopic fluid motion or mixing is much more rapid than that due to molecular motion (diffusion).
4) Thermodynamics provides a limit to the concentration of species within a phase and often governs the interfacial composition during transfer.
5) Temperature has only a modest influence on mass transfer rates under a given concentration driving force.

3.5.1.3 Principles of Equilibrium between Two Insoluble Phases

In the dissertation the following principles of equilibrium are applied (Helfferich, 1962).

1. At a fixed set of conditions, reflecting to temperature and pressure, there exists a set of equilibrium relationships which can be shown graphically in the form of an equilibrium distribution curve for each distributed substance by plotting the equilibrium concentrations of the substance in the two phases against each other.
2. For a system in equilibrium, there is no net diffusion of the components between the phases.
3. For a system not in equilibrium, diffusion of the components between the phases will occur so as to bring the system to a condition of equilibrium. If sufficient time is available, equilibrium concentration will eventually prevail.
4. In equilibrium the concentration in the two phases are not equal. Instead the chemical potential of the substance is the same in both phases. The equality of chemical potentials not concentrations causes the net transfer of solute to stop.
5. Under steady state conditions, the concentrations at any point in the column do not change with passage of time.

Equilibria between phases required a balance of all potentials, including chemical potential of each component in each phase (Luyben, 1990; Smith, 1963). Equilibrium between two phases occurs when the chemical potential of each component is the same in the two phases:

$$\mu_j^I = \mu_j^{II}, \tag{3.55}$$

where μ_j^I – is the chemical potential of the j^{th} component in phase I,

μ_j^{II} – is the chemical potential of the j^{th} component in phase II.

For ion exchange process the relative volatility α_{ij} of component i to component j is defined

$$\alpha^*_{ij} = \frac{y_i / x_i^*}{y_j / x_j^*}. \tag{3.56}$$

where x_i^* – composition in x phase for the i^{th} component

x_j^* – composition in x phase for the j^{th} component

y^*_i – composition in y phase for the i^{th} component

y^*_j – composition in x phase for the j^{th} component

This coefficient is frequently used in a number of systems. In a binary system,

$$\alpha^* = \frac{y^* / x^*}{(1-y^*)(1-x^*)} \rightarrow y = \frac{\alpha^* x^*}{1+(\alpha^*-1)x^*}. \tag{3.57}$$

It is assumed that the relative volatility is constant throughout the column and therefore 100% efficient stage is achieved – i.e., the liquid leaving the stage is in equilibrium with the resin on the stage. This means the simple liquid-resin equilibrium relationship can be used for the n^{th} stage of the ion exchange column.

$$y_n = \frac{\alpha^* x_n}{1+(\alpha^*-1)x_n} \tag{3.58}$$

where x_n – is the resin composition on the n^{th} stage (mole fraction more volatile component – *Na*).

y_n – is the liquid composition on the n^{th} stage (mole fraction more volatile component – *Na*)

α^* – is the relative volatility

In engineering calculations it is often convenient to express phase equilibrium by the relation.

$$y = ax + b \tag{3.59}$$

where ax – is an experimentally determined constant.

This phase equilibrium (3.59) will be used for this work.

Models, based on mass balances for ion exchange process are described in Boston & Sullivan Jnr. (1972), Ford (1984), Majtoba (1997), Dodds, Hudson, Kershenbaum & Streat, (1973) and Ford and Kelly (1985). These models are not used for control.

3.5.2 Development of a Mathematical Model

3.5.2.1 Diagram and Notations of the Process

The system can be modelled on the same basis as other stage-wise contact devices, distillation and absorption columns, for conversion of resin and liquid streams within the column. The mass balances of the ion exchange column can be written based on the interrelations between flows entering and leaving every stage. The mass balance equation looks at the relationship between x_{Na^+} and y_{Na^+}. The model equations are written according to the following diagram and notations from Fig. 3.3.

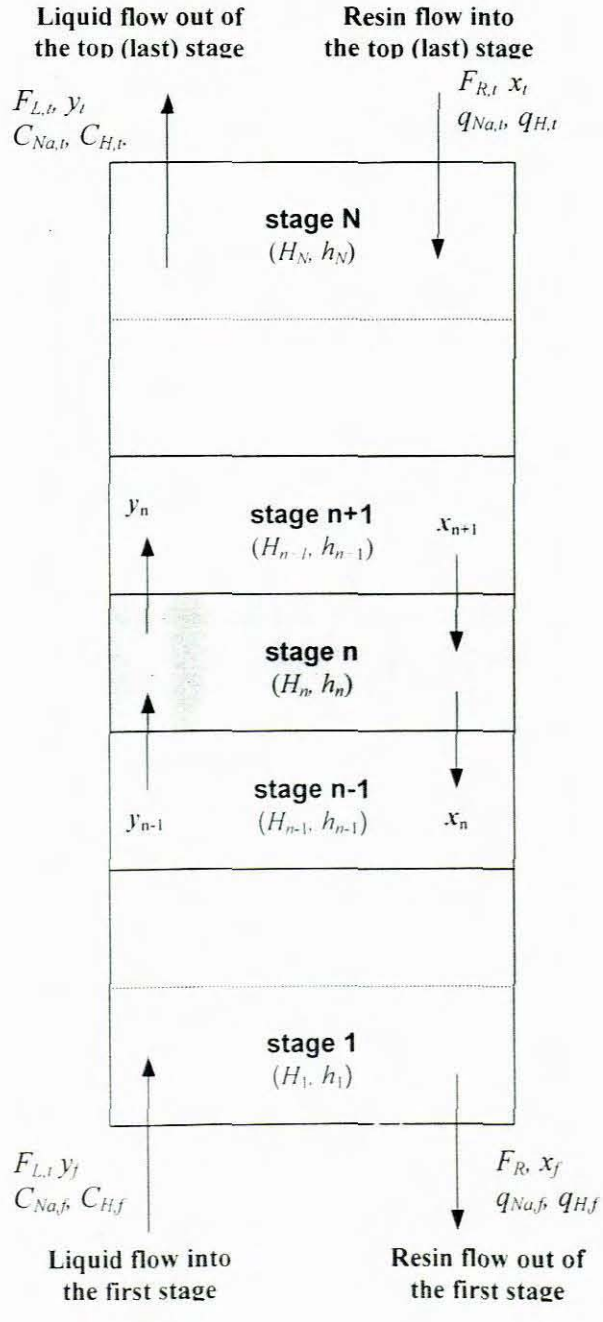


Figure 3.3. Structure used for Modelling the Ion Exchange Process.

The notations of the diagram are:

F_L – molar flow rate of the solution [moles/h],

F_R – molar flow rate of the resin [moles/h],

C_{Na}, C_H – concentrations of Na^+ and H^+ in the solution [mol/l],

q_{Na}, q_H – concentrations of Na^+ and H^+ in the resin [mol/l],

H_n – is the liquid holdups in the n^{th} stage,

h_n – is the resin holdups in the n^{th} stage,

$y_{n,Na} = \dfrac{C_{n,Na}}{C_{n,Na} + C_{n,H}}$ – is the mole fraction of Na^+ in liquid, in the n^{th} stage,

$y_{n,H} = \dfrac{C_{n,H}}{C_{n,Na} + C_{n,H}}$ – is the mole fraction of H^+ in liquid phase, in the n^{th} stage,

$x_{n,Na} = \dfrac{q_{n,Na}}{q_{n,Na} + q_{n,H}}$ – is the mole fraction of Na^+ in resin phase in n^{th} stage,

$x_{n,H} = \dfrac{q_{n,H}}{q_{n,Na} + q_{n,H}}$ – is the mole fraction of H^+ in resin phase in the n^{th} stage,

$y_{f,Na}$ – is the mole fraction of H^+ in resin phase in the n^{th} stage.

In calculation of the mole fractions every concentration is divided by atomic weight of the corresponding ions, for example,

$$y_{n,Na} = \frac{C_{n,Na}/AW_{Na}}{\dfrac{C_{n,Na}}{AW_{Na}} + \dfrac{C_{n,H}}{AW_H}}, \tag{3.60}$$

where AW – is the atomic weight.

Staged countercurrent process represents coupled graph in which the decomposition must take place stage by stage. The composition is a binary one – Na^+ ions in solution and H^+ ions in the resin begin at the beginning of the process.

The feed, the solution containing y_f mole fraction of component Na is fed to the column at molar flow rate F_L [moles/h]. The point of entry is the first stage. The resin is flowing through the column at the molar rate F_R from the top of the column. At each stage a

solution flowing upward is brought into contact with the resin at that stage and achieves thermodynamic equilibrium.

3.5.2.2 Assumptions for the Ion Exchange Process for the Development of the Model

The counter current ion exchange model is then developed based on the following assumptions:

- Both the volume and the amount of resin holdups (h) and liquid holdups (H) stage are equal just before the transfer. Equal stage volumes of resin are transferred between stages.
- The transfer of resin between any two stages is instantaneous and neither ion exchange reaction nor absorption takes place during this period. Hydro-dynamical delays in liquid and resin streams are neglected.
- The resin particles are uniform both in size and density at all levels so that segregation does not occur.
- The fluidized phase is perfectly mixed in each stage and the expanded fluidized bed fills the entire stage volume, i.e. the concentration is the same everywhere.
- There is no back mixing. (any volume changes that may occur are negligible)
- The operation of the process is at steady state and electroneutrality is maintained (for each equivalent of ion H^+ moving from solid phase to liquid phase an equivalent of Na moving in the opposite direction).
- There is a linear equilibrium relationship between liquid and resin. Each stage is assumed to be 100% efficient – the liquid leaving each stage is in equilibrium with the resin on the stage.
- The relative volatility α^* of the components in resin and liquid remains constant through the column.

3.5.2.3 Mass Balance Equations Derivation

The model is obtained on the basis of component Na mass balance on stage n. The following rates are calculated,

1. Rate of material input with liquid coming from stage (n-1) ≡ $F_{L,n-1} \cdot y_{n-1}$.
2. Rate of material input with resin coming from stage (n+1) ≡ $F_{R,n+1} \cdot x_{n+1}$.
3. Rate of material output with liquid leaving stage n for stage (n+1) ≡ $F_{L,n} \cdot y_n$.
4. Rate of material output with liquid leaving stage n for stage (n-1) ≡ $F_{R,n} \cdot x_n$.
5. Rate of accumulation Na in liquid phase on stage n ≡ $\frac{d(H_n y_n)}{dt}$.
6. Rate of accumulation of Na in the resin phase on stage n ≡ $\frac{d(h_n x_n)}{dt}$.

where Na is omitted in expressions as it is supposed that

$$y_{n,Na} = y_n$$

$$x_{n,Na} = x_n$$

According to component mass balance and the total mass balance respectively:

The Na mass balance is

$$\frac{d(H_n y_n)}{dt} + \frac{d(h_n x_n)}{dt} = (F_{L,n-1} y_{n-1} + F_{R,n+1} x_{n+1}) - (F_L y_n - F_{R,n} x_n), \tag{3.61}$$

and the total mass balance is

$$\frac{d(H_n)}{dt} + \frac{d(h_n)}{dt} = (F_{L,n-1} - F_{L,n}) + (F_{R,n+1} - F_{R,n}), \tag{3.62}$$

can be described.

According to the introduced assumptions

H_n = constant = H

h_n = constant = h

$F_{R,n}$ = constant = F_R

$F_{L,n}$ = constant = F_L

The last two assumptions imply that a simple liquid/resin equilibrium relationship can be used to relate the molar fraction of sodium in the resin leaving the i^{th} stage with the molar fraction of the liquid leaving the same stage given by formulas (equations for $y_{n,Na}$, $y_{n,H}$, $x_{n,Na}$, $x_{n,H}$) and connected by the separation factor.

$$\alpha_H^{Na} = \frac{y_{Na} x_H}{x_{Na} y_H}. \tag{3.63}$$

The fractions are connected by

$$x_{Na} + x_H = 1 \rightarrow x_H = 1 - x_{Na}, \tag{3.64a}$$

$$y_{Na} + y_H = 1 \rightarrow y_H = 1 - y_{Na}. \tag{3.64b}$$

From here it can then be deduced that

$$\alpha_H^{Na} = \frac{y_{Na}(1 - x_{Na})}{x_{Na}(1 - y_{Na})}, \tag{3.65}$$

and the molar fraction of the Na in the resin can be calculate using the separation factor and the molar fraction of the Na into the liquid, as

$$x_{Na} = \frac{y_{Na}}{\alpha_H^{Na} + y_{Na}(1 - \alpha_H^{Na})}, \tag{3.66}$$

and the equilibrium is defined by the separation factor. If the above expression is substituted into the model equations, the model will be expressed only by the sodium fraction into the solution. This equation is highly nonlinear and it is difficult to be used for calculation and control. That is why a linear assumption is needed (Himmelblau, and Bischoff, 1968) of the kind

$$y_{n,Na} = a_n x_{n,Na} + b_n, \tag{3.67}$$

where a_n and b_n are the coefficients determined experimentally for every column stages.

This equation (3.67) is true only for small perturbations about some stationary value of x_n over the equilibrium curve.

3.5.2.4 Development of the Model for the Aim of Optimal Control

- **Condition for Holdups**

After substitution of the conditions for constant holdups and flow rates the equation for the balance of Na component becomes:

$$H\frac{dy_n}{dt}+h\frac{dx_n}{dt}=F_L(y_{n-1}-y_n)-F_R(x_{n+1}-x_n). \tag{3.68}$$

- **Condition for Linear Equilibrium**

After substitution of (3.74) linearity equation, the equilibrium model is obtained:

$$H\frac{d(y_n)}{dt}+h\frac{d(a_ny_n+b_n)}{dt}=F_L(y_{n-1}-y_n)+F_R\left[(a_{n+1}y_{n+1}+b_{n+1})-(a_ny_n-b_n)\right]. \tag{3.69}$$

- **State Space Model Derivation**

After some mathematical transformation equation (3.69) can be written in the form

$$(H+a_nh)\frac{dy_n}{dt}=F_L(y_{n-1})+F_R(a_{n+1}y_{n+1}-F_Ra_ny_n+F_R(b_{n+1}-b_n), \tag{3.70a}$$

$$\frac{dy_n}{dt}=\frac{F_L}{H+a_nh}(y_{n-1}-y_n)+\frac{a_{n+1}}{H+a_nh}(F_Ry_{n+1})-\frac{a_n}{H+a_nh}(F_Ry_n)+\frac{b_{n+1}-b_n}{H+a_nh}F_R \tag{3.70b}$$

This is mass balance equation for the n-th stage. The model for the whole column can be obtained after giving values of $n = 1, 2,\ldots,N$. For the first stage

$$y_{n-1} = y_{1-1} = y_0 = y_f ,$$

is equal to the input concentration of Na into the solution.

For the last stage

$$y_N = z ,$$

is the output concentration of the solution,

$$y_{n+1} = y_{N+1} = 0 ,$$

because the input concentration of Na into the resin is zero.

The corresponding equations are

- For the first stage

$$\frac{dy_1(t)}{dt} = \frac{F_L}{H + a_1 h} y_f - \frac{F_L}{H + a_1 h} y_1(t) + \frac{a_2}{H + a_1 h} F_R(t) y_2(t) - \\ - \frac{a_1}{H + a_1 h} F_R(t) y_1(t) + F_R \frac{(b_2 - b_1)}{H + a_2 h}$$

- For the second stage

$$\frac{dy_2(t)}{dt} = \frac{F_L}{H + a_2 h} y_1(t) - \frac{F_L}{H + a_2 h} y_2(t) + \frac{a_3}{H + a_2 h} F_R(t) y_3(t) - \\ - \frac{a_2}{H + a_2 h} F_R(t) y_2(t) + F_R(t) \frac{(b_3 - b_2)}{H + a_n h}$$

- For the n^{th} stage

$$\frac{dy_n(t)}{dt} = \frac{F_L}{H + a_n h} y_{n-1}(t) - \frac{F_L}{H + a_n h} y_n(t) + \frac{a_{n+1}}{H + a_n h} F_R(t) y_{n+1}(t) - \\ - \frac{a_n}{H + a_n h} F_R(t) y_n(t) + F_R(t) \frac{(b_{n+1} - b_n)}{H + a_2 h} \qquad (3.71)$$

- For the N^{th} stage (last stage)

$$\frac{dy_N(t)}{dt} = \frac{F_L}{H + a_N h} y_{N-1}(t) - \frac{F_L}{H + a_N h} y_N(t) + 0 - \frac{a_N}{H + a_N h} F_R(t) y_N(t) + F_R(t) \frac{(0 - b_N)}{H + a_N h}$$

The following parameters can be generated from each stage equation.

$$\begin{aligned}
&\frac{F_L}{H+a_1h}=l_1, \quad \frac{a_2}{H+a_1h}=m_{12} \quad \frac{a_1}{H+a_1h}=m_{11}, \quad \frac{b_2-b_1}{H+a_1h}=k_1\\
&\frac{F_L}{H+a_2h}=l_2, \quad \frac{a_3}{H+a_2h}=m_{23} \quad \frac{a_2}{H+a_2h}=m_{22}, \quad \frac{b_3-b_2}{H+a_2h}=k_2\\
&\ldots \qquad\qquad \ldots \qquad\qquad \ldots \qquad\qquad \ldots\\
&\frac{F_L}{H+a_Nh}=l_N, \quad \frac{a_{N+1}}{H+a_Nh}=m_{NN+1} \quad \frac{a_{N+1}}{H+a_Nh}=m_{NN}, \quad \frac{b_{N+1}-b_N}{H+a_Nh}=k_N
\end{aligned} \tag{3.72}$$

or in common notation (3.71) can be rewritten as:

$$\frac{F_L}{H+a_nh}=l_n, \quad \frac{a_{n+1}}{H+a_nh}=m_{n,n+1} \quad \frac{a_n}{H+a_nh}=m_{n,n}, \quad \frac{b_{n+1}-b_n}{H+a_nh}=k_n$$

Equation (3.53) can now be rewritten in the form:

$$\begin{aligned}
&\dot{y}_1(t)=l_1y_f-l_1y_1(t)+m_{12}F_R(t)y_2(t)-m_{11}F_R(t)y_1(t)+k_1F_R(t)\\
&\dot{y}_2(t)=l_2y_1(t)-l_2y_2(t)+m_{23}F_R(t)y_3(t)-m_{22}F_R(t)y_2(t)+k_2F_R(t)\\
&\ldots \quad \ldots \quad \ldots \quad \ldots \quad \ldots \quad \ldots\\
&\dot{y}_n(t)=l_ny_{n-1}(t)-l_ny_n(t)+m_{nn+1}F_R(t)y_{n+1}(t)-m_{nn}F_R(t)y_n(t)+k_nF_R(t)\\
&\ldots \quad \ldots \quad \ldots \quad \ldots \quad \ldots\\
&\dot{y}_8(t)=l_8y_7(t)-l_8y_8(t)-m_{88}F_R(t)y_8(t)+k_8F_R(t)
\end{aligned} \tag{3.73}$$

After selecting $y=[y_1,y_2,y_3\ldots y_n,\ldots y_N]^T$ as a state space vector, the state space model of the ion exchange process can be written as:

$$\begin{aligned}
&\dot{y}(t)=Ay(t)+B\,y(t)\,F_R(t)+B_1\,F_R(t)+Wy_f(t), \quad y(0)=y_0\\
&z(t)=Cy(t)
\end{aligned} \tag{3.74}$$

where

$$A=\begin{bmatrix} \frac{-F_L}{H+a_1h} & 0 & 0 & \dots & 0 \\ \frac{F_L}{H+a_2h} & \frac{-F_L}{H+a_2h} & 0 & \dots & 0 \\ \dots & \dots & \dots & \dots & \dots \\ 0 & 0 & \frac{F_L}{H+a_{N-1}h} & \frac{-F_L}{H+a_{N-1}h} & 0 \\ 0 & 0 & \dots & \frac{F_L}{H+a_Nh} & \frac{-F_L}{H+a_Nh} \end{bmatrix}, B_1=\begin{bmatrix} \frac{(b_2-b_1)}{H+a_1h} \\ \frac{(b_3-b_2)}{H+a_2h} \\ \vdots \\ \frac{(b_N-b_{N-1})}{H+a_nh} \\ \frac{-b_N}{H+a_Nh} \end{bmatrix}$$

$$B=\begin{bmatrix} \frac{-a_1}{H+a_1h} & \frac{a_2}{H+a_1h} & 0 & \dots & 0 \\ 0 & \frac{-a_2}{H+a_2h} & \frac{a_3}{H+a_2h} & \dots & 0 \\ \dots & \dots & \dots & \dots & \dots \\ 0 & 0 & \frac{-a_{N-1}}{H+a_{N-1}h} & \frac{a_N}{H+a_{N-1}h} & 0 \\ 0 & 0 & \dots & 0 & \frac{-a_N}{H+a_Nh} \end{bmatrix}, W=\begin{bmatrix} \frac{F_L}{H+a_1h} \\ 0 \\ \dots \\ 0 \\ 0 \end{bmatrix}$$

$$C=\begin{bmatrix} 0 & 0 & \dots & \dots & 1 \end{bmatrix}$$

where $A \in R^{NxN}, B \in R^{NxN}, B1 \in R^{Nx1}, W \in R^{Nx1}, C \in R^{1xN}$

z – is the output of the model,

$F_R \in R^{1\times1}$ – is the control input,

$y \in R^{N\times1}$ – is the state space vector,

$y_f \in R^{1\times1}$ – is the input disturbance vector.

The developed model is a bilinear one.

Bilinear systems are characterized by simplicity and better capability to describe real process (Hohler, 1973; Guerra and Morales, 1995; Niculescu, Dion, & Dugard, 1995; Quinn, 1990; Ryan & Buckinghan, 1983).

Conditions that guarantee the stabilization of a SISO bilinear system via constrained output feedback global asymptotic stabilization and stabilization are analyzed in Celikovisky (1993). Structural stabilization conditions for bilinear systems of second order are given in Koditschek and Narendra (1983).

The common bilinear model is given by the equation

$$\dot{x}(t) = Ax(t) + \sum_{i=1}^{m} u_i(t) B_i x(t) + B_1 u(t) \tag{3.75}$$

where $x \in R^{n\times 1}$, $u \in R^{m\times 1}$, $A \in R^{n\times n}$, $B_i \in R^{n\times n}$, $B_1 \in R^{n\times m}$

In the considered case of ion exchange the control $F \in R^{1\times 1}$.

5.2.2 Development of Discrete Time Model

Digital control systems inherently involve the processing of sampled signals. Thus, unless the sampling period is very small it is more appropriate and convenient to perform the design and analysis of digital control systems using discrete time model rather than continuous time models (Davis, 1984; Cadzow & Martens, 1970, Lapidus, 1962).

One way of converting continuous time models to discrete time form is to use finite difference techniques, by introducing a finite difference approximation for the derivative at time $t = K\Delta t$

$$\frac{dy}{dt} = \frac{y_k - y_{k-1}}{\Delta t} \text{ or} \tag{3.76a}$$

$$\frac{dy}{dt} = \frac{y_{k+1} - y_k}{\Delta t} \tag{3.76b}$$

where Δt – is the integration (sampling) interval specified by the user.

y_{k+1} – is the value of y(t) at the moment (k+1)Δt,

y_{k-1} – is the value of y(t) at the moment (k-1)Δt,

y_k – is the value of y(t) at the moment (k)Δt.

It is important to note the calculated parameter values of the model depend on the sampling period Δt that is selected. This approach of discretizing is used in the dissertation because the model equations are bilinear and the analytical solution is not available to be used for calculation of exact discrete time model (Neuman and Baradello, 1979).

An important advantage of discrete time models is that they can be readily obtained by fitting experimental response data (Cadzow and Martens, 1970).

Discrete time models are more appropriate and convenient to perform the analysis and design of the digital control system. The exact discrete time model can be obtained based on the analytical solution of the differential equation for a piece-wise constant input. The requirement for the model to be exact is the input variable to be actual constant during the sampling instants. The discrete time model is then formed using the presentation:

$$y(t) = \frac{y(k+1) - y(k)}{\Delta t} \tag{3.77}$$

After substitution of (3.77) into the continuous time model, (3.73), the discrete version is obtained

$$
\begin{aligned}
y_1(k+1) &= y_1(k) + \Delta t\left[l_1 y_f(k) - l_1 y_1(k) + m_{12} F_R(k) y_1(k) - \right. \\
&\qquad \left. - m_1 F_R(k) y_1(k) + k_1 F_R(k)\right] \\
y_2(k+1) &= y_1(k) + \Delta t\left[l_2 y_1(k) - l_2 y_2(k) + m_{23} F_R(k) y_2(k) - \right. \\
&\qquad \left. - m_2 F_R(k) y_2(k) + k_2 F_R(k)\right] \\
\ldots \quad & \quad \ldots \quad \ldots \quad \ldots \quad \ldots \\
y_n(k+1) &= y_n(k) + \Delta t l\left[{}_n y_{n-1}(k) - l_n y_n(k) + m_{n,n+1} F_R(k) y_n(k) - \right. \\
&\qquad \left. - m_n F_R(k) y_n(k) + k_n F_R(k)\right] \\
\ldots \quad & \quad \ldots \quad \ldots \quad \ldots \quad \ldots \\
y_N(k+1) &= y_N(k) + \Delta t\left[l_N y_{N-1}(k) - l_N y_N(k) - m_N F_R(k) y_N(k) + k_N F_R(k)\right]
\end{aligned}
\tag{3.78}
$$

The output equation can then be written as:

$$z(k) = y_N(k) = Cy(k) \tag{3.79}$$

where

$$y(k) = \begin{bmatrix} y_1 \\ y_2 \\ y_3 \\ . \\ . \\ y_N \end{bmatrix}$$ – is the state space vector in discrete state space

The coefficients of the models are calculated on the basis of theoretical considerations and of the experiments with the ion exchange plant. The method of least squares can then used to fit the model data on the experimental data to finalize the model coefficients. The selected control action for the process is the up-flow time (T^s). It is connected with the value of the resin flow rate on the basis of mass balance in every stage.

$$F_R^s T^s = hd, \quad T^s = \frac{hd}{F_R^s}, \tag{3.80}$$

where the upper index s is for the steady state balance

3.6 EMPIRICAL MODEL DEVELOPMENT FOR THE ION EXCHANGE

3.6.1 Experiments with the Process

Development of empirical models from step response data is achieved by using data directly obtained from the experiments. The model is bilinear according to the control and the state but it is linear according to parameters l_n, m_{nn}, m_{nn+1}, k_n *and* $n = \overline{1,8}$. Every equation characterized by four parameters, the number of equations is 8 or the common number of parameters, the number is 31. In order to estimate the values of the parameters the experiments with the process has to be provided during which the values of the *input control variable* and the values of the output $z(k)$ are measured.

The necessity for building a model of the cation columns is to study dynamic behaviour of the ion exchange process as an object of control and also to estimate the influence of the main disturbance, the salt concentration of wastewater over the dynamic behaviour of the columns. The main disturbances for the process model control are any disturbances that affect the process steady state conditions. They may occur in three ways:

- Increase/decrease of the column up-flow time
- Increase/decrease of the feed concentration
- Increase/decrease of the liquid feed flow rate

- **Necessity of Experiments**

An understanding is required of the response of the column to disturbances of the steady state condition and to changes in the manipulated (control) variables. This response will be an important factor in determining,

- the process model development and fitting of the parameters
- characteristics of the control algorithm understanding.

With this in mind a series of experiments have to be done in which a step change to some input variables will be made and the rate of approach to the new steady state monitored.

- **Conditions of Experiments**

1. A pair of columns - cation load and its regeneration is used.
2. Measurements of the above three input variables is made at the input.
3. The plant operates first until it reaches the steady state of the load column and this will be confirmed by analysis (of at least 30 cycles),
   - the regeneration column operates with a large excess of acid to ensure complete stripping of Na^+ ions from the resin.
   - the load column could thus always be assumed to be filled with resin completely in H^+ form.
4. Three complete sets of samples are taken over three consecutive cycles, so that the initial steady state profile could be defined.
5. The sample points is taken at the mid-time of each cycle from the first and the last stage (ideally at each stage, but this is not possible due to the nature of design).
6. The up-flow times, feed concentrations and flow rates are changed by 50% up and down and samples taken during every cycle until new steady state is reached.
7. When one of the input variables is changed others are kept constant.

- **Experiments**

The flow chart of the experiments is

1) Starting of the ion exchange process with $T = 100\%$
2) Waiting until the process reaches steady state values for y_n, $n = \overline{1,8}$.

   During this time, measurement of the concentrations of the Na in liquid is done for input and output of the column.
3) Changing the value of T, incremented by 50% to $T = 150\%$.

   The process goes to a *"new steady state"*.
4) Measurement of the concentration of Na in the liquid until the process reaches a *"new steady state"*.
5) Decreasing the value of T with 50% and repetition of measurements.

The same experiments are done with the up-flow rate F_L and input flow concentration C_f. The data obtained for the up-flow period T will be used for parameter estimation as T is selected as a control action.

3.6.2 Problem Formulation for Parameter Estimation

3.6.2.1 Problem Formulation for Parameter Estimation

It is necessary to find the values of parameters l_n, m_{nn}, k_n, $n = \overline{1,N}$ and

$m_{n,n+l}$ $n = \overline{1,N-1}$ in such a way that the error between the measured values of the output

$$\bar{z}(k) = \bar{y}_8(k), \quad k = \overline{0,K} \tag{3.81}$$

and the output of the model,

$$z(k) = y_8(k), \quad k = \overline{0,K} \tag{3.82}$$

expressed by the criterion,

$$J = \sum_{k=0}^{K} \left\| e(k) \right\|^2 = \sum_{k=0}^{K} \left\| \bar{z}(k) - z(k) \right\|^2 \rightarrow \min \tag{3.83}$$

is minimized under the constraints of the model (3.73) and (3.74) where $k = \overline{0,K}$ is the number of points in time at which the values of the output of the process are measured. The criterion expresses the sum of the squares of the error between the measured and the calculated output values from the model trajectories. The criterion gives the best strategy for fitting the trajectory of the model to the measured trajectory. It has many advantages as it gives a unique trajectory for a given set of data.

3.6.2.2 The Method of Fastest Descent

This method is characterized with the simplest iterative gradient procedure for improvement of the initial estimation of the vector of parameters $P \in R^{m\times 1}$. The direction of the fastest descent is opposite to the direction of the gradient and at the initial point coincides with the direction in which the criterion reduces in the fastest way for infinitely small change of the vector of parameters. The direction of the fastest descent is given by the vector:

$$p^{(j+1)} = p^{(j)} + dp \tag{3.85}$$

where $dp = [dp_1, dp_2, ...dp_m]^T$ (3.86)

dp – is the direction of the descent

$$\delta p_i = -\frac{\alpha\left(\frac{\partial J}{\partial p}\right)}{\left[\sum_{i=1}^{m}\left(\frac{\partial J}{\partial p_i}\right)^2\right]^{1/2}} \tag{6.87}$$

In (3.66) and (3.68) $p^{(j+1)}$ is an improved value of parameters. p is the previous value of the gradient of j towards the i^{th} component of p and α is the step of the gradient. j is the index of iteration. The calculation of the components of the vector of gradient $\delta J/\delta p_i$ by direct analytical methods looking for derivatives is not possible since the model is dynamical and the criterion is not explicit function of the state and parameters. That is why the approximated values of the components are then determined by:

$$\frac{\delta J}{\delta p_i} = \frac{J(p_1, p_2, \ldots, p_{i+\Delta}, p_{i+1}, \ldots, p_m) - J(p_1, p_2, \ldots, p_i, p_{i+1}, \ldots, p_m)}{\Delta} \tag{3.88}$$

where Δ – is the small deviation of the i^{th} component of the vector of parameters

The considered method is used in the scheme of identification to calculate the improved estimated values of the parameters in such a way that the least squares criterion is minimized. The solution of the model equations (3.73), (3. 74), $z(k)$ where on the place

of parameters are substituted with their initial values p_0. The solutions are for the initial values of the state $y(k) = y_0$ which is given. These equations are solved for $k = \overline{0,K}$. With the obtained values of the output (3.82) the value of the criterion (below) is calculated for the i^{th} component.

$J(p_1, p_2, p_i^j, p_{i+1} \ldots, p_m)$.

3.6.2.3 Method of Solution Selection

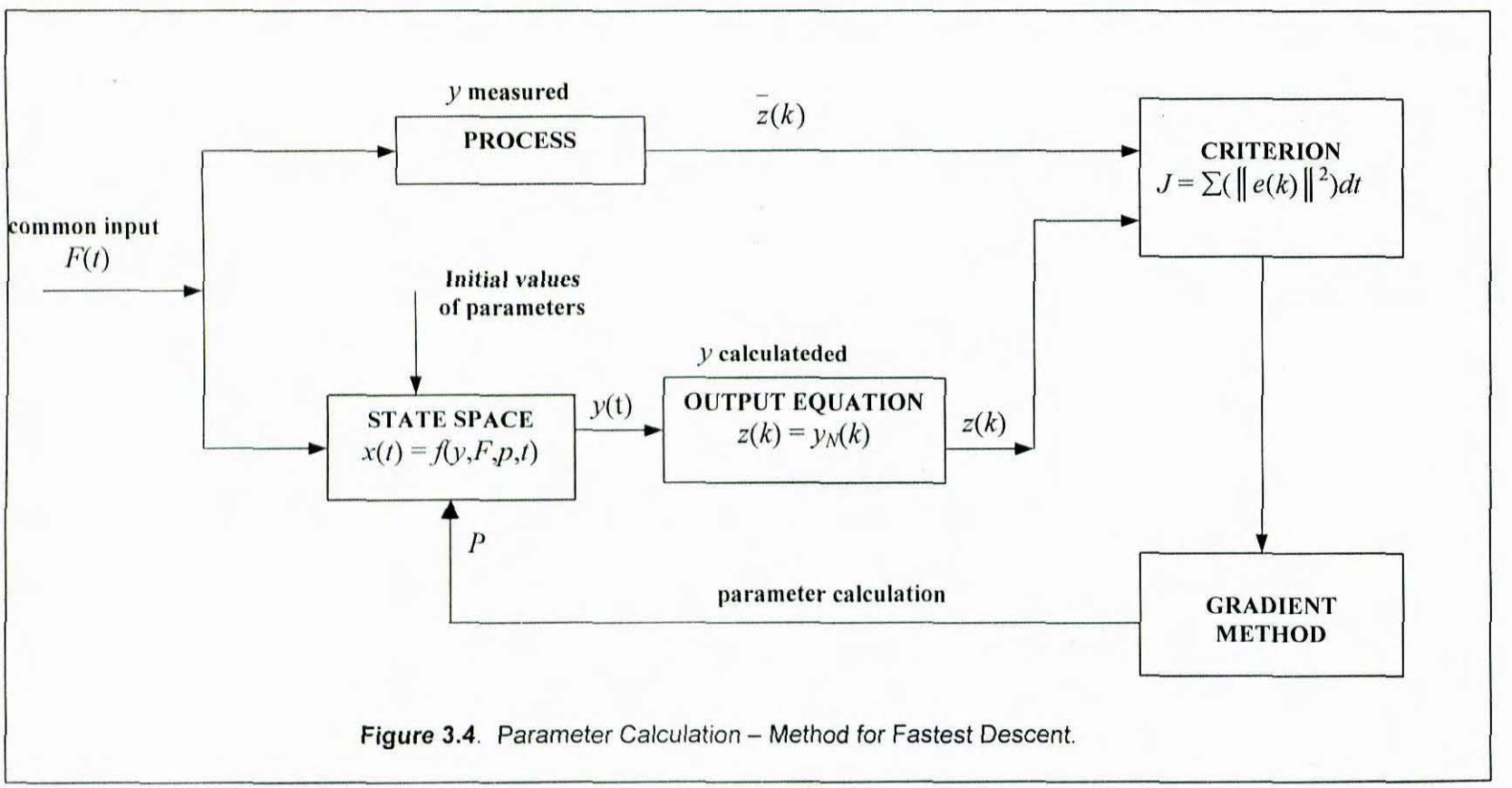


Figure 3.4. *Parameter Calculation – Method for Fastest Descent.*

The looked for solution will give the minimum criterion (3.83) – the criterion is the function of the output of the model – and in this way it is the function of the state $y(k)$. The state, $y(k)$ is the function of the parameter of finally the criterion is the function of the parameters. When we are looking for the minimum of criterion towards parameters, this means that according to the theory of optimality the criterion's first derivative according to each of the parameter is necessary to be equaled of zero. The optimal solution can be found by using the gradient methods using first derivatives of the criterion towards the parameters. The requirement is:

$$\frac{\delta J}{\delta p_i} = 0, \quad i = 1,2,3...m, \; P = [p_1, p_2, ..., p_m] \tag{3.84}$$

where $P \in R^{m\times 1}$ – is a vector of parameters with dimension m.

As the criterion is not the explicit function of the vector of parameters it is necessary to use the dependence of the output from the state space equations in connection with the algorithm of some of the different gradient methods for computation. The method of the fastest descent will be used.

3.6.2.4 Algorithm of the Method

- **Algorithm 3.1:**

1) The algorithm is an iterative one
   - at the beginning of the iteration, initial values of $\bar{z}(k)$; $k = \overline{0,K}$ and y_8 from the experiments are given
   - the following are initialized:

     Δ, the value of deviation

     M, the number of iterations

     y_0, initial values of the state vector

     α, step of the gradient procedure

     m, the number of parameters

     K, number of steps in optimization period K

     ε, error for calculation of the estimated value

     $p^{(j)} = p_0$, the initial estimation of the vector of parameters

     N, the number of stages

     j, is the index of calculation

   $$p = [p_1, p_2, \ldots p_r]^T = [l_1, m_{12}, m_1, k_1 \ldots l_n, m_{n,n+1}, m_n, k_n \ldots l_N, m_N, k_N]$$

2) The solution of the equations (3.73), (3.74) where on the place of the parameters are substituted, their initial values, p_0
   - the solutions are for initial values of the state variables $y(k) = y_0$ (which are given)
   - the equations are solved for $k = \overline{0,K}$.

3) with the obtained values of the output (3.82), the value of the criterion (3.83) is calculated

   $-J(p_1, p_2, \ldots, p_m)$

4) set $j = 1$,

- set the deviated value of the parameters for i^{th} component

$$\Delta p_i^{(j)} = p_i^{(j)} + \Delta$$

- For every $i = 1,2,3....m$ and separately for every $p_i^{(j)}$

a) Solve the model equation (3.73), (3.74) where the components of the vector p are the same with only $p_i^{(j)}$ changed to $\Delta p_i^{(j)}$

b) Calculate the deviated value of the criterion for i^{th} component.

$$J(p_1, p_2, \Delta p_i^{(j)}, p_{i+1}, \ldots p_m)$$

c) Gradients

$$\frac{\partial J}{\partial p_i^{(j)}}$$

are calculated using equation (3.88)

These calculations are for every $i = 1,2,\ldots,m$.

5) Calculation of the gradients for the fastest descent dp_i

- calculation of the weighted sum

$$S = \left[\sum_{i=1}^{m} \left(\frac{\partial J}{\partial p_i^{(j)}} \right)^2 \right]^{1/2}, \tag{3.89}$$

- calculation of the direction of the gradients

$$dp_i^{(j)} = \frac{\alpha \left(\dfrac{\partial J}{\partial p_i^{(j)}} \right)}{S}, \qquad i = 1,2,3,\ldots,m. \tag{3.90}$$

6) calculation of the error for termination of the calculation

$$ep_i = \left| p_i^{(j+1)} - p_i^{(j)} \right|, \; i = 1,2,\ldots,m. \tag{3.91}$$

7) criterion for termination of the calculation

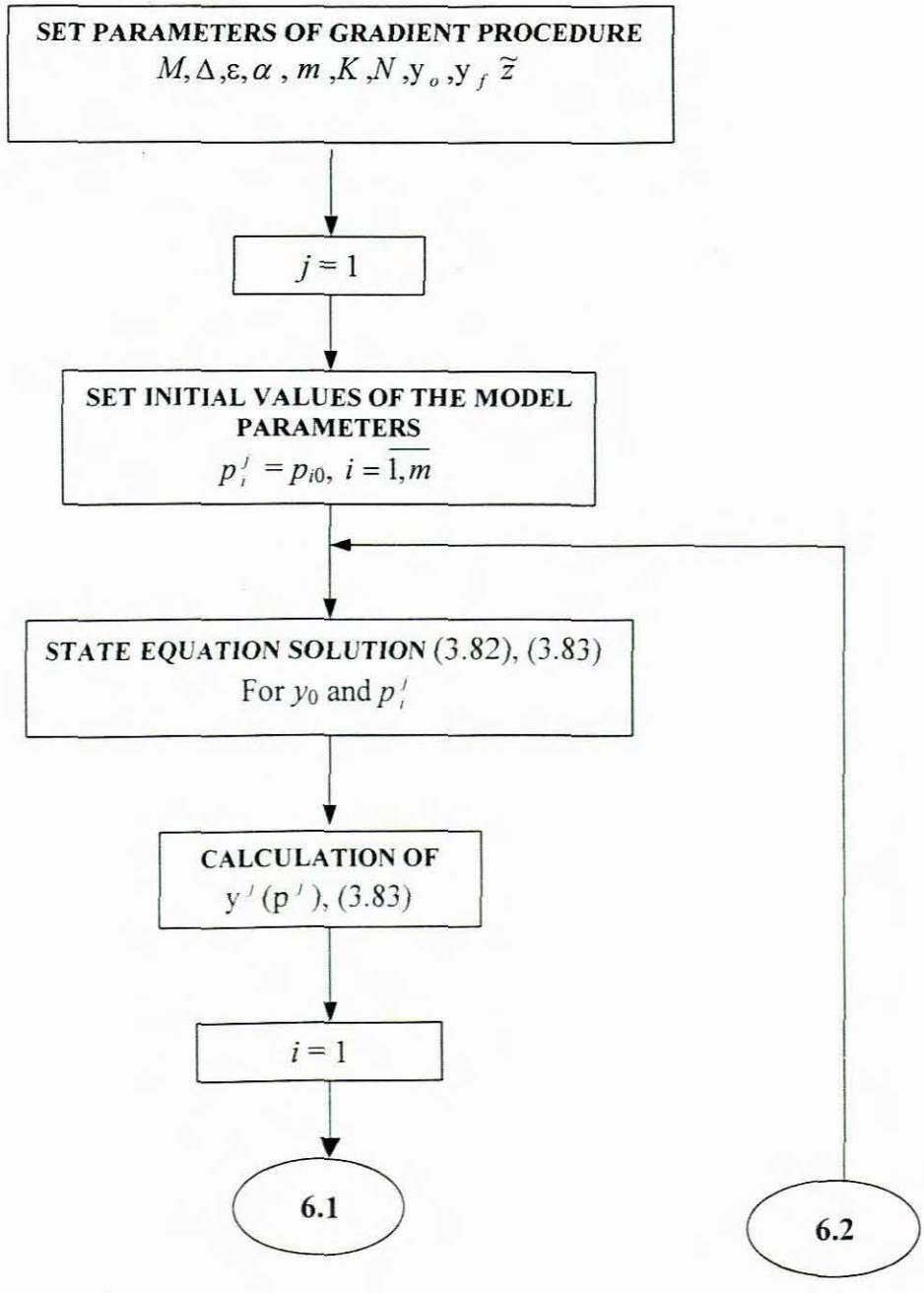
- if $\left| dp_i^{(j)} \right| \le \varepsilon$, (3.92)

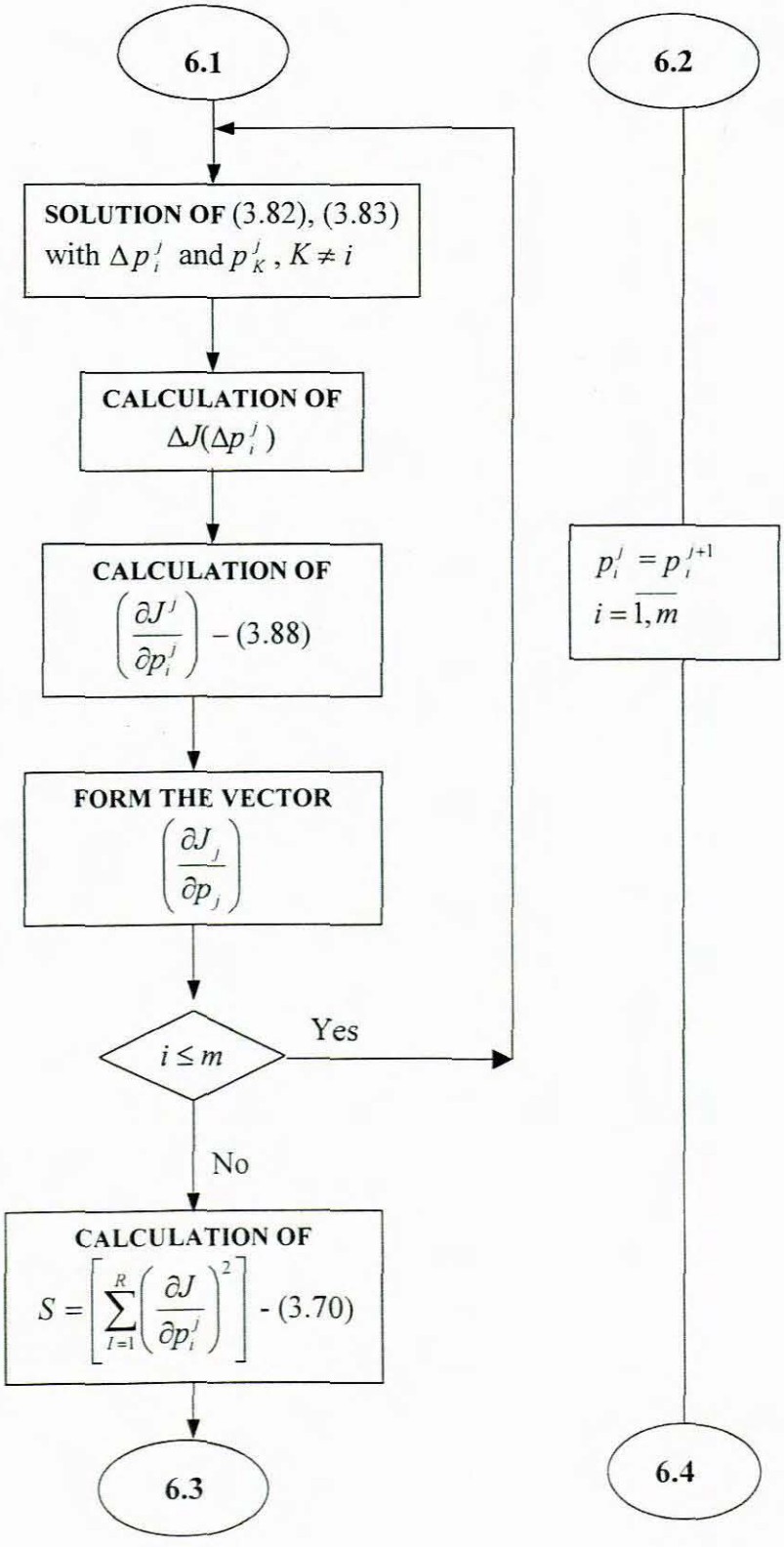
where ε is a small number at every $i = 1,2,3,\ldots$m.

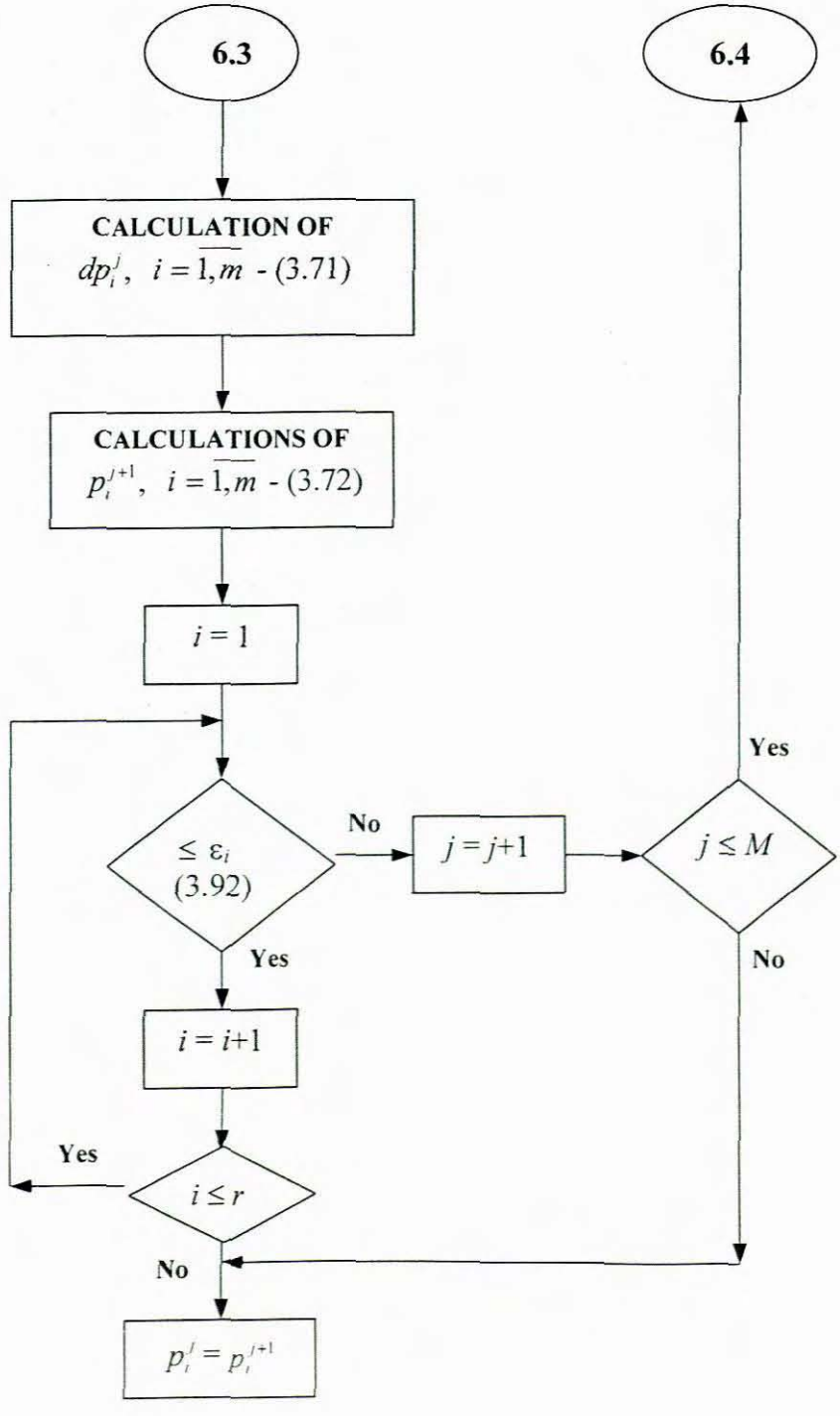
* the optimal solution is obtained and the calculation is terminated

- if $\left|dp_i^{(j)}\right| \geq \varepsilon$,
  * the calculation is continued until the maximum number of iterations, M is reached. The calculations are repeated from step (6)
- if $j > M$,
  * the calculations are terminated.

- **Block Diagram of The Algorithm**




6.1
6.2
SOLUTION OF (3.82), (3.83) with Δp_i^j and p_K^j, $K \neq i$
CALCULATION OF $\Delta J(\Delta p_i^j)$
CALCULATION OF $\left(\frac{\partial J^j}{\partial p_i^j}\right)$ – (3.88)
FORM THE VECTOR $\left(\frac{\partial J_j}{\partial p_j}\right)$
$i \leq m$
Yes
No
$p_i^j = p_i^{j+1}$
$i = \overline{1,m}$
CALCULATION OF $S = \left[\sum_{I=1}^{R}\left(\frac{\partial J}{\partial p_i^j}\right)^2\right]$ - (3.70)
6.3
6.4


6.3
6.4
CALCULATION OF
dp_i^j, $i = \overline{1,m}$ - (3.71)
CALCULATIONS OF
p_i^{j+1}, $i = \overline{1,m}$ - (3.72)
$i = 1$
$\leq \varepsilon_i$
(3.92)
No
$j = j+1$
$j \leq M$
Yes
No
Yes
$i = i+1$
Yes
$i \leq r$
No
$p_i^j = p_i^{j+1}$

3.7 CONCLUSION

Theoretical background to modeling is given including the use of differential equations for models. Common principles in developing mathematical models are discussed including the types of models, the procedure for theoretical modeling and the problem formulation methods.

Conservation principles, mass balances, component balances, parameter estimation and model verification are outlined.

The procedure for experimental model development for parameter estimation is formulated. Different methods for identification problem are discussed – these methods include differential approximation, gradient methods, methods of direct search, methods of quasilinearization, methods of sensitivity functions and the least squares method.

A mathematical model for the ion exchange process is developed based on the component balance of the exchanging ion species – after having made some assumptions about the process' dynamic behavior. Characteristics of the ion exchange as an object of control are detailed using a developed discrete model.

A procedure for experiments and their necessity is developed to solve the model validation problem. The proposed solution to the parameter estimation problem is the method of fastest descent. An algorithm is developed for the solution.

The developed model is a realistic model which incorporates all the important dynamic effects of the process, which is not more complicated than relevant and necessary and keeps the number of equations and parameters at a reasonable level.

Future development to the estimation algorithm is to consider it in connection with optimal control problem, as one optimization problem (Teo et al, 1991; Roberts and Lin, 1991; Becerra and Roberts, 1996). Then the process control will be more realistic and robust one.

Chapter Four

Optimal Control Strategy

The nature of the subject of control engineering or control systems often referred to as control is concerned broadly with the analysis and design of the systems to control particular variables in dynamic processes in a desired way. The two main requirements are:

- to maintain a variable sensible to control and close to a desired value in spite of disturbances to the process,
- to alter a process variable to a new value quickly and yet accurately without excessive overshoot or oscillation (instability) of the system.

Process control design involves selection of appropriate controller parameters to achieve acceptable *dynamic behaviour* of the process. Control is concerned with dynamics in a wider sense, with seeking understanding of the dynamic behaviour of mechanical, electrical, chemical, biological, economical and other forms of system with a view to control variables in desired matter. These variables can be position, velocity, current, temperature pressure, flow rate, chemical concentration and currency exchange (economical). A control system is an interconnection of components forming a system configuration that will provide a desired response. The input output relationship represents the cause-effect relationship of the system (process) which in turn represents processing of input signal to provide a certain output signal. Control engineering deals

Chapter Four

Optimal Control Strategy

---

with monitoring and taking certain actions based on the monitored system (process) as required by the goals of the process (Hunter, 1987; Liptak, 1999;Thurston, 1980).

Sequential control is a process used in many automatic systems where a sequence of operations such that the conclusion of one operation is the start of the next.

This chapter deals with the application of control principles as mentioned above in controlling an ion exchange plant as used for water purification application.

4.1 INTRODUCTION

Careful control operating conditions are necessary to ensure that for a given desalination level, maximum water output is balanced with minimum regenerant chemicals consumption. This is achieved by designing an optimal control for the two main regimes of work:

- start-up required to be done for a minimum time to reach steady state
- steady state operation under the influence of slowly varying disturbances.

To achieve optimal control a personal computer (PC) based control system is used, whereby the column cycle times and regenerant feed rates are automatically optimized on the basis of salinity as determined by the measurement of pH and conductivity at different points on the plant. These measurements are then processed and used for the calculation of the sodium (Na) concentration at the output stream (cation load output stream for this investigation). Based on the calculation, a control action is then activated which involves the switching of relevant valve for the relevant control required. The following streams are currently used for measurement and monitoring:

- *feed-water input stream (cation input),*
- and cation output (acidic stream).

The figure below (Fig.4.1) shows the points where the pH and conductivity samples are taken. The computer control also performs on-line monitoring function with a comprehensive measurement and record keeping for the plant variables. Computer based monitoring and record keeping further helps in plant operation while eliminating the need for highly skilled operator (Randall, 1984).

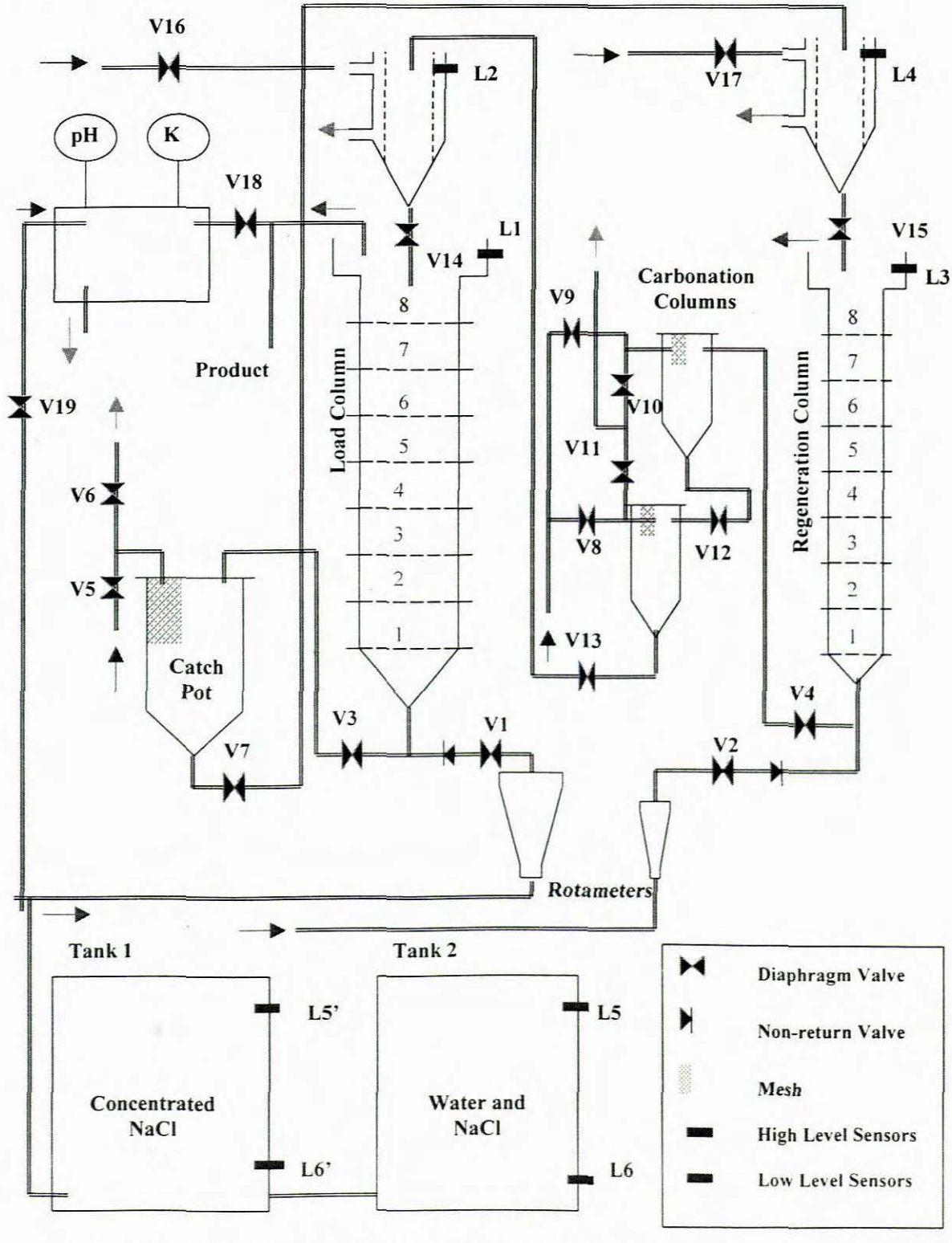


Figure 4.1. pH and Conductivity (K_{Na}) Measuring Points and Level Sensors.

4.2 MINIMUM TIME CONTROL OF ION EXCHANGE PROCESS

4.2.1 Optimization of the Minimum Startup Period

It has been established experimentally that the cation exchange column needs about thirty cycles to reach steady state conditions (Dodds, Hudson, Kershenbaum, and Streat, 1973; Xin, and Guihua, 1996). The starting time is very long. The time taken for the process to reach a new steady state when the feed water changes its concentration is also too long. Therefore it is necessary to determine the minimum time for the startup of the ion exchange process. The problem for minimum time control of the ion exchange is formulated and solved on the basis of a decomposition method. This method uses an augmented functional of Lagrange in order to overcome the difficulties with the singular type of control and nonquadratic criterion. A special coordination vector is constructed to achieve separability of the dual problem. The problem for minimum time can be decomposed in time domain and be solved in a two level hierarchical structure. A computer program is developed to solve this problem.

4.2.2 Minimum Time Problem Formulation

The aim is to minimize the time necessary for the process to reach the steady state values of concentration of the salt in the water (Fig.4.2). It is necessary to find a control trajectory which in minimum time leads the process from an initial state to a desired state. The problem for minimum time is a dynamic one and the used model is dynamical (Tsoneva & Popchev, 1995).

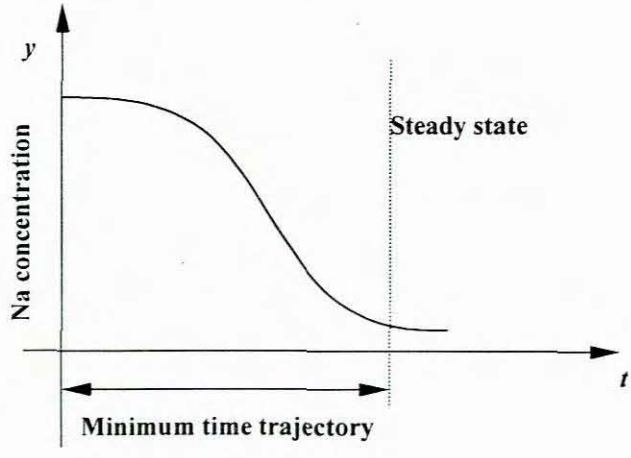


Figure 4.2. Minimum Startup Time Trajectory.

The problem for startup optimal control of continuous technological processes is formulated as a discrete problem for minimum time control in the following way:

Find the control,

$$u(k) = F_R(k), \quad k = \overline{0, K-1}, \tag{4.1}$$

which in minimum time

$$J = K\Delta t, \tag{4.2}$$

leads the system

$$y(k+1) = [1 + \Delta tA]y(k) + \Delta tBy(k)F_R(k) + \Delta tB_1F_R(k) + \Delta tWy_f(k), y(0) = y_0, \tag{4.3}$$

$$z(k) = Cy(k), \tag{4.4}$$

from the initial state $y(0) = y_0$, (4.5)

to the desired steady state $y(K) = \bar{y}$, (4.6)

for a minimum time and satisfies the constraints

$$v_{\min}(k) \le v(k) \le v_{\max}(k), \tag{4.7}$$

$$v(k) = y(k), \quad k = \overline{0, K}, \quad v(k) = F_R(k), \quad k = \overline{0, K-1}, \quad v = \Delta t, \tag{4.8}$$

where Δt is the sampling period,

$K\Delta t$ is the time for reaching the steady state,

$v_{\min}(k), v_{\max}(k)$ – are minimum and maximum values respectively for the state, control vectors and for the sampling,

$\bar{y}$ – is the desired steady state,

K – is the number of steps in the optimization interval.

4.2.3 Conditions for Optimality

The time $K\Delta t$ that is to be minimized is a product of two numbers. One of them can be fixed and the other can be minimized. As the sampling period is easier to be included in the model equations and to be minimized, the number of sampling steps will then be fixed. Then the problem for minimization of the startup time is transformed into a problem for minimizing of the sampling period time. The stated problem is characterized by *nonquadratic criterion* and *bilinear model* with the control entering linearly the model equations. The sampling period also enters linearly in to the model equations. This means that the solution of the optimal control problem can be singular because the first derivative of the function of Lagrange towards the control variable will not include this variable (*i.e. it will not be its analytical function*). To overcome these difficulties an augmented function of Lagrange is proposed. The quadratic term for the model equation is introduced by some penalty coefficient. The Lagrange functional has the form:

$$L_a = K\Delta t + p(K)^T[y(K) - \bar{y}] + \mu[y(K) - \bar{y}]^2 +$$
$$+ \sum_{k=0}^{K-1} \{\lambda(k)^T[y(k+1) - (1+\Delta tA)y(k) - \Delta tBy(k)F_R(k) - \Delta tB_1F_R(k) - \Delta tWy_f(k)] +$$
$$+ (1/2)\mu[y(k+1) - (1+\Delta tA)y(k) - \Delta tBy(k)F_R(k) - \Delta tB_1F_R(k) - \Delta tWy_f(k)]^2\}, \quad (4.9)$$

where $\lambda(k) \in R^{nxn}$ are the conjugate variables,

μ – is the penalty coefficient.

The problem is solved on the basis of the necessary conditions for optimality. As the model is a bilinear one and the augmented function of Lagrange is used the nonlinear two-point boundary value problem must be solved. In order to overcome these difficulties, the decomposition in two-level computing structure is applied on the basis of prediction of the conjugate vector. It is possible by the decomposition method of Tamura (1975),the values of the conjugate variables $\lambda(k)$ to be used as coordinating ones and on the basis of these values the initial problem given by the augmented Lagrangian to be decomposed in time domain. Then the nonlinear two-point boundary value problem can be solved by solving $(K+1)$ optimization sub-problems separately at each moment in time $k = \overline{0, K}$. However the values of the variables $y(k+1)$ and Δt in the square term connect the variables in the time domain making it impossible to decompose the augmented Lagrangian fully in time domain. To overcome these difficulties the values of the sampling period and of the state vector at moment k+1 are used as the coordinating ones. Together with the conjugate variables they form the coordinating vector. The values of this vector are determined from the second level of the hierarchical computing structure as follows:

$$\lambda(k) = \lambda^{l}(k), \qquad k = \overline{0, K-1}, \tag{4.10}$$

$$\rho(k) = y(k+1) = \rho^{l}(k), \quad k = \overline{0, K-1}, \tag{4.11}$$

$$\Delta t = \Delta t^{l}, \tag{4.12}$$

where l is the index of the coordinating procedure.

The expressions (4.10) – (4.12) are substituted in the Lagrange functional (4.9).

$$L_a = K\Delta t^l + \lambda^l(K)^T\left[y(K)-\bar{y}\right]+\mu\left[y(K)-\bar{y}\right]^2 +$$
$$+\sum_{k=0}^{K-1}\left\{\lambda^l(k)^T\left[\rho^l(k)-(1+\Delta t^l A)y(k)-\Delta t^l By(k)F_R(k)-\Delta t^l B_1 F_R(k)-\Delta t^l Wy_f(k)\right]+\right.$$
$$\left.+(1/2)\mu\left[\rho^l(k)-(1+\Delta t^l A)y(k)-\Delta t^l By(k)F_R(k)-\Delta t^l B_1 y(k)-\Delta t^l Wy_f(k)\right]^2\right\}. \tag{4.13}$$

The expression (4.13) can then be fully decomposed in time domain. The necessary conditions for optimality can also be decomposed fully in time domain and these conditions are:

$$\frac{\partial L_a}{\partial y(k)}=0,\ \frac{\partial L_a}{\partial F_R(k)}=0,\ \frac{\partial L_a}{\partial \lambda(k)}=0,\ \frac{\partial L_a}{\partial \rho(k)}=0,\ \frac{\partial L_a}{\partial \Delta t}=0, \tag{4.14}$$

After differentiating the augmented Lagrangian according to every of the variables in equation (4.13) the necessary conditions can now be written as follows:

$$\frac{\partial La}{\partial y(k)} = -\left[(1+\Delta t^l A)^T+\Delta t^l BF_R(k)\right]\cdot$$
$$\cdot\left\{\lambda^l(k)+\mu\left[\rho^l(k)-(1+\Delta t^l A)y(k)-\Delta t^l By(k)F_R(k)-\Delta t^l B_1 F_R(k)-\Delta t^l Wy_f(k)\right]\right\}=$$
$$=e_y(k)=0, \tag{4.15}$$

$$\frac{\partial L_a}{\partial y(K)}=\lambda(K)+\mu\left[y(K)-\bar{y}\right]=e_y(K)=0 \tag{4.16}$$

$$\frac{\partial L_a}{\partial F_R(k)} = -\left[\Delta t^l y(k)^T B^T+\Delta t^l B_1^T\right]\cdot$$
$$\cdot\left\{\lambda^l(k)+\mu\left[\rho^l(k)-(1+\Delta t^l Ay(k)-\Delta t^l By(k)F_R(k)-\Delta t^l B_1 F_R(k)-\Delta t^l Wy_f(k)\right]\right\}=$$
$$=e_{F_R}(k)=0, \tag{4.17}$$

$$\frac{\partial L_a}{\partial \lambda^l(k)} = \rho^l(k)-(1+\Delta t^l A)y(k)-\Delta t^l By(k)F_R(k)-\Delta t^l B_1 F_R(k)-\Delta t^l Wy_f(k)=$$
$$=e_\lambda(k)=0, \tag{4.18}$$

$$\frac{\partial L_a}{\partial \lambda^l(K)} = y(K) - \bar{y} = e_\lambda(K) = 0, \tag{4.19}$$

$$\frac{\partial L_a}{\partial \rho^l(k)} = \lambda^l(k) + \mu\left[\rho^l(k) - (1+\Delta t^l A)y(k) - \Delta t^l By(k)F_R(k) - \Delta t^l B_1 F_R(k) + Wy_f(k)\right] =$$
$$= e_\rho(k) = 0. \tag{4.20}$$

The necessary conditions for optimality according to the sampling period are only calculated for the last period $k = K$, then $y(k) = y(K), F_R(K) = 0$. The augmented Lagrangian is:

$$L_a = K\Delta t^l + \lambda^l(K)^T\left[y(K) - \bar{y}\right] + \mu\left[y(K) - \bar{y}\right]^2. \tag{4.21}$$

Since $y(K)$ is a function of Δt, $y(K)$ is expressed by the model equation

$$y(K) = (1+\Delta t^l A)y(K-1) + \Delta t^l By(K-1)F_R(K-1) + \Delta t^l B_1 F_R(K-1) - \\ - \Delta t^l Wy_f(K-1), \tag{4.22}$$

$$L_a(K) = K\Delta t + \\ + \lambda(K)^T\left[(1+\Delta t^l A)y(K-1) - \Delta t^l By(K-1)F_R(K-1) + \Delta t^l B_1 F_R(K-1) + \Delta t^l Wy_f(K-1) - \bar{y}\right] + \\ + \mu\left[(1+\Delta t^l A)y(K-1) + \Delta t^l By(K-1)F_R(K-1) + \Delta t^l B_1 F_R(K-1) + \Delta t^l Wy_f(K-1) - \bar{y}\right]^2. \tag{4.23}$$

The necessary condition for optimality of the functional according to the sampling interval is:

$$\frac{\partial L_a(K)}{\partial \Delta t} = K + \lambda(K)^T\left[Ay(K-1) + By(K-1)F_R(K-1) + B_1 F_R(K-1) + Wy_f(K-1)\right] + \\ + \mu\left[(1+\Delta t^l A)y(K-1) + \Delta t^l By(K-1)F_R(K-1) + \Delta t^l B_1 F_R(K-1) + \Delta t^l Wy_f(K-1) - \bar{y}\right]^T \cdot \\ \cdot\left[Ay(K-1) + By(K-1)F_R(K-1) + B_1 F_R(K-1) + Wy_f(K-1)\right] = e_{\Delta t}(K) = 0. \tag{4.24}$$

Let

$$Ay(K-1) + By(K-1)F_R(K-1) + B_1 F_R(K-1) + Wy_f(K-1) = e(K-1) = 0. \tag{4.25}$$

The equation (4.24) can be expressed as:

$$K + \lambda(K)^T e(K-1) + \mu y(K-1)^T e(K-1) + \mu \Delta t e(K-1)^T e(K-1) - \mu \bar{y}^T e(K-1) = \\ = 0. \tag{4.26}$$

Equation (4.26) represents the necessary condition for optimality according to the sampling period. Δt appears linearly in this equation and can be expressed analytically by the equation:

$$\Delta t = \frac{-K - \lambda(k)^T e(K-1) - \mu y(K-1)^T e(K-1) + \mu \bar{y}^T e(K-1)}{\mu e(K-1)^T e(K-1)}. \tag{4.27}$$

The second derivative according to Δt is given by:

$$\frac{\partial L_a^2(K)}{\partial^2 \Delta t} = \mu e(K-1)^T e(K-1) > 0. \tag{4.28}$$

The second derivative of Δt is always positive which means that the sampling period determined from the necessary condition for optimality (4.26) is the optimal one. The condition (4.28) is sufficient for the minimization of the startup time.

4.2.4 Two Level Calculating Structure

Equations (4.15) – (4.21), (4.26) determine the necessary conditions for optimality of the minimum time control problem. The optimal solution is obtained when these equations are fulfilled. The values of the coordinating variables are set previously at the second level of the calculating structure by the coordinator. Then the necessary conditions for optimality for the state and control variables can be decomposed fully in time domain. In this way the initial problem is decomposed into $K+1$ sub-problems of the mathematical programming, everyone determined at separate moment of a discrete time k. The solutions of these sub-problems depend on the optimal values of the coordinating variables, then the solutions of the sub-problems will also be the optimal one on the bases of the connection of the solutions of *primal* and *dual* problems. For this reason a condition for reaching of the optimal solution of the initial problem is fulfillment of the

necessary conditions (4.19) – (4.21), (4.26). Then solution of the initial problem is done by solution of one coordinating problem on the second level and K+1 sub-problems on the first level.

4.2.4.1 Coordinating Sub-problem

The interconnections in time domain $\rho(k)$ can be directly expressed from equation (4.19). This equation represents the gradient of the functional of Lagrange towards the conjugate variables. It can be substituted in equation (4.21) and then lastly be written as:

$$\frac{\partial L_a}{\partial \rho^l(k)} = \lambda^l(k) + \frac{\partial L_a}{\partial \lambda^l(k)} = \lambda^l(k) + \mu e_\lambda^l(k). \tag{4.29}$$

Equation (4.29) represents the expression for a *gradient procedure* for calculation of the conjugate variables with a step equal to the penalty coefficient. Then the coordinating variables are calculated according to the following algorithm:

$$\lambda^{l+1}(k) = \lambda^l(k) + \mu e_\lambda^l(k), \tag{4.30}$$

$$e_\lambda^{\,l}(k) = \rho^l(k) - (1 + \Delta t^l A) y(k) - \Delta t^l B y(k) F_R(k) - \\ - \Delta t^l B_1 F_R(k) - \Delta t^l W y_f(k), \tag{4.31}$$

$$e_\lambda^{\,l}(K) = y^l(K) - \bar{y}, \tag{4.32}$$

$$\rho^{l+1}(k) = \rho^l(k) - \frac{\lambda^l(k)}{\mu}, \quad k = \overline{0, K-1}, \tag{4.33}$$

$$\Delta t^{l+1} = \frac{\mu \bar{y}^T e^l(K-1) - K - \lambda^{l,T}(K) e^l(K-1) - \mu y e^{l,T}(K-1) e^l(K-1)}{\mu e^{l,T}(K-1) e^l(K-1)}. \tag{4.34}$$

In (4.30) – (4.33) the values of $y(k)$ and $F_R(k)$ are obtained from the solutions of the first level sub-problems with a set from the coordinating level values of the coordinating variables. Coordinating process terminates upon satisfaction of the condition

$$\left\| e_{\lambda}^{l+1}(k) - e_{\lambda}^{l}(k) \right\| \le \varepsilon_{\lambda}, \varepsilon_{\lambda} > 0, \quad k = \overline{0,K}. \tag{4.35}$$

If these conditions are not satisfied, the coordinating problem is solved again with the new values of state and control variables obtained after solving the first level sub-problems with the new values of the coordinating variables and the new values of the penalty coefficient. Its value for the new iteration can be calculated by means of the gradients at the second level in order for the coordinating convergence to be better, according to the Algorithm 4.1, below.

Algorithm 4.1:

1) The error is computed

$$e^{l+1} = \left\{ \left\| e_{\lambda}^{l+1}(k) \right\|^2 + \left\| e_{\rho}^{l+1}(k) \right\|^2 + \left(e_{\Delta t}^{l+1} \right)^2 \right\}^{1/2}, \tag{4.36}$$

2) The new penalty coefficient is calculated from the conditions

   if $l = 1$ or $e^{l+1} < e^{l}$ then $\mu^{l+1} = \mu^{l}$,

   if $l > 1$ and $e^{l+1} \ge e^{l}$, then,

$$\mu^{l+1} = a\mu^{l}, \quad a = [0.1,\ 10.0]. \tag{4.37}$$

4.2.4.2 First Level Sub-problem

The first level sub-problems are determined according to the necessary condition for optimality (4.15) – (4.18), under the set from the second level values of the coordinating variables. These sub-problems can not be solved analytically and gradient procedures are used:

$$y^{l,j+1}(k) = y^{l,j}(k) - \alpha e_y^{l,j}(k), \quad k = \overline{0,K}, \tag{4.38}$$

$$\begin{aligned} e_y^{l,j} &= -\left[(1+\Delta t^l A)^T + \Delta t^l B^T F_R(k)\right]\left[\lambda^l(k) + \mu e_\lambda^l(k)\right] = \\ &= -\left[(1+\Delta t^l A)^T + \Delta t^l B^T F_R(k)\right]\left[\lambda^{l+1}(k)\right], \end{aligned} \tag{4.39}$$

$$e_y^{l,j}(K) = \lambda^l(K) + \mu\left[y^{l,j}(K) - \bar{y}\right], \tag{4.40}$$

$$F_R^{l,j+1}(k) = F_R^{l,j}(k) - \alpha e_{F_R}^{l,j}(k), \quad k = \overline{0,K-1}, \tag{4.41}$$

$$\begin{aligned} e_{F_R}^{l,j} &= -\left[\Delta t^l y^T(k) B^T + \Delta t^l B_1^T\right]\left[\lambda^l(k) + \mu e_\lambda^l(k)\right] = \\ &= -\left[\Delta t^l y^T(k) B^T + \Delta t^l B_1^T\right]\left[\lambda^{l+1}(k)\right]. \end{aligned} \tag{4.42}$$

The calculations of the trajectories for state and control continue until the conditions for the errors

$$\left\| e_y^{l,j+1}(k) - e_y^{l,j}(k) \right\| \le \varepsilon_y, \quad \varepsilon_y > 0, \quad k = \overline{0,K}, \tag{4.43}$$

$$\left\| e_{F_R}^{l,j+1}(k) - e_{F_R}^{l,j}(k) \right\| \le \varepsilon_{F_R}, \quad \varepsilon_{F_R} > 0, \quad k = \overline{0,K-1}, \tag{4.44}$$

are fulfilled.

The obtained values for the *sampling interval* and for the trajectories of state and control are projected over the constraint's domain (4.14), (4.15) respectively to account for the constraints:

$$v^{l,j} = \begin{cases} v_{\min}(k), v^{l,j}(k) < v_{\min}(k), & v = \Delta t, \\ v^{l,j}(k), v_{\min}(k) \le v^{l,j} \le v_{\max}(k), & v = F_R, \quad k = \overline{0,K-1}, \\ v_{\max}(k), v^{l,j}(k) > v_{\max}, & v = y, \quad k = \overline{0,K-1}. \end{cases} \tag{4.45}$$

The minimum time optimal control problem is solved in two level computing structure Fig.4.3 according to the following algorithm:

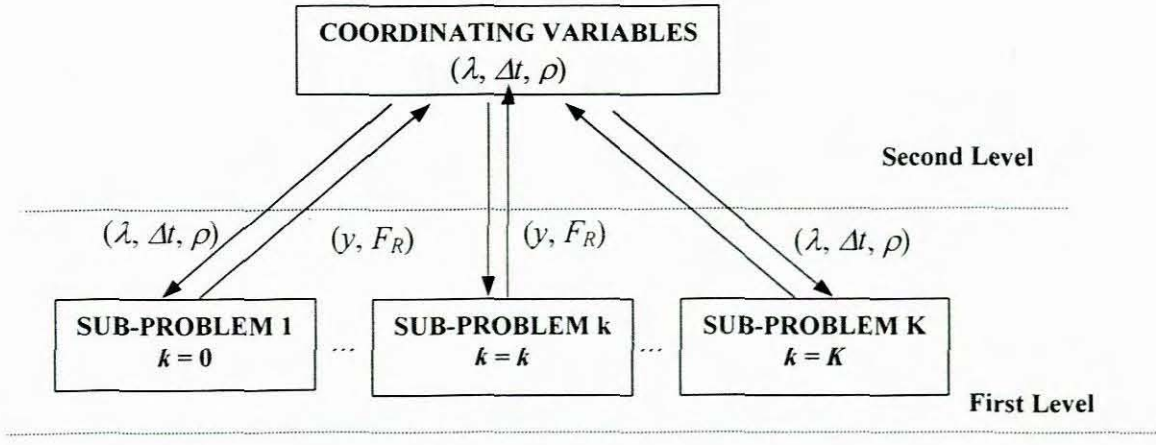


Figure 4.3. The Two Level Structure for Minimum Startup Time.

Algorithm 4.2:

1) at the second level:
   - the values of the numbers of steps K,
   - the number of iterations for the gradient procedures,
   - the values of the coordinating variables $\lambda(k)$, ρ, $k = \overline{0,K}$, Δt,
   - penalty coefficient μ,
   - errors ε_v,
   - Initial values of y, F_R are set up l =1.
2) at the first level the initial trajectory of the control $F_R(k)$, $k = \overline{0,K-1}$ is set and
   - the initial trajectory of the state $y(k)$, $k = \overline{0,K}$ is calculated,
   - the trajectories of the coordinating variables $\rho(k)$, $k = \overline{0,K-1}$ are determined from (4.6), j = 1.

3) at the first level the gradients $e_y^{l,j}(k)$, $k=\overline{0,K}$, $e_{F_R}^{l,j}(k)$, $k=\overline{0,K-1}$ and the new trajectories of the state and control are calculated from equations (4.34) – (4.38). They are projected over the constraints domain to equation (4.45).
4) at the first level the new gradients are calculated and the conditions (4.43), (4.44) are checked.
   - if they are satisfied the obtained trajectories for state and control are transferred to the second level
   - if not steps (3) and (4) are repeated with $j = j + 1$
5) At the second level the gradients (4.19) – (4.21) are calculated
   - the conditions in (4.40) are checked,
   - if they are satisfied ,the optimal solution of the coordinating subproblem and of the problem (4.2) – (4.8) is obtained,
   - if these conditions are not satisfied, new value for the penalty coefficient is calculated, steps (3), (4), (5) are repeated and so on.

4.3 PROBLEM FOR PROCESS CONTROL IN THE PRESENCE OF DISTURBANCES

4.3.1 Multilayer Control Structure

Continuous time technological processes have some parameters like temperature, level, concentration, pressure which can change occasionally under the influence of some slow disturbances (such as change in the ingredients of raw material). The slow varying disturbances lead to indefinite optimal control execution – they cause a drift to set-points. The well known solution to the problem for a control synthesis in the presence of slowly varying (parameters) disturbances of such nature is a feed-forward control. Disturbances change the initial condition, relevant to the steady state error, which is the base for the control synthesis. Application of PI (Proportional and Integral) control, robust controls requires the state and control to reach previously determined desired (nominal) steady state values. Unfortunately this requirement cannot be justified by a number of processes because the steady state and the control also depend on the disturbance (Athans, 1971; Lefkowitz, 1966). Therefore it becomes necessary to first find a control that can lead the control system to these new steady state values. The hierarchical approach to multilayer control is most suitable for this purpose. The following structure represents a hierarchical multilayer control system (Mesarovic, 1970; Roberts & Lin, 1991).

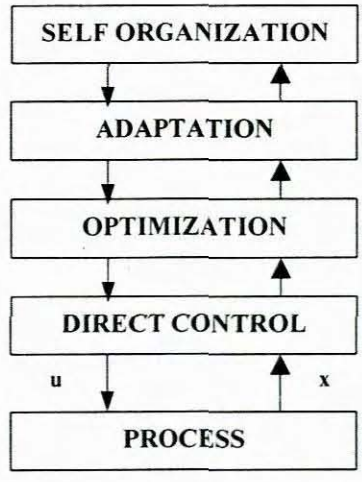


Figure 4.4. The Hierarchical Multilayer Control Structure.

The structure consists of four layers and their control functions are:

- self-organization
- adaptation
- optimization and
- direct control (the controller)

Combined action of all control layers aims at achieving one global optimization goal for long period of time. Decomposition of the whole problem for optimization can be done according to the frequency of the disturbances, acting over the system for the considered period:

- very low frequency – like month or year
- higher frequency – day or month
- very high frequency – minute or hour

At the self-organization layer, the low frequency of the disturbances is used to make changes in the structure of the system. At the adaptation layer, the model must be re-estimated using the 'new' (changed) structure, received from the self-organization layer (Skogestad, Halvorsen, Larsson & Govatsmark 1999). The control action at this layer is

towards changes in the process model. The problem is solved to achieve a *real time model adaptation* because the model changes every time the process experiences a disturbance which changes the model parameters. At the optimization layer, keeping in mind the optimal goals of the process, obviously with the change in the model structure, the optimal control must be resolved using the "new model". The changes in the disturbances in turn introduce a new set-point to the control system (y^{sp}). Therefore new parameters for the controller must be set using the new set-point. The direct control layer is where the new controller is designed and realized. A new error signal must be determined from the calculated new set-point coming from the optimization layer (Deshpande & Chen, 1993). The calculated error signal is then used by the controller which provides the control action (closing/opening of valves, starting/stopping of the pump). The layers are all interconnected by the measured values of the process variables.

4.3.2 The Problem for Optimal Control in the Presence of Slowly Varying Disturbances

The aim is to develop a unified methodology for synthesis of optimal control of multivariable systems which ensures action integrity of the separate layers for optimization and direct control to achieve a global criterion function, in the presence of slowly varying disturbances. This methodology is usually based on the approach of the *repetitive optimization*. The problem is decomposed into two subproblems for: (Celikovsky, 1993; Kozietulski, 1981, Rosenbrock, 1970)

1. steady state optimization and
2. steady state stabilization.

Decomposition of the global optimization problem into sub-problems depends on the frequencies of the disturbances (how often do they occur). The steady state optimization

deals with the determination of new optimal steady state values as introduced by disturbances. The values of its solution will directly depend on the values of the disturbances which can be considered constant for some interval of time i.e. until the next disturbance occurs. Each disturbance is considered piecewise constant as shown in the (Fig.4.5). The aim of the second sub-problem, the optimal stabilization, is to maintain the obtained optimal steady state of the process using its dynamic model. Based on the solution of the two sub-problems, the global aim is reached by layering of the controls from the two interconnected sub-layers and using the measured process output variables and slowly varying disturbances as a link between them.

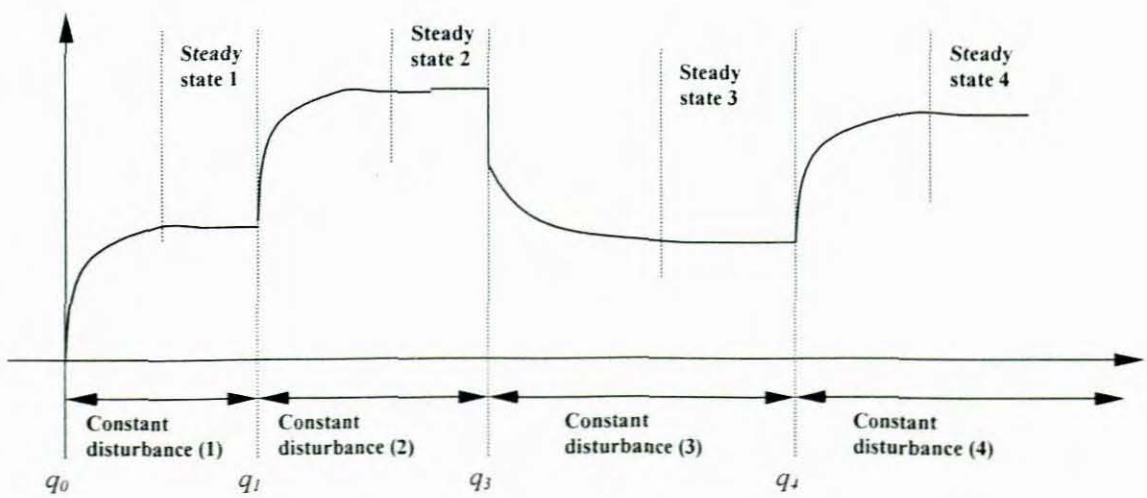


Figure 4.5. Slowly Varying Disturbances Effect on Steady State.

The considered two layer problems are solved for higher than in the adaptation layer frequency of disturbances and the solution of these problems force the plant (process) to follow the optimal steady state values. The steady state optimization problem is solved repetitively after long periods of time, at moments when the values of the main slowly varying disturbance is changed. The dynamic model sub-problems are solved:

- every time when the steady state optimization problem is solved, using the calculated optimal steady state values for recalculation of the controller parameters
- or are solved at the beginning of the process and then the same control parameters are used to minimize the error between the new steady state values of the variables and their real values.

The figure below shows the control structure based on the above strategy.

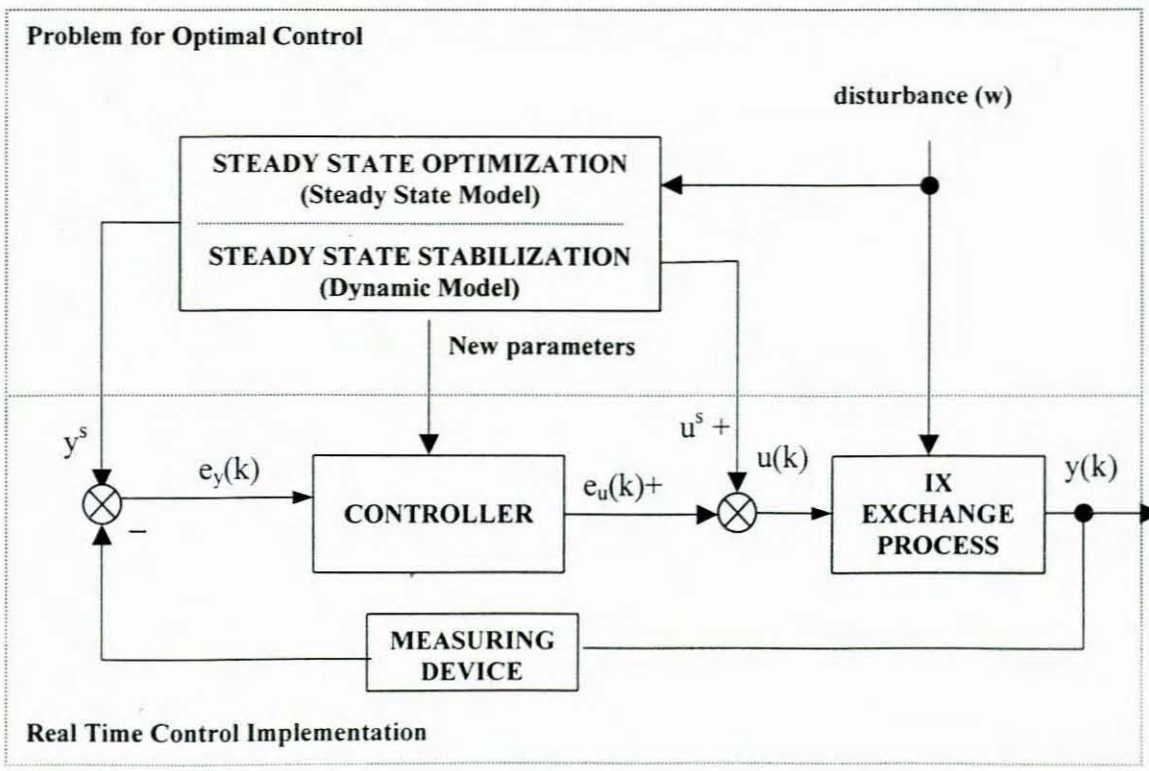


Figure 4.6. The Control Structure for Optimization of Steady State.

where, y^s and u^s are the optimal solutions of the optimization problem in steady state.

$e_y(k)$ is the error between the optimal steady state values and the real output of the process, $-e_y(k) = y^s - y(k)$,

$e_u(k)$ is the error between the optimal steady state control and the control to the plant, $-e_u(k) = u(k) - u^s$.

4.3.3 Formulation of the Problem for Optimal Control in the Presence of Slowly Varying Disturbances

Find the control,

$$F_R(k), \quad k = \overline{q, q+K+1}, \qquad i = \overline{1, N}, \tag{4.46}$$

which minimizes the functional,

$$J = \sum_{k=q}^{q+K-1} \frac{1}{2}\left[\left\| y(k) - y^{sp}(k) \right\|_Q^2 + \left\| F_R(k) - F_R^{sp}(k) \right\|_R^2 \right], \quad \begin{matrix} K \to \infty \\ K \neq \infty \end{matrix} \tag{4.47}$$

and satisfies the constraints

$$y(k+1) = \left[1 + \Delta t A\right] y(k) + \Delta t B\, y(k)\, F_R(k) + \Delta t B_1\, F_R(k) + \Delta t\, W y_f(k), \tag{4.48}$$

$$y(q) = y_q, \tag{4.49}$$

$$z(k) = y_N(k) = C\, y(k), \; C = \left[0 \quad 0 \quad \ldots \quad 0 \quad 1\right], \tag{4.50}$$

where $k = \overline{q, q+K-1}$,

and is determined at the current moment $q = 1,2,3,\ldots$.

The optimization problem (4.46) – (4.50) is characterized by the quadratic criterion (4.47) in which y^{sp} and F_R^{sp} are set-points for the state and the control respectively. They are determined as nominal values for the ion exchange process.

$\left.\begin{matrix} Q_i \in R^{N \times N} \\ R_i \in R^{1 \times 1} \end{matrix}\right|$, – are symmetrical (positively) definite weighing matrices,

q – is the current moment for the solution of the problem for the synthesis of the repetitive control. It represents the moments at which the slowly varying disturbance changes its value,

K – is the number of steps in the optimization problem,

y_q – is the initial condition of the state vector at the moments $q = 1,2,3\ldots$,

$y_f(k) = y_f = const$, $k = \overline{q, q+K-1}$ is the slowly varying disturbance,

$A \in R^{N \times N}, B \in R^{N \times N}, B_1 \in R^{N \times 1}$ *and* $W \in R^{1 \times 1}$ – are matrices of the model.

In the formulation of the problem it is assumed that:

1. the control plant (4.48), (4.50) is controllable and observable and works in real time,
2. the disturbance is determined - $y_f(k) = y_f$ = constant for $k \in [q, q+K-1]$ can be measured and remains constant during the whole period of $[q, q+K-1]$,
3. the initial condition y_q is determined by the actual measured state at the moment $q \geq 0$, $q = 0,1,2...$,
4. in each sequential moment $q \geq 0$, the optimal control can be determined at the period $[q, q+K-1]$.

4.3.4 Decomposition of the Steady State Problem into Two Layer Structure

When the determined disturbance, $y_f(k)$ is slowly varying:

$$y_f(k) = const, \qquad k = \overline{q, q+K-1}, \tag{4.51}$$

the optimal solution of the problem can be represented in the following way:

$$\begin{aligned} y(k) &= y^s + e_y(k) && \text{– state,} \\ F_R(k) &= F_R^s + e_{F_R}(k) && \text{– control,} \\ z(k) &= z^s + e_z(k) && \text{– output,} \end{aligned} \tag{4.52}$$

Expression (4.52) represents the current state, control and output of the process as the sum of the steady state and dynamic components. The obtained model identification model must now be decomposed according to the two-layer structure so that it can be used in solving the optimal control problem. The aim is to decompose the model into two models that can be used separately thus making the model amenable (Popchev. & Tsoneva, 1993, 1990).

Substitution of the state, control and output (4.52) into the dynamical equations for the model (4.48) – (4.50) can be represented in the following way:

$$y^s + e_y(k+1) = (I + \Delta tA)\left[y^s + e_y(k)\right] + \Delta tB\left[y^s + e_y(k)\right]\left[F_R{}^s + e_{F_R}(k)\right] + \\ + \Delta tB_1\left[F_R{}^s + e_{F_R}(k)\right] + \Delta t\, Wy_f, \qquad (4.53)$$

$$z^s + e_z(k) = C\left[y^s + e_y(k)\right].$$

From the equation (4.53),

$$y^s + e_y(k+1) = Iy^s + \Delta tA\, y^s + I\, e_y(k) + \Delta tA\, e_y(k) + \\ + \left[\Delta tB\, y^s + \Delta tB\, e_y(k)\right]\left[F_R{}^s + e_{F_R}(k)\right] + \qquad (4.54) \\ + \Delta tB_1 F_R^s + \Delta tB_1 e_{F_R}(k) + \Delta tWy_f =$$

$$= Iy^s + \Delta tAy^s + Ie_y(k) + \Delta tAe_y(k) + \Delta tBy^s F_R{}^s + \Delta tBy^s e_{F_R}(k) + \Delta tBe_y(k)F_R{}^s + \\ + \Delta tBe_y(k)e_{F_R}(k) + \Delta tB_1 F_R{}^s + \Delta tB_1 e_{F_R}(k) + \Delta t\, Wy_f = \\ = \left[I + \Delta tA\right]y^s + \left[I + \Delta tA + \Delta tBF_R{}^s\right]e_y(k) + \Delta tBe_{F_R}(k)e_y(k) + \qquad (4.55) \\ + \left[\Delta tBy^s + \Delta tB_1\right]e_{F_R}(k) + \Delta tBy^s F_R{}^s + \Delta tB_1 F_R{}^s + \Delta t\, Wy_f,$$

where, y^s, F_R^s, z^s - are the components of the optimal solution of the initial problem (4.46) – (4.50) which depend on the slowly varying disturbance.

$e_y(k)$, $e_{F_R}(k)$, $e_z(k)$ – are the components expressing the transitional behaviour of the process moving from the steady state values of the variables at the moment (q-1) to the values of the steady state variable at the moment q – Fig.4.7.

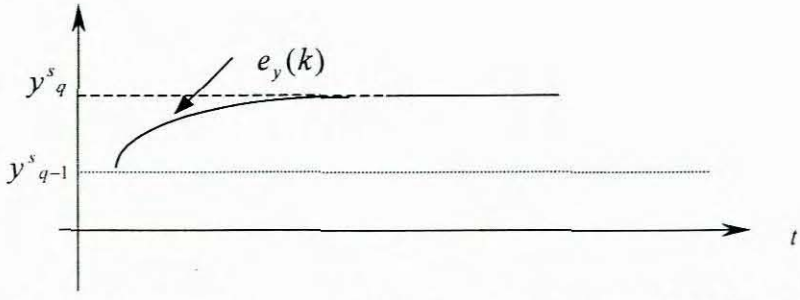


Figure 4.7. Calculation of the Error for Newly Reached Steady State.

As the values of $e_y(k)$, $e_{F_R}(k)$, $e_z(k)$ do not depend on the action of the disturbances, the solution of the problem (4.46) – (4.50) can be simplified by solving two separate subproblems, relevant to the different types of components. From here the initial model can be decomposed into steady state model and the model of deviations from the steady state – the dynamic model (Shacham, 1984). From the initial decomposition equation (4.54), the process model can be represented as a sum of two models:

- steady state (4.56) and dynamic model (4.57).

$$\begin{aligned} y^s &= [I + \Delta t A] y^s + \Delta t B y^s F_R^s + \Delta t B_1 F_R^s + \Delta t W y_f, \\ z^s &= C y^s, \end{aligned} \tag{4.56}$$

$$\begin{aligned} e_y(k+1) = [I + \Delta t A + \Delta t B F_R^s]\, e_y(k) + \\ + \left[\Delta t B y^s + \Delta t B_1\right] e_{F_R}(k) + \Delta t B e_y(k)\, e_{F_R}(k), \end{aligned} \tag{4.57}$$

$$e_z(k) = C\, e_y(k).$$

Both models after the decomposition of the initial model are bilinear ones. The steady state model can also be written in the form:

$$\begin{aligned} y^s &= y^s + \Delta t A y^s + \Delta t B y^s F_R^s + \Delta t B_1 F_R^s + \Delta t W y_f, \\ 0 &= \Delta t A y^s + \Delta t B y^s F_R^s + \Delta t B_1 F_R^s + \Delta t W y_f, \\ A y^s &+ B y^s F_R^s + B_1 F_R^s + W y_f \approx 0, \end{aligned} \tag{4.58}$$

This model shows that the deviations approach zero as the process approaches steady state. As the disturbance y_f can be considered piecewise constant, the steady state optimization problem can be solved where the solution will depend on the value of the disturbance, as y^s, and $F_R{}^s$ in equation (4.58) directly depend on the value of y_f.

The criterion also is decomposed into two parts - for steady state optimization and for the dynamic stabilization.

$$J = J_s + J_d =$$
$$= \tfrac{1}{2}\left[\left\|y^s - y^{sp}\right\|_Q^2 + \left\|F_R{}^s - F_R^{sp}\right\|_R^2\right] + \tfrac{1}{2}\sum_{k=q}^{q+K-1}\left[\left\|e_y(k) - y^s\right\|_Q^2 + \left\|e_{F_R}(k) - F_R{}^s\right\|_R^2\right]. \tag{4.59}$$

Then using both models and criteria separate problems for a steady state optimization and dynamic stabilization can be solved. The problems are formulated as problems with quadratic criterion function and bilinear models.

4.3.5 The Problem for Steady State Optimization

4.3.5.1 Problem Formulation

The steady state optimization problem is formulated as follows:

Find the control $F_R{}^s$ in such a way that,

$$J_s = 1/2\left[\left\|y^s - y^{sp}\right\|_Q^2 + \left\|F_R{}^s - F_R{}^{sp}\right\|_R^2\right], \tag{4.60}$$

is minimum under the constrains,

$$\begin{aligned} &Ay^s + By^s F_R{}^s + B_1 F_R{}^s + Wy_f = 0, \\ &z^s = C\,y^s. \end{aligned} \tag{4.61}$$

4.3.5.2 Problem Solution

The solution is based on the function of Lagrange

$$L_s = 1/2\left[\left\|y^s - y^{sp}\right\|_Q^2 + \left\|F_R{}^s - F_R{}^{sp}\right\|_R^2\right] + \lambda_s{}^T\left[Ay^s + By^s F_R{}^s + B_1 F_R{}^s + Wy_f\right], \qquad (4.62)$$

where, $\lambda_s \in R^N$ - is the vector of the Lagrange multipliers.

This problem is a *convex one* as the criterion function is characterized by quadratic terms according to y^s and $F_R{}^s$. The solution of the problem can be found from the necessary conditions for optimality of the Lagrange function. In the considered case the conditions are also sufficient and the obtained solution is unique. The necessary conditions for optimality are:

$$\frac{\partial L_s}{\partial y^s} = 0, \quad \frac{\partial L_s}{\partial F_R{}^s} = 0, \quad \frac{\partial L_s}{\partial \lambda_s} = 0 \cdot \qquad (4.63)$$

From the equation of the Lagrangian these conditions are derivative of *Ls* towards y^s $F_R{}^s$ and λ_s.

$$\frac{\partial L_s}{\partial y^s} = Q(y^s - y^{sp}) + A^T\lambda_s + B^T\lambda_s F_R{}^s = 0 = e_{y^s}, \qquad (4.64)$$

$$\frac{\partial L_s}{\partial F_R{}^s} = R(F_R^s - F_R^{sp}) + (By^s)^T\lambda_s + B_1{}^T\lambda_s = 0 = e_{F_R}, \qquad (4.65)$$

$$\frac{\partial L_s}{\partial \lambda_s} = Ay^s + By^s F_R{}^s + B_1 F_R{}^s + Wy_f = 0 = e_{\lambda_s}. \qquad (4.66)$$

The solution of the obtained nonlinear system of equations will determine the optimal solution of the steady state problem. The system (4.64) – (4.66) has three equations with three unknown variables, y^s, $F_R{}^s$ and λ_s. y^s and $F_R{}^s$ are solutions of the primal (initial) problem and λ_s is the solution of the dual problem. Because the solution of the primal problem y^s and $F_R{}^s$ depends on the values of the Lagrange multipliers the calculation procedure could be built using the principle of coordination of the aims of the subproblems, where the aims are represented by the subfunctions of the Lagrange

function in which λ_s has some previously given value. This means that if the optimal solution for λ_s is obtained, the optimal solutions for y^s and F_R^s can be obtained. For this purpose it becomes necessary to introduce some hierarchical computing structure, which will realize this strategy of the solution of the system of equations. This structure is shown in the Fig.4.8.

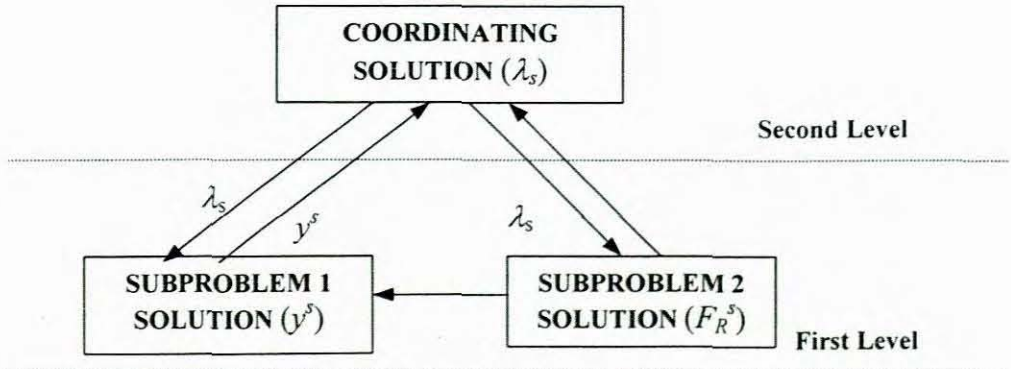


Figure 4.8. Two-Level Calculating Structure for Steady State Model.

In this way the calculation is decomposed into two levels:

- Second level (one sub-problem) – this is coordinating the solutions of the other sub-problems in order to receive the optimal solution of the optimization steady state problem.
- First level (two sub-problems) – calculating for every value of the coordinating variable λ_s the values of the state and the control.

In the coordinating sub-problem (Second Level), the solution is based on the necessary condition for optimality (4.66). As analytical solution for the dual variable is not possible to be obtained, the gradient method will be used in the form:

$$\lambda_s^{(j+1)} = \lambda_s^{(j)} + \alpha^{(j)} e_{\lambda_s}^{(j)} \tag{4.67}$$

where j – is the index of the gradient procedure.

The process of calculation of λ_s is gradient one with $\alpha^{(j)}$ step of calculation procedure;

- $e_{\lambda s}{}^{(j)}$ is the value of the gradient function of Lagrange at the j^{th} iteration and j is the index of iteration.
- The value of the gradient $e_{\lambda s}{}^{(j)}$ gives the direction of the search of maximum of Lagrange function towards λ_s. Its j^{th} value is obtained using the values of the state y^s and the control F_R^s at each j^{th} iteration.

$$e_{\lambda_s}^{(j)} = Ay^{s(j)} + By^{s(j)}F_R{}^{s(j)} + B_1F_R{}^{s(j)} + Wy_f\,. \tag{4.68}$$

The optimal solution will be obtained when $e_{\lambda_s}{}^{(j)}$ is very close to zero, or

$$\left\|e_{\lambda_s}^{(j)}\right\|^2 \le \varepsilon \rightarrow e^{(j)}{}_{\lambda_s}{}^T e^{(j)}{}_{\lambda_s} \le \varepsilon\,, \tag{4.69}$$

where, $\varepsilon > 0$ - is a small (positive) value of the error.

When the norm of the error is smaller than ε the necessary condition for optimality of λ_s has been fulfilled.

In the sub-problems for the state and the control (First Level), the values of $\lambda_s^{(j)}$ are substituted in the equation of the necessary conditions for y_s and $F_R{}^s$. Then y^s and $F_R{}^s$ are obtained analytically from equations (4.64) – (4.66) in the following order:

$$Q(y^s - y^{sp}) + A^T\lambda_s + B^T\lambda_s F_R^s = 0\,, \tag{4.70}$$

$$\begin{aligned} y^s - y^{sp} &= -Q^{-1}A^T\lambda_s - Q^{-1}B^T\lambda_s F_R^s\,, \\ y^{s(j)} &= y^{sp} - Q^{-1}A^T\lambda_s^{(j)} - Q^{-1}B^T\lambda_s^{(j)}F_R^{s(j)}\,. \end{aligned} \tag{4.71}$$

This expression is substituted in the equation for $F_R{}^s$ and $F_R{}^s$ is then expressed by other variables.

$$\begin{aligned} &R(F_R{}^s - F_R{}^{sp}) + (By^s)^T\lambda_s + B_1{}^T\lambda_s = 0, \\ &R(F_R{}^s - F_R{}^{sp}) + (y^s)^T B^T\lambda_s + B_1{}^T\lambda_s = 0, \\ &RF_R^s - RF_R^{sp} + (y^s)^T B^T\lambda_s + B_1{}^T\lambda_s = 0, \\ &RF_R^s - RF_R^{sp} + \left[y^{sp} - Q^{-1}A^T\lambda_s - Q^{-1}B^T\lambda_s F_R{}^s\right]^T B^T\lambda_s + B_1{}^T\lambda_s = 0, \end{aligned}$$

$$RF_R^s - RF_R^{sp} + \\ +(y^{sp})^T B^T \lambda_s - \lambda_s{}^T AQ^{-1}B^T \lambda_s - F_R{}^s \lambda_s BQ^{-1}B^T \lambda_s + B_1{}^T \lambda_s = 0. \tag{4.72}$$

From here F_R^s can be expressed.

$$F_R{}^{s(j)} = (R - \lambda_s{}^{T(j)} Q^{-1} B^T \lambda_s^{(j)})^{-1} RF_R{}^{sp} + \\ +(R - \lambda_s{}^{T(j)} Q^{-1} B^T \lambda_s^{(j)})^{-1} \left[(y^{sp})^T B^T \lambda_s^{(j)} + \lambda_s{}^{T(j)} AQ^{-1} B^T \lambda_s^{(j)}\right] - B_1{}^T \lambda_s^{(j)}. \tag{4.73}$$

This equation gives the value of F_R^s using only the values of the weighting matrices R and Q, the matrices of the model and the value of the dual variable, λ_s. When F_R^s is calculated its value is set to the expression for y^s. It is possible that the analytical solution for F_R^s to be substituted in (4.71). Then the second and first sub-problems will not be connected by F_R^s signal and y^s will be given fully independently by the equation:

$$y^{s(j)} = y^{sp} - Q^{-1} A^T \lambda_s^{(j)} - Q^{-1} B^T \lambda_s^{(j)} \left\{\left(R - \lambda_s^{T(j)} Q^{-1} B^T \lambda_s^{(j)}\right)^{-1} RF_R^{sp} + \right. \\ \left. +\left(R - \lambda_s^{T(j)} Q^{-1} B^T \lambda_s^{(j)}\right)^{-1} \left[(y^{sp})^T B^T \lambda_s^{(j)} + \lambda_s^{T(j)} AQ^{-1} B^T \lambda_s^{(j)}\right] - B_1^T \lambda_s^{(j)}\right\} \tag{4.74}$$

The obtained values are used for calculation of the gradient of λ_s, ($e_{\lambda s}$). The gradient is then used for the calculation of the error. The algorithm for calculations in the two-level structure is given, in Algorithm 4.3, below.

4.3.5.3 Algorithm for Calculation of Steady State Optimization Subproblem

Algorithm 4.3:

1) Set initial value of $\lambda_s^{(j)}, j = 1$

   $\lambda_{s_i}{}^{(1)} = \lambda_{si0}, \qquad i = \overline{1, N},$

   where, N – is the number of stages.

2) Set the index, j for coordination procedure

   $j = 1, \qquad j = \overline{1, M},$

where, M - is a certain big number of step of the procedure, necessary for conversion of the calculations towards the optimal value of the criterion.

3) Calculate $F_R^{s(j)}$ from the obtained equation

$$F_R^{s(j)} = (R - \lambda_s^{T(j)} Q^{-1} B^T \lambda_s^{(j)})^{-1} R F_R^{sp} -$$
$$-(R - \lambda_s^{T(j)} B Q^{-1} B^T \lambda_s^{(j)})^{-1} \left[(y^{sp})^T B^T \lambda_s^{(j)} - \lambda_s^{T(j)} A Q^{-1} B^T \lambda_s^{(j)} + B_1^T \lambda_s^{(j)} \right].$$

4) Calculate $y^{s(j)}$ from the equation

$$y^{s(j)} = y^{sp} - Q^{-1} A^T \lambda_s^{(j)} + B^T \lambda_s F_R^{s(j)}.$$

5) Calculate the value of the gradient $\alpha^{(j)}$ $\lambda_s^{(j)}$

$$e_{\lambda_s}^{(j)} = A y^{s(j)} + B y^{s(j)} F_R^{s(j)} + B_1 F_R^{s(j)} + W y_f,$$

Check for the condition for the end of the coordinating gradient procedure:

a) if the condition is satisfied – stop the calculation. The obtained solution is the optimal one,

$$y^s = y^{s(j)},$$
$$F_R^s = F_R^{s(j)},$$
$$\lambda_s = \lambda_s^{(j)},$$

b) if not - then calculate:

$$\lambda_s^{j+1} = \lambda_s^j + \alpha^j e_{\lambda_s}^{(j)}.$$

6) Set the next value of j

$j = j + 1$,

check if $j \le M$:

a) if $j \le M$ go to number (3) and repeat all calculations with the new values for λ_s^{j+1},

b) if $j > M$ - stop the calculation.

7) Calculate T^s – the time of the up-flow period – equation (3.80).

$$T^s = \frac{hd}{F_R^s}$$

where h – is the resin hold-up

d = 2/3 – is the amount of resin going from one stage to another during the pull-down period

- **Up-flow Time Determination**

The resin flow rate depends on cycle time and the resin volume pull-down which in turn depends on the resin holdup (inventory) of the stages. The resin rate is usually 2/3 and not more than ninety percent (90%) of the holdups in order to prevent back mixing of the resin. The stage holdup depends on *stage volume* and *resin bed expansion* (BE), which is determined by the liquid flow rate.

The resin flow rate in the load column and its corresponding regeneration column must be balanced (equal), so that each column in pair must be designed with the other in mind. From the equations (1.2), (3.80) the following conclusion can be made:

$$F_R^{SR} = F_R^S \tag{3.80}$$

$$F_R^{SR} T^{SR} = h_R d_R$$

$$F_R^S T^S = hd$$

$$F_R^{SR} = F_R^S = \frac{h_R d_R}{T^{SR}} = \frac{hd}{T^S}$$

If $d_R = d$ and $h_R < h$ as the diameter of the regeneration column is smaller, then T^{SR} has to be smaller than T^S in the same proportion.

4.3.5.4 LabVIEW Program for Solution of the Optimization Problem and Real Time Control Implementation

1. Optimization Problem Solution
   a) Program Inputs (variables)
      - set-points y^{sp}, F_R^{sp} ,
      - values of the matrices of the model A, B, B_l, W,
      - values of the weighted matrices Q and R,
      - number of steps in the optimization interval - K ,
      - initial value of the dual variable λ_s ,
      - measured value of the concentration of NaCl in input flow, given by pH and conductivity measurements (y_f^S).

   b) Program Outputs
      - the value of the up-flow time (period) T^s - sent to the file for use for the change in up-flow period.

2. Program for Implementation of the Control in real time
   - The program reads the value of T^s from the file and,
   - opens and closes the relevant solenoid valve according to the requirement as determined by T^s.

4.3.6 The Dynamic Optimization Sub-problem – Problem for Design of the Closed Loop Control

4.3.6.1 Problem Formulation

The problem for design of closed loop design is formulated for the dynamic part of the model, the part describing the deviation of states from the steady states. The criterion is the dynamic part of the common criterion. The problem is:

Find the control $e_{F_R}(k), \quad k = \overline{0, K-1}$ such that the criterion

$$J_d = \left\{ \tfrac{1}{2} \sum_{q=0}^{q+K-1} \left[\left\| e_y(k) \right\|_Q^2 + \left\| e_{F_R}(k) \right\|_R^2 \right] \right\} + \tfrac{1}{2} \left\| e_y(q+K) \right\|_S, \tag{4.75}$$

is minimized under the constraints,

$$e_y(K+1) = \left[I + \Delta t A + \Delta t B F_R^s \right] e_y(k) + \Delta t B e_y(k) e_{F_R}(k) + \left[\Delta t B y^s + \Delta t B_1 \right] e_{F_R}(k) \tag{4.76}$$

$$\begin{aligned} e_y(q) &= e_{y_q}, \\ e_z(k) &= C e_y(k). \end{aligned} \tag{4.77}$$

The problem (4.75) – (4.76) is nonlinear one as the model equation is bilinear. It is not possible to design equation for the linear state space controller. The approach that can be accepted is first to linearize the closed loop system by nonlinear control, and then to design linear controller for the linear closed loop system which will make the whole system optimal (Rosenbrock, 1970).

4.3.6.2 Nonlinear Control Design Approach

Recently, a general theory for the design of nonlinear feedback controllers has been developed based on the concepts from differential geometry (Isidori, 1996, Kravaris and Kantor, 1990 a, b, Krener, 1984). Using nonlinear coordinate transformation and nonlinear state feedback, the original nonlinear model and linear control techniques can then be employed. The system can be linearized in either an input-output sense or a state space sense.

However there are few reports that evaluate effectiveness of the linearization methods. There are well known criticisms and potential drawbacks of feedback linearization (Henson and Seborg, 1991). The controller may generate unnecessarily large control effort to cancel nonlinearities and it can not be optimal with respect to any criterion. The effect of parameter variations on the controlled variables destroys the linearizing effect of the controller and closed loop system has poor robustness properties (Frecman and Kokotovic, 1995).

In the past years the optimal and robust control theories have attracted many researchers attention and have obtained many developments. When the plant is highly nonlinear, it is reasonable to design a controller, based on its nonlinear model, which reflects the structural nonlinearities. At the same time some compensation of the parameter uncertainties is necessary. Therefore a total controller design method is needed which include both nonlinear and linear compensations. Some papers in the last years considered and solved this problem. The qualities of nonlinear controller have been improved by design of linear control of the linearized system based on quadratic criterion (Stroz and Gillers, 1995). Lyapunov's functions (Frecman and Kokotovic, 1995), integral actions (Soroush, 1995), quadratic stabilization (Hull, Schumacher, & Qu, 1995), H∞ control synthesis (Sugie, Simizu, & Imura, 1993).

The purpose of the dissertation is to propose a robust controller design method for the ion exchange process. In order to cope with both the parameter uncertainty and the

structured nonlinearity of the process, the proposed method consists of linear steady state optimization that ensures robust optimal control and a nonlinear compensation that achieves state space linearization of the closed loop with the nonlinear controller process. The structure of the closed loop system is shown on Fig. 4.9.

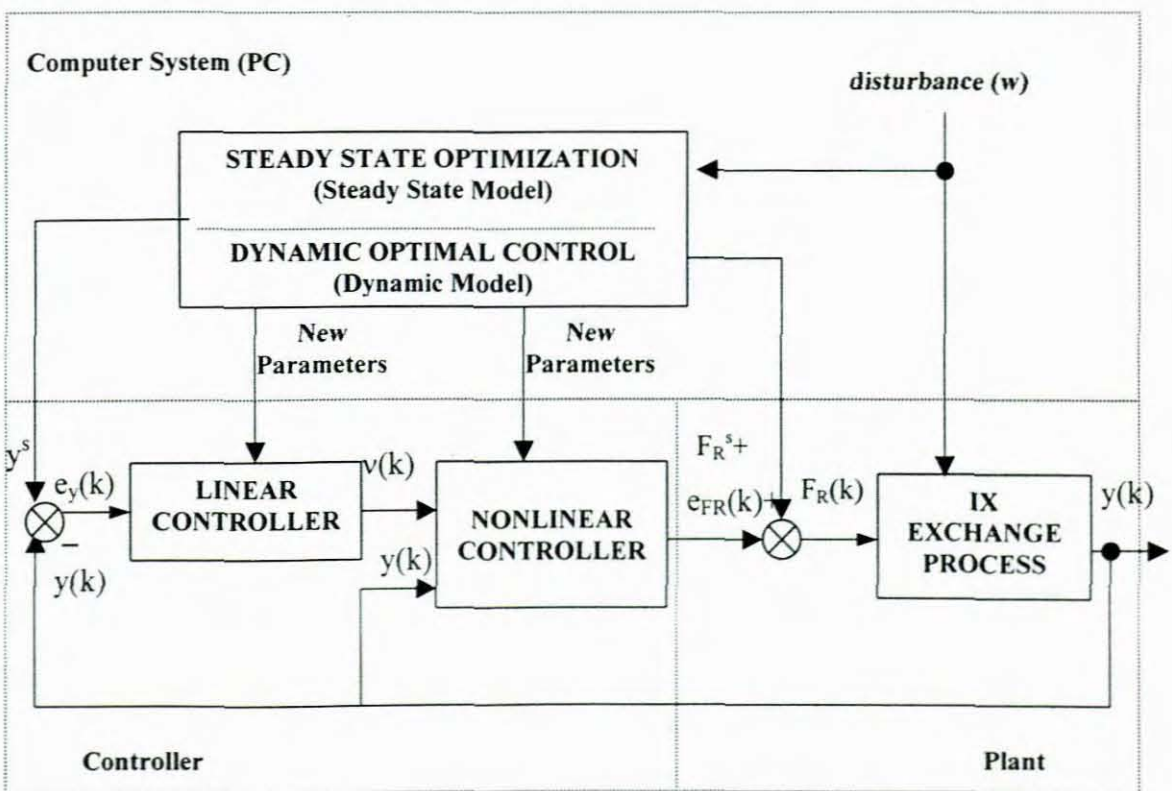


Figure 4.9. The Closed Loop Structure for Dynamic Model Optimization.

The method consists of two steps:

1. Nonlinear controller synthesis on the basis of the dynamic model of the process through state space linearization.
2. Linear control synthesis on the basis of the quadratic criterion and linearized model of the closed loop nonlinear system.

4.3.6.3 Nonlinear Control Formulation

The state space linearization control design consists of calculating a state feedback that transforms the nonlinear initial system into a decoupled linear system.

The problem is to find a static state feedback law of the form:

$$e_{F_R}(k) = \varphi[\cdot] + [\cdot]\nu(k),$$
$$[\cdot] = [y(k), A, B, B_1, W, \Delta t, y^s, F_R^s]. \tag{4.78}$$

such that:

1) The system (4.76), (4.77) is locally stable around the steady state point y^s, F_R^s.
2) The tracking error $y^s - y(k)$ is governed by a pre-specified stable linear model, called *reference model.*
3) The closed loop system (4.76), (4.77), (4.78) is robust in some limits towards, parameter variation,

where $\nu(k)$ – is an external reference signal for the nonlinear controller,

φ and β – are smooth vector functions in a neighbourhood of the set-point and,

$\beta[\cdot] \neq 0$

The aim is the map between the input $\nu(k)$ and the state vector $y(k)$ to be linear, equal to the reference model.

The applicability conditions for the linearization of multi-variable systems by nonlinear control (Isadori, 1995):

- the system must have a well defined vector relative degree,
- the system must be minimum phase,

are fulfilled. It is assumed that the process is completely controllable and all states are accessible to feedback control.

The problem is to synthesize the control in such a way that the process dynamics exactly tracks desired ones, given by the reference model.

$$e_y(k+1) = Le_y(k) + v(k), \tag{4.79}$$

$$e_-(k) = e_y(k), \tag{4.80}$$

where L – is the diag$\{li\}$, li = const, $i = \overline{1, N}$

$L \in R^{NxN}$ – is the constant matrix, given by the desired dynamics.

4.3.6.4 Nonlinear Control Problem Solution

The right part of the equations (4.76) and (4.79) are equalized

$$[I + \Delta tA + \Delta tBF_R^s]e_y(k) + \Delta tBe_y(k)e_{F_R}(k) + [\Delta tBy^s + \Delta tB_1]e_{F_R}(k) =$$

$$= Le_y(k) + v(k). \tag{4.81}$$

The nonlinear control can be expressed as a function of the state space after some algebraic transformation of equation (4.81).

$$\left[1 + \Delta tA + \Delta tBF_R^s\right]e_y(k) + \left[\Delta tBe_y(k) + \Delta tBy^s + \Delta tB_1\right]e_{F_R}(k) =$$

$$= Le_y(k) + v(k), \tag{4.82}$$

$$\left[\Delta tBe_y(k) + \Delta tBy^s + \Delta tB_1\right]e_{F_R}(k) =$$

$$= Le_y(k) - \left[1 + \Delta tA + \Delta tBF_R^s\right]e_y(k) + v(k). \tag{4.83}$$

Both sides from the left of (4.83) are multiplied by $\left[\Delta tBe_y(k) + \Delta tBy^s + \Delta tB_1\right]^T$,

$$\left[\Delta tBe_y(k) + \Delta tBy^s + \Delta tB_1\right]^T\left[\Delta tBe_y(k) + \Delta tBy^s + \Delta tB_1\right]e_{F_R}(k) =$$

$$= \left[\Delta tBe_y(k) + \Delta tBy^s + \Delta tB_1\right]^T\left[L - 1 + \Delta tA + \Delta tBF_R^s\right]e_y(k). \tag{4.84}$$

The matrix $\left[\Delta tBe_y(k) + \Delta tBy^s + \Delta tB_1\right]^T\left[\Delta tBe_y(k) + \Delta tBy^s + \Delta tB_1\right]$ is a square matrix and let us suppose that its inverse exists. Then the control can be expressed as,

$$e_{F_R}(k) = M\left[L - 1 + \Delta tA + \Delta tBF_R^s\right]e_y(k) + Mv(k), \tag{4.85}$$

$$M = \left\{\left[\Delta tBe_y(k) + \Delta tBy^s + \Delta tB_1\right]^T \left[\Delta tBe_y(k) + \Delta tBy^s + \Delta tB_1\right]\right\}^{-1} \cdot \cdot \left[\Delta tBe_y(k) + \Delta tBy^s + \Delta tB_1\right], \tag{4.86}$$

Equations (4.85), (4.86) represent the nonlinear control. It depends on the values of desired linear dynamics L, the reference input v and the current error $e_y(k)$. This control can be realized in real time after measurement of $u(k)$.

It can be seen that the control e_{F_R} is very sensitive to variations in the model parameters, and also on the values of previously calculated y^s and F_R^s.

The closed loop system (4.76), (4.85), (4.86) is linearized. It has linear dynamics and it is possible to use optimal control theory to find the external reference ν such that the error $e_y(k)$ is minimized and the control reference signal is also minimized.

4.3.6.5 Synthesis of Linear Stabilization Control

In equation (4.85) the value of the reference signal is not known. It can be obtained as linear optimal control according to the criterion (4.75) and the reference model. The problem can be formulated in the following way:

Find the control

$\nu(k)$, $k = \overline{0, K-1}$ which minimizes the functional

$$J = \tfrac{1}{2}\left\{\sum_{q=0}^{q+K+1} \left\|e_y(k)\right\|_Q^2 + \left\|\nu(k)\right\|_R^2\right\} + \tfrac{1}{2}\left\|e_y(q+K)\right\|_S^2, \tag{4.87}$$

under the constraints

$$e_y(k+1) = Le_y(k) + \nu(k), \tag{4.88}$$

$$e_y(q) = e_{y_{q0}}. \tag{4.89}$$

The solution of the optimal control problem is based on the functional of Hamilton

$$H = \sum_{q=q}^{q+K-1} \tfrac{1}{2} \left\{ \|ey(k)\|_Q^2 + \|v(k)\|_R^2 + p^T(k)\left[Ley(k) + v(k)\right] \right\}, \tag{4.90}$$

where $p(k) \in R^n$ – is the conjugate variables' vector.

The optimal control is found on the basis of the necessary condition for optimality of H

$$\frac{\partial H}{\partial v(k)} = 0 = Rv(k) + p(k+1), \tag{4.91}$$

$$\frac{\partial H}{\partial e_y(k)} = p(k) = Qe_y(k) = L^T p(k+1), \tag{4.92}$$

$$\frac{\partial H}{\partial e_y(K)} = Se_y(K) = p(K), \tag{4.92a}$$

$$\frac{\partial H}{\partial p(k+1)} = e_y(k+1) = Le_y(k) + v(k). \tag{4.93}$$

The control v(k) can be expressed from (4.91)

$$v(k) = -R^{-1} p(k+1). \tag{4.94}$$

Then equation (4.94) is substituted in (4.93)

$$e_y(k+1) = Le_y(k) - R^{-1} p(k+1). \tag{4.95}$$

Equations (4.92) and (4.95) represent the two point boundary value problem. As the process is represented by a linear system, the above problem can be solved by the substitution

$$p(k) = G(k)e_y(k), \tag{4.96}$$

where $G(k)$ – is the symmetrical matrix.

Then

$$p(k+1) = G(k+1)e_y(k+1). \tag{4.97}$$

The expressions (4.96) and (4.97) are substituted in (4.93) and (4.95)

$$G(k)e_y(k) = Qe_y(k)L^T G(k+1)e_y(k+1), \tag{4.98}$$

$$e_y(k+1) = Le_y(k) - R^{-1}G(k+1)e_y(k+1), \tag{4.99}$$

From the last equation (4.99)

$$\left[I + R^{-1}G(k+1)\right]e_y(k+1) = Le_y(k), \tag{4.100}$$

$$e_y(k+1) = \left[I + R^{-1}G(k+1)\right]^{-1} Le_y(k), \tag{4.101}$$

equation (4.101) is then substituted in (4.98)

$$G(k)e_y(k) = Qe_y(k) + L^T G(k+1)\left[I + R^{-1}(k+1)\right]^{-1} Le_y(k), \tag{4.102}$$

$$G(k) = Q + L^T G(k+1)\left[I + R^{-1}(k+1)\right]^{-1} L. \tag{4.103}$$

The obtained equation is a Riccatti equation. It is solved from the last moment $k = q+K$ till the beginning $k = q$ with end condition determined from (4.92a)

$$Se_y(k) = p(K) = G(K)e_y(k), \tag{4.104}$$

$$G(K) = S. \tag{4.105}$$

The control $v(k)$ depends on the solution (4.103), (4.105) and is determined in the following way:

$$v(k) = -R^{-1}p(k+1), \tag{4.106}$$

From (4.92)

$$\begin{aligned} &p(k) = Qe_y(k) + L^T p(k+1), \\ &G(k)e_y(k) - Qe_y(k) = L^T p(k+1), \\ &p(k+1) = (L^T)^{-1}\left[G(k) - Q\right]e_y(k), \end{aligned} \tag{4.107}$$

Then

$$\begin{aligned} &v(k) = -R^{-1}(L^T)^{-1}\left[G(k) - Q\right]e_y(k) = He_y(k), \\ &H = -R^{-1}(L^T)^{-1}\left[G(k) - Q\right] \end{aligned} \tag{4.108}$$

Control depends on the real value of the error $e_y(K)$.

4.3.6.6 Common Nonlinear Control for the Nonlinear Process

The common nonlinear control according to (4.78) and (4.85, 4.86) is

$$e_{F_R}(k) = \overline{M}\left[L - 1 + \Delta tA + \Delta tBF_R^s\right] \cdot e_y(k) - \overline{M}R^{-1}(L^T)^{-1}\left[G(k) - Q\right] \cdot e_y(k) \qquad (4.109)$$

where

$$e_{F_R}(k) = \overline{M}.V.e_y(k) - \overline{M}He_y(k) = \overline{M}[U - H]e_y(k) = \\ = \overline{M}1e_y(k). \qquad (4.110)$$

$$V = \left[L - 1\Delta tA + \Delta tBF_R^s\right]. \qquad (4.111)$$

This control can be realized in real time as it is a function only of the current value of the state error. When the optimization interval is sufficiently long $k \to \infty$, $k \neq \infty$ then the solution of the Ricatti equations tend to be constant

$G(k) \to G(0) = G = const$

and this constant value can be used in (4.108)

The control consists of two parts:

- nonlinear one, which linearizes the nonlinear model, and
- linear, which minimizes the error and control effort and reduces the influences of parameter variations.

The calculation of the common nonlinear control is according to the following Algorithm 4.4.

Algorithm 4.4

1) The values of the model matrix are set; A, B, B_1, W.
2) The values of the steady state variables y^s and F_R^s are obtained from the steady state optimization.
3) The matrix L is determined.
4) Riccatti equation (4.103), (4.105) is solved, and $G(k) = G(0)$ is determined.
5) The matrix H is determined (calculated) from (4.108).
6) The matrix $\overline{M}$ is determined (calculated) from (4.86).
7) The Matrix U is calculated from (4.111).
8) The matrix $\overline{M}1 = \overline{M}[U - H]$ is calculated and can be used for real time implementation.

4.4 CONTROL SEQUENCES

4.4.1 Sequence Control

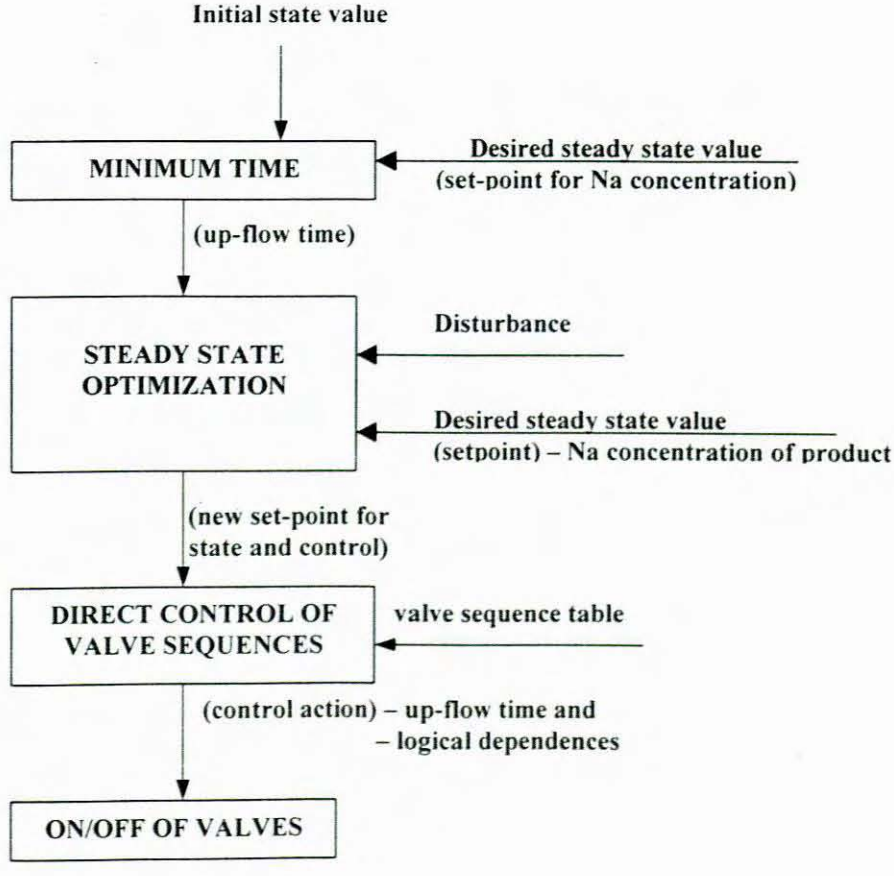


Figure 4.10. The Control Computing and Implementation Structure.

4.4.2 Cation Regeneration Column Sequence

During this sequence the cation load sequence is also running the only time to check is during resin transport from load catchpot to top regeneration catchhpot.

Algorithm 4.5:

1) Up-flow Period:

- Open V2 (make sure that V4 and V15 are closed).

2) Settle Period:

- Check the current status of L3,
- If the signal is ON:
  - ✓ Make sure V4 and V9 are closed (to make sure pull-down does not occur),
  - ✓ Assume resin is in the top catchpot,
  - ✓ Open V17 (fill up the liquid on the top catchpot),
  - ✓ Until L4 comes ON,
  - ✓ Close V17 and open V15,
  - ✓ Wait for 5 minutes,
  - ✓ Flush V15 (by leaving V17 on) for 10 seconds,
  - ✓ Start with pull-down period,
- If L3 is OFF (no signal):
  - ✓ Open V17,
  - ✓ Receive L4 signal,
  - ✓ Close V17,
  - ✓ Open V15,
  - ✓ Wait for 1 minute,
  - ✓ Flush V15 (by opening V17 for 10 seconds),

- ✓ Close V17 and V15,
- ✓ Transport from load (V7 and V5 open),
- ✓ Wait 1 minute.

3) Pull-down Period:

- Open V4 and V9,
- Wait for L3 signal (or 1 minute),
- Close V4 and V9,
- If L3 comes ON – during pull-down:
  - ✓ Transport resin from load (Open V5 and V7),
  - ✓ Wait 5 minutes,
  - ✓ Stop transport (Close V5 and V7),

4) Resin Transport to Load Top Catchpot:

- Check V16 status,
- If V16 is OPEN (fluidization of load top catchpot),
  - ✓ Wait for the timer (V14 opens and closes),
  - ✓ Open V8, V11, V10, V12 and V13,
  - ✓ Wait 1 minute.

During regeneration transportation, the next regeneration up-flow must be started simultaneously.

The cation load primary and secondary sequences are as follows, respectively:

LOAD PERIOD	VALVE																
	V1	V2	V3	V4	V5	V6	V7	V8	V9	V10	V11	V12	V13	V14	V15	V16	V17
(P1) UPFLOW	O	X	C	X	X	C	C	X	X	X	X	O	X	C	X	X	X
(P2) SETTLE	C	C	C	C	C	C	C	C	C	C	C	O	C	C	C	C	X
(P3) PULLDOWN	C	C	O	C	C	O	C	C	C	C	C	O	C	C	C	C	X
(P4) FLUIDIZATION	X	X	X	X	X	X	X	X	X	X	X	O	C	C	X	O	X
(P5) TRANSPORT	X	X	C	X	O	C	O	X	X	X	X	O	X	X	C	X	X

Table 4.1. Valves Switching Sequence for the Cation Loading Column.

4.4.3 Cation Load Column Sequence

Algorithm 4.5:

1) Up-flow Period:

- Open V1 (close all other valves on the cation column – what about V7 and V5).

2) Settle Period:

- Close V1,
- Wait for 3 minutes,

3) Pull-down Period:

- Open V6 and V3,
- Wait for 5 minutes or signal from L1,
- Close V3 and V6,
- Check V13,
- If V13 is OPEN (transport from regeneration column is ON):
  - ✓ Wait until V13 is CLOSED,
- If V13 is CLOSED:
  - ✓ Open V16,
  - ✓ Wait for L2 signal,
  - ✓ Close V16,
  - ✓ Open V14,
  - ✓ Switch ON the timer,
  - ✓ Wait for 3 minutes,
  - ✓ Open V16,
  - ✓ Flush V14 (open V16 for 10 seconds),
  - ✓ Close V16,

- ✓ Close V14,
- ✓ Stop Time,

- Next Up-flow period.

The cation regeneration primary and secondary sequences are as follows, respectively:

REGENERATION PERIOD	VALVE																
	V1	V2	V3	V4	V5	V6	V7	V8	V9	V10	V11	V12	V13	V14	V15	V16	V17
(P1) UPFLOW	X	O	X	C	X	X	X	C	C	C	C	O	C	X	C	X	X
(P2) SETTLE	X	C	X	C	X	X	X	C	C	C	C	O	C	X	C	X	X
(P3) PULLDOWN	X	C	X	O	X	X	X	C	O	C	C	O	C				X
(P4) FLUIDIZATION	X	C	X	C	X	X	X	X	C	X	X	O	X	X	C	X	O
(P5) TRANSPORT	X	X	X	C	X	X	X	O	C	O	O	O	O	C	C	X	C

Table 4.2. Valves Switching Sequence for the Cation Regeneration column.

The secondary sequence is operated concurrently with the primary sequence it deals with the regeneration cycle times. The control of the primary cycle and the secondary cycle is based on digital values obtained from the level sensors and digital values that perform the actual control actions (switching of valves). These values thus create an overall cyclic operation of the plant because they link the loading column with the regeneration column.

4.5 REAL TIME CONTROL IMPLEMENTATION

1. Determination of the moments of $q = 0,1,2....$
2. Measurement of the input concentration of the input flow.
3. Set the values of the setpoints y^{sp} and $F_R^{sp.}$
4. Calculation of y^s and F_R^s from optimization program to calculate T^s.
5. Using T^s as the control action.

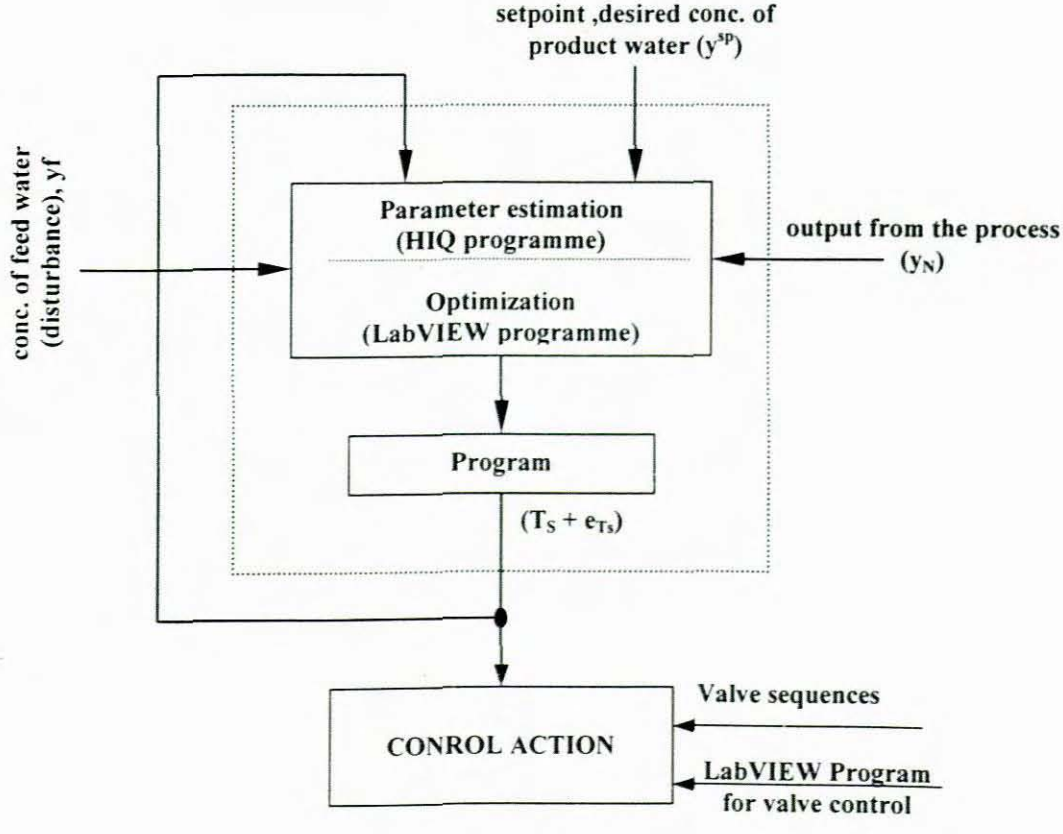


Figure 4.11. Real Time Control Implementation.

4.6 CONCLUSION

Minimum startup time for the ion exchange is formulated and the solution is proposed based on the Lagrange functional. The problem is then decomposed into a two-level structure to solve the two-point boundary problem.

The problem of optimal control of processes in the presence of slowly varying disturbances solved based on the repetitive control. The problem is formulated for steady state control. This results in decomposition of the problem into two subproblems steady state and dynamic one. The gradient method is applied in solving the steady state model and the algorithm for this method is given. Up-flow time is determined from the control variable solved using the gradient method.

For the dynamic model the designed control structure has characteristics of the optimal feed-forward and feedback control. The feed-forward control attempts to eliminate the effects of measurable disturbance – the input flow concentration. The feedback control corrects for unmeasurable disturbances and modelling errors. The following positive effects are recorded: (Deshpande, 1995; Edgar & Seborg, 1980; Genesio & Tesi, 1988; Roberts, 1999)

- The feed-forward control takes corrective action when the disturbance is measured, or before the process is upset. Because the controller can act before the disturbance affects the process, perfect control action is theoretically attainable.
- The feed-forward control does not affect the system stability and enhances the effectiveness of the feedback control.
- The feed-forward control is optimal according to the selected criterion and leads the process to the optimal steady states for given disturbance.
- The feedback control affects the system regardless of the type of source of disturbance.

- The feedback control can be realized with PI, PID controllers and little knowledge about the process because its tuning can be performed on-line. In this case the exact process model is not required.
- The designed feedback control performs two actions
  1) linearizing the closed loop system according to desired dynamics of linear stable system, and
  2) Optimally stabilizing the output of the linearized system according to some steady state value.

In this way the total behaviour of closed loop system is linearized and robust according to unmeasured disturbances and model parameter variations.

The control structure requires measurement of the input flow concentration as main disturbance, which can be done easily with the existing pH and conductivity meters. Realization of the linearizing and optimizing dynamic control requires: (McGravey & Fidher, 1986)

- measurement of the output flow concentration which can be done with pH and conductivity meters from one side, and
- measurements of the concentrations of salt in every stage which at the moment can not be realized because the stages are not accessible. These measurements will be developed at later phases of the building of the ion exchange plant.

For real time realization of these methods of solutions, LabVIEW is used for solving the steady state problem. A Matlab program is developed for solving the dynamic problem. Both these programs are included in the APPENDIX B.

Another program for optimal control realization is developed for sequential control of the plant. This program is based on the control sequences (Table 4.1, 4.2). This program is also attached to APPENDIX B.

Chapter Five

Data Acquisition and Control Instrumentation

The chapter deals with instrumentation of the project. It describes connections and interface of hardware components from the computer system to the plant. The hardware system is designed such that:

- it provides an operator with interactive data display including the values of the analog measurements of pH and conductivity of flows, status of levels and valves in the plant.
- realizes manual or fully automated control of the flows in the process.

The construction of instrumentation with the main goal in mind is to develop a system that does not require any sophisticated processing technologies but be able to achieve a high level of water purification.

5.1 INTRODUCTION

The strategy developed to control the ion exchange process requires:

1. Real data for the value of the concentration of the sodium chloride (NaCl) in input waste water and output product water received on the basis of measurement of the concentration of the pH and conductivity.
2. Real data for the levels of the mixture of resin and water in hoopers and catchpots (APPENDIX A).
3. Real data for the levels of solutions in tanks for concentrated NaCl solution and wastewater.
4. Control actions realized by switching the flows in the process.
5. Realization of some logical conditions for the sequence operation of the process.
6. Automatic receiving of data from the process and automatic realization of the control action.
7. These requirements are realized using different types of sensors and instrumentation:
    - pH, conductivity, level sensors
    - the personal computer (PC)
    - input/output modules: analog card (PC71) and digital modules IDC5, OAC5A mounted on a digital rack PB24
    - input/output cards for PC: data acquisition card (PC30GA), adapter card (ADPT5050) and digital increased current interface card (PC38X)
    - solenoid valves
    - diaphragm valves and
    - pumps

5.2 THE IMPORTANCE OF DATA ACQUISITION

Data acquisition is very important for implementation of any control system. Prior to the implementation of any control system for the process, reliable data should be acquired by the computer system and data should be available to the computer in a convenient form. Data (information) from different points in the process can be collected using sensors. The fundamental of the data acquisition system (DAQ) is the measurement of physical phenomena in real world and converting these physical signals in a form that is suitable for a computer system. Before a computer can measure a physical signal, a sensor, sometimes called a transducer must convert the physically measured signal (temperature, light intensity, pH, wind speed etc.) into an electrical signal such as voltage or current. Most of the DAQ systems would require that a signal conditioning be used to convert the voltage or current produced by the sensors to a convenient signal that the computer can use. It is also important that these signals should be within the limits that of the computer otherwise major problems might occur (Hunter, 1987; Johnson, 1993; Liptak, 1999).

Another important aspect for data acquisition is acquiring of reliable data, which in this case is achieved by using high quality sensors and conditioning of data using reliable signal conditioning modules. Data is also collected at different points in the plant using real time, meaning there is no interruptions to the DAQ system during the returning of acquired data.

The developed data acquisition system consists of the following parts: the personal computer (PC), signal conditioning (amplification, isolation, multiplexing, sampling, filtering, excitation and linearization), analog inputs, digital inputs, digital outputs. Each part will be considered separately. A software program is needed by the computer to interface all the DAQ system components. In some cases a DAQ input/output may be used or the signals may be directly connected to the computer using serial or parallel communication techniques. The computer system as hardware and together with this software program will take the final control in acquiring of data, analyzing, processing,

and display and or storage of the results. Every computer needs driver software to provide the communication link between hardware and software.

Data acquisition (DAQ) board specifications should be evaluated to check the accuracy of the board. All plug in boards, like DAQ board are sophisticated measuring devices that can give different accuracy's depending on the board being used (depending on different manufacturers). Care should be taken for the boards that are inadequately specified because the specifications omitted may be the one to cause inaccurate measurements (National Instruments Corp., 1999).

"What you don't know about your DAQ board can hurt your measurements." – National Instruments (NI)

5.3 THE HARDWARE SYSTEM BUILT FOR THE CATION COLUMNS

To obtain proper results from a DAQ system depends on the following elements for the system; the personal computer, transducer (sensor), signal conditioning, digital to analog hardware (including DAQ computer plug-in board) and software being used. The physical phenomenon being measured is the pH and conductivity of flows and resin (solution) level. These signals will then be processed with the goal of optimal control of the ion exchange plant.

Like in most DAQ system signal conditioning is required in this application since we are dealing with signals from sensors which are of very low current and cannot be used directly with the DAQ card. Signals from the computer (DAQ card) used for control actions in the process are low current signals. They have to be conditioned to high current signals to be able to drive the field devices, e.g. solenoid valves.

Proper results of the DAQ system are influenced by the different units used in the system involving, the computer system, transducer (sensor), signal conditioning and mostly the analog to digital conversion system of the DAQ card. These system components will now be discussed in detail. They are connected with the aim to:

1. Design input/output (I/O) circuits for connecting the computer system to control the plant equipment (valves, sensors, etc.)
2. Achieve a fully automated plant with manual option available for the operator.

The considered instrumentation system consists of different modules which are all from different manufacturers but are compatible. They are:

- The personal computer (PC) is from **National Management Services (NMS)** computer systems
- The DAQ card (PC30GA_ADV), external extension board, adapter (ADPT 5050), the signal conditioning modules, the current to voltage converter card (PC 71) and increased current interface card (PC 38X) and the interfacing cables (the DAQ DB50F/M and all ribbon cables) are all from **Eagle Technologies**.

- The signal conditioning modules, digital IO modules (IDC 5A and OAC 5A) and the digital IO modules mounting rack (PB 24H) are from **OPTO 22**.
- The pH sensor and meter is supplied by **Filtration and Dosing**.
- the **bürkert** CONDUCTIVITY 8225 meter is a **Bürkert** product.
- The digital input electro-LV1 liquid level controller is supplied by **Electro-Mechanica**.
- The solenoid valves, Bulletin 8320 from **ASCO**
- The liquid feed and resin transport pumps (**ASCO**).
- The diaphragm valves **ASCO** product
- The DC power supplies **International Power.**

All these units are somehow linked to provide a complete system that meets the requirements for optimal control of the plant. Most of the instrumentation hardware has been bought off shelf.

The global objective of the instrumentation section is to operate diaphragm valves which control the actual stream flows of the plant columns. Since the plant is mainly consisted of liquid flows, it would not be feasible to install electronic (solenoid) valves in the plant columns. This problem is solved by implementing a system where the electronic (solenoid) valves control the airflow, which in turn controls the diaphragm valves. The solenoid valves are installed outside the control room, due to noise, and the computer and other control modules are all inside the control room (APPENDIX A). Instrumentation block diagram is shown in Fig.5 1 below.

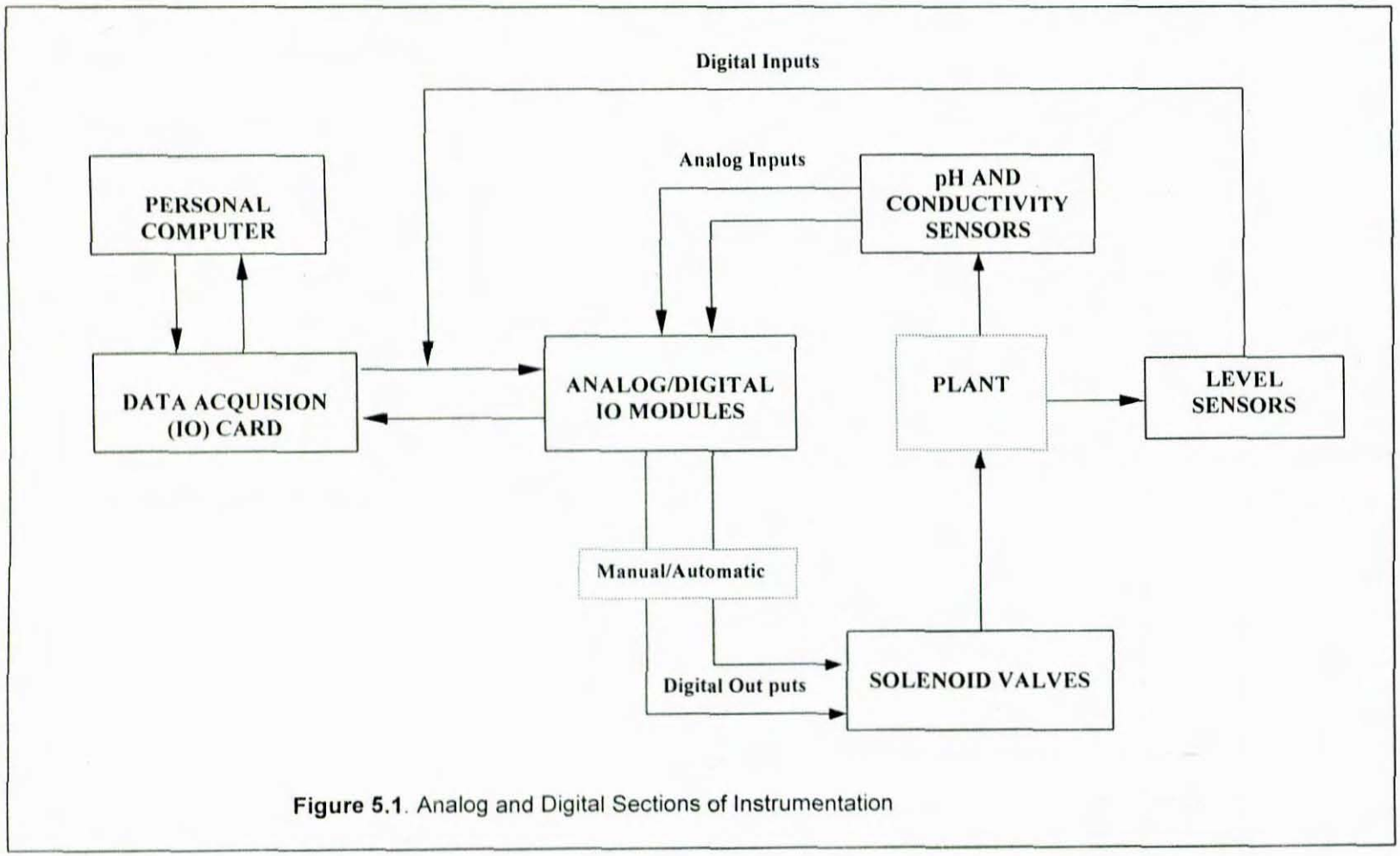


Figure 5.1. Analog and Digital Sections of Instrumentation

The instrumentation can be divided into two broad sections. There is a section that deals with analog data from pH and conductivity sensors and another section that deals with digital data that switches ON or OFF (digital signals) the solenoid valves in the plant and with digital input signals from level sensors. These sections are interlinked such that they perform a combined duty of controlling the plant.

5.3.1 The Computer System

The computer will drastically affect the speed at which data is being continuously acquired. Data transfer capabilities of the PC can also significantly affect the performance of the DAQ system. All PC's are capable to use programmed input/output and interrupt data transfers. The limiting factor in acquiring large amount of data is often the disk space (hard drive) available to store this data. This is due to the disk access time needed to get the required data. Determination of the operating system of the PC and its platform will yield the greatest long term on investments and still meet the short-term goals.

A standard personal computer (PC) with a Pentium II microprocessor is the front end for operation, with the status indicators and controls for the operator of the ion exchange plant. It features a 17-inch monitor for visual display of icons for status indicators and controls. A standard keyboard (101) and a mouse for easy control of actions are used. The computer runs on Windows 95 as its platform and for operation stability and LabVIEW is used as control software.

For data transfer capabilities, ion exchange process is classified as process with slow varying disturbances and a Pentium II is fast enough to handle the process, making it suitable for the control of the plant. The processor speed of 330MHz is also large enough for streaming of data. The computer has 64MB (mega bytes) of static memory which makes processing faster and improves disk access time. A lot of processing is required to

decide on the next control action of the plant and hard disk of 10GB (giga bytes) is installed. For keeping of records, a CD_ROM writer will be installed in future once the plant is fully functional where data about the past behaviour of the plant will be kept as long as required.

Printing facility is currently not installed but is also envisaged for the future. It will be used for printing of relevant messages for example, alarms or any other suspicious occurrence etc.

5.3.2 The Analog Section

This section is composed of the following:

- DAQ board (PC30GA_ADV)
- ADPT5050 adapter connector
- Current to Voltage Converter (PC71 card)
- pH sensor (pH S-mA)
- Conductivity sensor (**bürkert** CONDUCTIVITY 8225)

The DAQ card (PC30G_ADV) is used for acquisition of both analog and digital signals (input and outputs) and then for the processing of the received data. The signals from the pH and conductivity sensors are a low current signal which cannot be connected directly to a computer therefore it must first be converted to voltage signals. This is achieved using the current to voltage converter card, the PC71.The pH sensor is used to measure pH in the input feed and output stream of the cation load column. The conductivity sensor is used to measure conductivity of the feed stream and the output stream of the cation load column. Data acquired from both sensors is then used for calculation of the concentration of salt in the input and output column flows (Siemens Pazo, 1999).

It is important to understand the nature of the signal being measured, the configuration used to measure the signal and the effects of the surrounding environment. Based on this

information it can be determined which of the signal conditioning elements are needed, Fig.5 2.

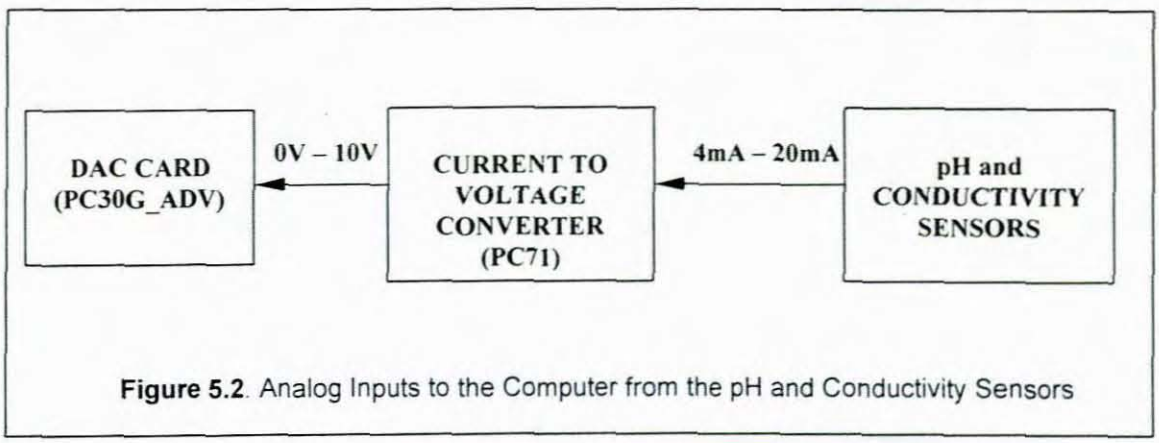


Figure 5.2. Analog Inputs to the Computer from the pH and Conductivity Sensors

Electrical signals (current/voltage) must be optimized for the input range of the DAQ board. If acquired analog signals are very low-level signals the signal conditioning must first amplify or isolate these signals. Signal conditioning may include the following, attenuation or amplification, isolation (filtering), multiplexing, sampling, linearization and excitation. For this case amplification, linearization and excitation are all embedded in the transducers used, pH and conductivity. Isolation and filtering are achieved by the current to voltage converter, multiplexing and sampling are performed by the DAQ board the PC30GA-ADV.

Sensors are probes used to sense the physical phenomena and provide electrical signals equivalent to the measured phenomena (e.g. temperature, pH etc.). The signal produced by these probes is either in voltage or in current form. The pH and conductivity sensors have internal amplification. It is used to amplifying signals to high-level signals which minimizes the electrical noise effects on the reading. Amplification is used to achieve the highest possible accuracy but the amplified signal should be such that the maximum voltage range does not exceed the input voltage range of the analog-to-digital converter of the DAQ board.

Linearization is another common signal conditioning function used to convert the non-linear response as produced by some sensors due to the nature of the physical phenomena being measured. The measure signal must then be processed such that changes in the signal are made linear to provide linear changes for the analog-to-digital signal (Jones, 1991, Johnson, 1993; Liptak, 1999; National Instruments, 1998b).

5.3.2.1 DAQ card (PC30GA_ADV)

This is an input/output (IO) data acquisition card used to acquire signals from field devices to the computer and to send signals to the field devices from the computer. It provides the communication link between the computer and the field devices thus providing automated control of the plant. It is the core between instrumentation hardware and software interfacing. For this section the components of interests are:

- sixteen analog input channels.
- three analog output channels
- analog ground

The PC30GA_ADV is a family of PC30F/G series DAQ boards that are half-size (of historical PCI boards), low cost and high accuracy analog and digital input/output (IO) for use with IBM computers. These boards use FPGA technology which allows many of the PC30 boards to be software controlled thus reducing the number of jumpers on the boards. PC30GA_ADV has the following features:

- sampling rate of 100kHz A/D
- 24 programmable digital IO lines
- 16 single ended (SE) or 8 differential analog inputs using 16 A/D
- programmable gains of 1, 10, 100 and 1000.
- 4 DACs, digital to analog converter channels

The PC30GA_ADV features 16 analog to digital (A/D) for each analog channel with several separate components, the input multiplexer, programmable gain amplifier, sample and hold unit, the A/D converter and FIFO buffer. Sampling is the method in which the analog-to-digital converter reads the analog signal to convert it to a digital signal. Sampling rate determines how often does the analog-to-digital conversion can take place when reading a channel (acquiring data). A faster sampling rate acquires more data points in a given time and can therefore often form a better representation of the original signal (Eagle Technology, 1996a).

The PC30GA_ADV A/D subsystem allows for 16 single ended or 8 differential analog inputs and can be configured for unipolar input range of 0 – 10V or bipolar range of –5V to + 5V and –10V to +10V. For unpolar inputs, the output code is straight binary and for bipolar it is offset binary to take care of the sign. The multiplexer selects one of the 16 single ended or 8 differential input channels. Multiplexing is a technique used for measuring several signals with a single measuring device. Multiplexing is achieved by the analog-to-digital converter (ADC) which samples (reads) one channel (input/output), switches to the next and so on, until all the channels are read. Scan rate can be used to specify the rate at which scanning must take place in each channel. It is defined as the number of samples to acquire per channel. Because the same ADC is used to sample the number of samples instead of one, the effective sampling rate of each individual channel is inversely proportional to the number of channels being sampled. The channel is selected by a channel address obtainable from the channel list. The channel list contains a list of channel to be converted and may be up to 31 channels in length. When the end of the list is reached, the A/D loops back to the first channel in the list. The channel list may be disabled to enable compatibility with older products (Eagle Technology, 1996a).

The instrumentation/programming gain amplifier amplifies the signal from the multiplexer by one of the four programmable gains. The gain is automatically selected by the gain code stored in the board's gain memory. The sample and hold unit holds the

selected input channel steady for the duration of the A/D conversion process. For accurate acquiring of signals the instrumentation amplifier must settle within a certain period of time. Settling time is defined as the time required for a signal being amplified to reach certain accuracy and stay within specified range of accuracy. The DAQ board cannot return an error message to the computer when the instrumentation amplified does not settle because the errors occur in the analog stages of the DAQ board.

There are two methods to ensure that data is accurate:

1. Selecting a DAQ board with an instrumentation amplifier that is guaranteed to settle at all sampling rates and gains.
2. Reducing the sampling rate to the one that the DAQ board can accurately attain.

The instrumentation amplifier is mostly likely not to settle when several channels are sampled at high gains and high rates (National Instruments Corp. 1998a).

Resolution refers to the number of bits that the ADC uses to represent the analog signal. The higher the resolution the higher the number of division that range is 'broken' into and therefore the smaller the detectable voltage change. This refers to the minimum and maximum voltage levels that the ADC can divide into smaller divisions. Increasing the number of divisions will be more accurate because the voltage is 'broken' into finer divisions. The smaller divisions represent the voltage more accurately. An n-bit digital converter will divide the analog signal range into 2^n divisions with each division representing a binary code. Most available DAQ boards offer selectable ranges so that the board is configurable to handle a variety of different levels. The range is therefore somehow related to the resolution of the DAQ board. The range together with the resolution and the gain available on a DAQ board determine the smallest detectable change in the voltage. This is often called the code width, it is found by dividing the voltage range by the gain times two raised to the order of bits in the resolution.

$$\textit{Voltage change} = \textit{code width} = \textit{voltage range}/(\textit{gain} \times 2^n) \qquad (6.1)$$

Relative accuracy is a measure in least significant bits (1 LSB) of the worse case deviation from the ideal DAQ board transfer function (a straight line) (National Instruments Corp. 1998a).

- Analog to Digital Converter (A/D)

The A/D subsystem accepts analog voltage inputs from the sensors, etc. and converts them into 12-bit digital code. The PC30GA_ADV uses a monolithic A/D converter. This digital code is then transferred to the host processor which processes it according to the software in the use at that time. The sampling rate is controlled by a jumper and software. The ADC performs the actual A/D conversion. The conversion is begun by an A/S strobe by the timing and control selection. From A/D data is then transferred by polled IO or direct memory access (DMA) modes (later covered in detail in DAQ software). The FIFO buffer (first in first out) is a temporary store of converted results. The FIFO store data while the PC is performing other functions, such as memory refresh and while the CPU changes memory buffer. Changing memory buffers allows the entire memory space of the PC30 to perform DMA into the memory space of the computer without break.

The PC30GA_ADV contains a logic which allows any sequence of channels up to a sequence length of 31 to be selected and sampled under hardware control. This allows full throughput to be achieved even when converting multiple input channels. A/D conversions may be monitored by either polled IO, direct memory access (DMA) or by interrupts. In the polled IO mode the software continuously polls the board's status register to check for completion of the current A/D conversion. DMA is used to transfer data directly from the A/D to memory and in interrupt mode, the board automatically generates an interrupt on completion of each conversion. The A/D also has its key specifications: (Eagle Technology, 1996a)

- A/D resolution of 12-bits
- nonlinearity, less than ± ¾ LSB
- A/D full scale voltage ranges 0 – 10V, –5V to + 5V and –10V to + 10V.
- number of A/D inputs, (16 single ended or 8 differential)
- A/D throughput of 100kHz

- Analog Signals

Analog input specifications can give information on both the capabilities and the accuracy of the DAQ product. Basic specifications available on most DAQ's specify the number of channels, sampling rate, resolution and input range. The number of channel inputs are specified for a single-ended inputs and differential inputs. Single-ended inputs are measured with reference to one common ground point. The PC30GA_ADV can accept 16 analog inputs (CH0 – CH15). In the differential analog inputs, each signal has its own ground reference. Noise levels are reduced when this kind of connection is used because the common-mode noise picked up by the leads is cancelled out.

If signals are connected using single ended (SE) inputs, i.e. all signals are connected with reference to analog ground and all the 16 channels become available for use. The only down side of this type of connection is the vulnerability of signals to noise since *common mode rejection* is lost. Single ended inputs are very sensitive to noise and should not be used with lead lengths of greater than 18 inches (457.2mm) or for inputs with a gain greater than 10. In differential mode, only 8 channels are available, channels from CH9 to CH15 become the return lines for channels CH0 to CH8. Analog inputs are limited to a voltage of between –10V and +10V or 40mA. For this application since only four analog channels are required, therefore differential mode will be used. To maintain the specific accuracy all devices connected to the analog inputs of the analog channels of the

PC30GA_ADV card must have source impedance of less than 1000Ω. To maintain specific accuracy, all inputs to the PC30GA_ADV card must be within 110% of full scale of input voltage, i.e. overloading any analog input into the PC30GA_ADV by more than 10% may cause other input channels to become inaccurate or noisy. The PC30GA_ADV input operating at maximum gain corresponds to an input voltage of 5.5mV. This means the minimum voltage signal that can be measured will be 5.5 mV/1000, which corresponds to 5.5 μV (Eagle Technology, 1996a; National Instruments Corp. 1998a).
The analog input signals are from the pH and conductivity sensors. These signals are current signals of 4 – 20mA. Since the analog channels of the PC30GA_ADV can only accept voltage signals as inputs these signals need to be first converted to voltage signals. This is achieved using a PC71 card which converts current signals to voltage equivalent. More information on this card can be read from instrumentation chapter or isolation section of this chapter. The card provides 0 – 20mA or ± 20mA current to voltage of 0 – 10V or ± 5V conversion to be interfaced with the PC30GA_ADV analog channels. The PC71 adapter card also boast of the following signal conditioning features: (Eagle Technology, 1996a)

- precision low noise differential operational amplifiers (op-amp)
- isolation current conversion techniques
- use of filters to eliminate "ground currents" and ground noise

5.3.2.2 Interface Adapter (ADAPT 5050) Card

The adapter 5050 features three connectors, IDC-50 male type, the DB-50 male connector and 51-pin screw terminal blocks, for use with external devices. All three connectors are mapped one – one. That is pin 1 of IDC-50 connector is pin 1 of the DB-50 connector and pin 1 of the 51-pin screw terminal blocks, pin 2 of IDC-50 connector is

pin 2 of the DB-50 connector and pin 2 of the 51-pin screw terminal blocks and so on. This adapter does not provide any amplification or any sort of conditioning, its function is to provide easy connection between the inputs and the DAQ board thus reduces strain on the board and cables (EagleTechnology, 1996a).

5.3.2.3 Current to Voltage Converter (PC71 Card)

Isolation is another common application for the signal conditioning used to isolate the transducer from the computer for safety purposes. This is used when the signals being read have high-voltage transients that could damage the computer. If the signals being acquired are each referenced to different grounds the difference in ground potential can lead to what is known as ground loop. This can cause inaccurate representation of the signal being acquired. Using isolated signal conditioning modules will eliminate the ground loop problem while ensuring that the signal being acquired is accurate. This is achieved by the use of the current to voltage converter which has internal isolation.
The PC 71 card is an external board used with DAQ cards to provide a precision 0mA to 20mA or ± 20mA current to voltage conversion. The output voltage from this card can be set to be 0V to 5V or ± 5V so that it can be into the analog or digital channels of the DAQ card. The PC 71 has the following advantages over the conventional 'shunt resistor' networks:

- it uses precision low noise differential op-amplifiers.
- it uses isolation current conversion techniques.
- it used filters to eliminate ground currents and ground noise.

The PC 71 card can be used as a current to voltage converter or a direct voltage bypass. On the latter mode the card allows voltage that has been acquired at its input to be passed through to its output without any modification to it. This is easily achieved using jumper settings (J2 to J17). The PC 71 has the following features, analog current/voltage inputs

(TL1 and TL2), analog output connectors (TL4), and digital IO ports from the DAQ card (PC30G_ADV), TL5, TL6 and TL7 and power supply connector block (TL3). The power supply terminals are also protected against short circuits by means of pico fuses. If the current drawn from the ± 12V or + 5V power supply exceeds 125mA then the fuse will blow. For current signal connection, the positive line must be connected to I_X + and to I_X -for a negative line. For voltage signal connection, the positive line must be connected to V_X and the negative line to analog ground (AGND).

The PC 71 card has two interfacing connectors. The IDE50 male connector (J1) is provided for direct interfacing with the DAQ card using a ribbon cable. Since the PC30G_ADV has a DB50 connector, a conversion from DB50 to an IDE50 is needed. The PC 71 assumes a One – One mapping (DB50 Pin 1 = IDE50 Pin 1, DB50 Pin 2 = IDE50 Pin 2 etc.) when converting the DB50 connections to IDE50 connections.

Before attempting to interface 4 – 20mA current output transducers (pH and conductivity sensors) to the PC 71 card, it is important that the card is tested first. When isolating the digital and analog grounds, the user must ensure that the DAQ card (PC30GA_ADV) is configured for the digital ground not ± 5V since they share the same channel (Eagle Technology, 1996b).

5.3.2.4 pH Sensor (pH S-mA)

pH and conductivity probes are used for measuring physical phenomenon of acidity/alkalinity of certain streams in the plant and present these measurements as electrical signals. These signals are industrial standard current signals of between 4 – 20mA. The pH measurement is based on determination of the cell voltage of a galvanic cell consisting of two electrodes, measuring and reference electrodes dipped into a solution. These electrodes are electrolytically connected by the solution. A potential which is dependent on the concentration of hydrogen ions is generated on the pH-sensitive glass diaphragm of the measuring electrode.

Solutions that conduct electric current due to acids, alkalis and most salts soluble in water are described as electrolytes. Such electrolytic solutions contain ions (positively or negatively charged atoms). This ionization in solutions is called electrolytic dissociation. The degree of dissociation is a measure of strength of the acid or alkali of the solution which is known as pH measurement. A potential depending on the concentration of hydrogen ions in the measured medium is generated on the pH sensor (Siemens Pazo, 1999). The output signal from the pH S-mA sensor is an industrial standard of 4 – 20mA. In the measuring electrode a spherical glass membrane is used as the pH sensor based on the dependence of glass membrane on the hydrogen ion activity. This sphere is filled with a buffer solution of known pH. The potential difference between the inner and outer surfaces of the glass membrane is used for pH measurement when the sensor is immersed in a solution. The reference electrode has electrical contact to the measured medium via a diaphragm so that the circuit is closed via the measured solution. The output signal from this pH sensor is 4 – 20mA

5.3.2.5 Conductivity Sensor (bürkert CONDUCTIVITY 8225)

The conductivity transmitter (8225) combines a sensor and a transducer with display in one enclosure. The sensor component consists of replaceable probes. The transducer component converts the measured signal and displays the actual value. The output signal is provided via a 4-pole plug. The **bürkert** CONDUCTIVITY 8225 used is a transducer without relay. It functions in a two-wire circuit and it requires a power supply of 12 – 30VDC. The connecting line conducts the measuring signal and power supply on the same line and must not be installed in combination with high voltage or high frequency signals. The output signal of this transmitter is an industrial standard of 4 – 20mA signal proportional to the measured conductivity. The transducers with relays function in a 3-wire circuit.

Conductivity is defined as the ability of a solution to conduct electrical current. The load carriers are ions in the solution, dissolved salts or acids. In order to measure conductivity two electrodes are used at a fixed length apart and with specific surface. An AC voltage is connected to the electrodes and current produced by ion conduction is measured. This current is a direct function of the conductivity of the solution. The 8225 transmitter can be equipped with four different electrodes with cell constants of 0.01, 0.1, 1.0 and 10. The cell to be used is selected according to the measuring range, a 0.01 for ultra-pure to pure water from 0.1 – 10μS/cm. From 0.1 for pure to industrial water from 0.5 – 200μS/cm, 1.0 for pure, industrial and sewage water from 5 μS/cm – 10mS/cm and a 10 for industrial, sewage and concentrated solutions from 500 – 100mS/cm. The cell constant is an average value over the whole measuring range. The output signal from this sensor is a 4 – 20mA current signal. The **bürkert** CONDUCTIVITY 8225 offers three different modes for temperature compensation, linear compensation, compensation with memorized coefficient and teach-in function.

5.3.2.6 Power Supplies

A 24V external power supply is used for powering the pH sensor with its meter and the conductivity sensor with its meter. The power supply has the following specifications:

- 220V AC input voltages
- 10 – 13% of the operating frequency (47 – 63Hz)
- 10 – 15% tolerance of the input voltage (230 – 240V)
- Output ripple of 5 – 15V units, 5.0mV peak-peak voltage maximum
- Overvoltage protection circuit (crowbar) used to prevent damage to sensitive loads such as TTL logic circuits.
- Operating temperature of 25 – 65°C and
- Isolation breakdown voltage of 2500V AC and power dissipation of 3 –9W.

Another 12V power supply is provided for external supply of the PC 71 card since the card did not operate properly when the internal PC power supply was used. The specifications for this power supply are the same as that of the 24V power supply above.

5.3.3 Noise

Any unwanted signal that appears in the digitized signal of the DAQ board is noise. Proper shielding should not only be added around sensitive analog sections on a DAQ board, but must also be built into the layer of the DAQ boards with ground planes to help reduce the noise. Noise can create problems with the measurement accuracy of a PC-based DAQ system. Amplification of low-level analog signals directly on the DAQ board also amplifies noise picked up from the lead wires or from within the computer chassis. If the input signal is in microvolts range, noise can drown the signal, leading to meaningless data (National Instrument, 1998a).

Even when a measurement setup avoids ground loops or analog input stage the signal will almost inevitably include some amount of noise (unwanted signal) picked up from the environment. This is mostly true for low-level signals that are amplified using on board amplifier that is available in most data acquisition board. In order to minimize noise coupling from this and other sources, a proper cabling and shielding may be necessary.

There are four principal noise coupling (pick-up) mechanisms, conductive, capacitive, inductive and radioactive. Noise can be from one of the following, ac power cables, computer monitor, switching logic signals and high voltage or high current ac or switching circuits. The noise will be coupled to the signal by either, common impedance (conductive) electric field (capacitive), magnetic field (inductive) and electromagnetic (radioactive) according to National Instrument (1998a).

- **Testing of Analog Section Instrumentation**

Analog section was tested in the laboratory by acquiring analog signals using the pH and the conductivity sensors. Signals were acquired successfully. Solutions of different concentrations were used to determine the successfulness of the tests.

5.3.4 The Digital Section

This section is composed of the following:

- DAC (PC30G ADV) card
- Interface Adapter (ADAPTER 5050 card)
- Current Buffer (PB 38X) card
- Digital (Input/Output) Module Mounting Rack (PB 24H)
- Digital Input Modules (IDC 5A)
- Digital Output Modules (OAC 5A)
- Level sensors (**electro-**LV1 LIQUID LEVEL CONTROLLER)
- Solenoid Valves (Bulletin 2830)
- Diaphragm Valves

The scheme below Fig.5.3 shows the digital modules interconnection.

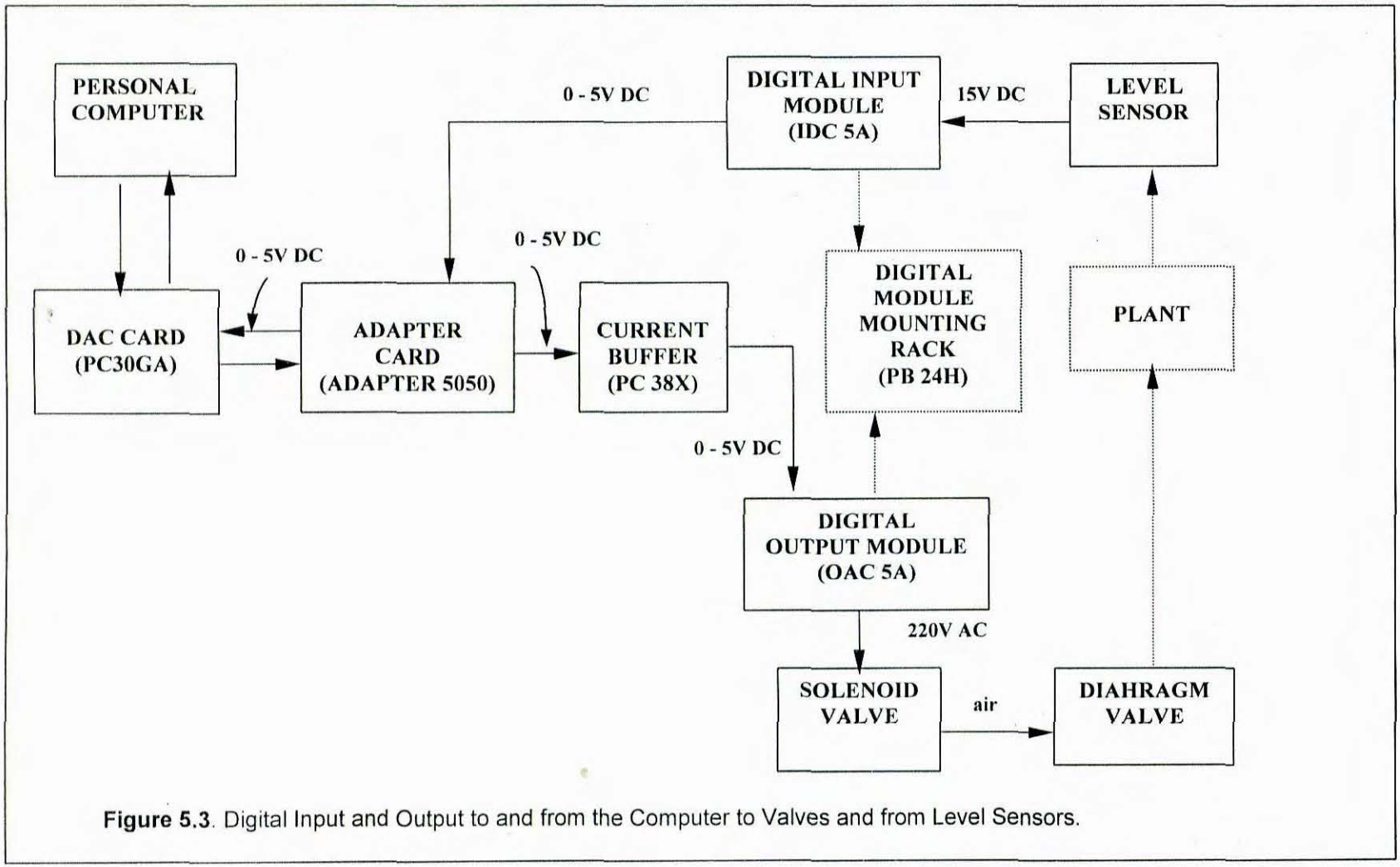


Figure 5.3. Digital Input and Output to and from the Computer to Valves and from Level Sensors.

Digital input/output (DIO) interfaces are often used to control processes, generate patterns for testing and communicate with peripheral equipment.

1) In each case the important parameters include the number of digital lines available,
2) the rate at which digital data on these lines can be accepted and sourced and
3) the drive capability of the lines.

If the digital lines are used for controlling events such as turning on and off heaters, motors or valves, a high data rate is usually not required because the equipment does not need to respond very quickly. In each of these examples, the amount of current required to turn the devices on and off must be less than the available drive current from the board. If the board has less current an external current driver can be added between the board output and the device being driven (relay are mostly used).

In the digital section of the instrumentation the DAQ board (PC 30G_ADV) is used for acquiring of digital signals to (digital inputs) and from (digital outputs) the computer. The DAQ board accepts digital signals from the level sensors and processes these signals to determine relevant control action. The digital output signals from the computer provide the required control action by switching the solenoid valves. The adapter 5050 is an interface card used for easy connection to any external connection to the card. The current buffer, PC 38X is used for current increase to drive the digital output modules since current from the DAQ card is low and not sufficient for these modules. Digital modules mounting rack (PB 24H) is used for housing the digital input modules, the IDC 5A and the digital output modules, the (OAC 5A). The digital input modules (input for DAQ card) the IDC 5A accept digital signals from the level sensors and present these signals to the DAQ card via the adapter 5050 for processing. The digital output modules (output from DAQ card), the OAC 5A accept signals from the card to drive the valves via the adapter 5050 and the PB 38X modules. Solenoid valves are electrical valves used to switch the pneumatic (diaphragm) valves. The diaphragm valves are used to do the actual switching in the plant. The difference will be that diaphragm valves are only pressure

driven and can withstand the liquid substances as opposed to the solenoid valves which are electronic valves and thus would not be installed in the plant. The level sensors are used to determine height (level) of solution in the catchpots and hoppers.

5.3.4.1 DAQ Card (PC 30G_ADV)

The card has the following digital features:

- 24 digital IO channels (8-bits ports).
- digital ground

These digital channels are bidirectional, meaning that they can be either inputs or outputs depending on the software. They are arranged to three 8-bit ports (PA, PB, PC). There should be no current drawn directly from the DAQ card

- Digital to Analog Converter (D/A)

The D/A subsystem consists of a quad D/A converter configured as two 12-bit D/A converters and two 8-bit D/A converters. Digital outputs from the host processor are converted to analog voltage output as required by the application in hand. Four DACs are independent of one another and can operate at a throughput of up to 130kHz. Their output voltage ranges can be independently configured as either 0 – 10V unipolar, -10v to +10V or –5V to +5V bipolar. This feature will not be used for our application so no further details are given.

- Digital Signals

The digital signals of the PC30GA_ADV are arranged in three 8-bit ports, port A, B and C, providing 24 digital input/output (DIO) lines. These ports are divided into two groups, group A consisting of port A and last 4 lines of port C (upper half of port C) and group B consisting of port B and the first 4 lines of port C (lower port C).
Each group can be configured into one of the three modes, mode 0, mode 1, and mode 2. Mode 0 provides simple IO operation, mode 1 is a strobed IO with hand shaking operation and mode 2 provides the strobed bi-directional IO (Eagle Technology, 1996a).

Mode 0

- Two 8-bit ports (port A and port B) with 4 bits (upper and lower halves of port C).
- Any port can operate either as an input or an output
- This mode provides simple IO operations
- Ports defined as inputs when read reflect the digital inputs on the port
- Ports defined as outputs are set to the value most recently written to the port
- Any port can be configured as input or output but not for both

Mode 1

- Two groups each consisting of one 8-bit and one 4-bit port.
- Each 4-bit port is used for control and status of each of the 8-bit port
- The 8-bit ports may be used for either input or output
- Input and output operations are latched
- This mode of operation provides IO operations with a simple handshake protocol.
- Handshake signals are assigned to port C

Mode 2

- One 8-bit bi-directional port and one 5-bit control port
- Can be used in group A only
- The 5-bit port is used for control and status of 8-bit port

- Input and output operations are latched
- Port B can still be used in mode 0 or 1
- This mode provides means for bi-directional IO operation on port A
- Port C is assigned handshaking signals

Digital input signals received by the DAQ card from the level sensors via digital input module, the IDC 5A for processing. Once the signals have been processed by the computer, control action is taken using digital output signals. A set of two level sensors for detection of high and low levels of the liquid at the following points in the plant are used. A set of two level sensors is connected at the cation load column hopper, cation regeneration column hopper and at the cation load column catchpot. This means that six digital IO lines are required for digital inputs. Before connecting these signals to the DAQ card it must be ascertained that they are of safe voltage levels to the card. This is achieved using digital input modules, IDC 5A,wich converts signals at its input to a numerical value (TTL logic) that can be interpreted by the computer. This module also provides isolation between field inputs and the logic output of the module.

5.3.4.2 Digital Output Signal

These signals are specifically kept for the control actions in the plant, turning ON/OFF of valves. This makes high data rates irrelevant since instruments to be driven have slow response. The number of digital lines matches the number of events being controlled. Currently only ten valves are to be controlled as indicated on the table below. For physical positions of these valves, refer to instrumentation chapter.

The amount current required to turn the devices (solenoid valves) ON/OFF must be less than the available drive current from the DAQ card. It this application it is evident that current from the PG30G_ADV card will not be sufficient to drive the solenoid valve which requires about 40mA. And thus in the signal conditioning stage, amplification is

required and this is achieved by the use of PC 38X, increased current adapter. This card increases the amount of current from the DAQ card to provide enough current for the driving the solenoid valves.

Digital ground line is a jumper selectable from PC30GA_ADV since it is shared with a + 5V supply from the card. This signal is a ground return line for digital inputs and outputs. Any digital circuitry tied to the digital lines should be referenced to this line.

5.3.4.3 Current Buffer (PC 38X Card)

The PC 38X is a 24 channel TTL IO driver, interface card. It is an industrial interface card used to provide increased current driving capabilities to all the 24 digital IO channels from the DAQ card. The design of the PB 38X is based on high-voltage current Darlington transistor array. The Darlington pairs feature high-voltage outputs with clamp diodes for switching inductive loads. The PB 38X card features a 50 pin-header connector (for connecting with PC 192, an extension board) and also 37 pin D-type female connector (D37) for connecting with the PC 36. The card uses a screened multicore cable with a D-type connector to interface with the DAQ card (PC30G_ADV)

5.3.4.4 Digital (Input/Output) Module Mounting Rack (PB 24H)

The PB 24H rack can accommodate up to twenty-four single channel input/output modules. It features a 50-pin edge connector for easy interfacing with other computer modules. To make sure that the signals have enough current to drive the modules before they are connected to the rack they are current boosted using the PB 38X card. The digital IO mounting rack (PB 24H) has an onboard LED indicators which shows the status of the digital level that drives the valve. This acts as the verification to what is

displayed by the computer. An external 5V power supply is provided to supply power to the mounting rack and in turn the modules.

5.3.4.5 Digital Input Modules (IDC 5A)

The IDC 5A digital input module converts digital signal at its input into a numerical value that can be interpreted by the computer. The module output is a TTL logic digital signal of 0 – 5V (ON or OFF). Each module has filtering on the input and the hysterisis amplifier for high noise reduction and "transient free" switching. The IDC 5A provides up to 4000V rms of optical isolation between field inputs and the logic output of the module.

Input signals to the IDC 5A modules are from the (high and low) level sensors. The digital output from the module is the digital input to the DAQ card. These modules are mounted on the digital mounting rack (PB 24H). Six of these signals are needed since three signals of HIGH and LOW will be received from the level sensors. These signals are high and low from the cation load catchpot and hopper respectively, high and low of the cation regeneration catchpot and hopper, and high and low from two tanks used for storage of wastewater and concentrated NaCl, refer to fig. 5 below.

5.3.4.6 Digital Output Modules (OAC 5A)

The solid state relay module circuit helps in maintaining the integrity of the valuable measurement and control signals and protects equipment from detrimental effects of noise and this is a requirement for the DAC card. This is achieved from the solid state relay module, the digital output module arranged to send ON/OFF signals to switch the solenoid valve. The module converts 0 – 5V signal to 220V AC in order to control the solenoid AC valves. This module connects to the mounting rack which supplies it with

the necessary 5V to drive its output. On receiving a LOW from the computer the module's output is 0V resulting from open contacts. On receiving a HIGH it closes its contacts, which switches the 220V AC from the external supply to drive a solenoid valve in this case.

The module provides isolation between input and output of 4000V rms and has a maximum load current of 2mA. In this application each module must drive solenoid valve which requires an input voltage of 220V at 41mA which can be handled by the OAC 5A module.

5.3.4.7 Level Sensors (electro LV1 LIQUID LEVEL CONTROLLER)

The level sensors here are used to give information about the levels in catchpots and hoppers. One set determines if there is solution in the hoppers (low level sensors) and another set, if the catchpots are full (high level sensor). Signals from the sensors are handled as digital signals to the computer. The low-level sensor is detecting that the level is lower than the sensor. It gives a signal when it is not immersed in the liquid. The high level works in the opposite way, when it is immersed in the liquid it then gives a signal but when it is not it gives no signal. The signals before being sent to the computer are sent to the digital input module (IDC 5A) which converts these signals to TTL logic, 0V – 5V signals.

The LV1 offers fail-safe control and monitoring of high and low levels of conductive liquids. The unit can be configured for "filling or emptying" applications by means of DIP-switches.

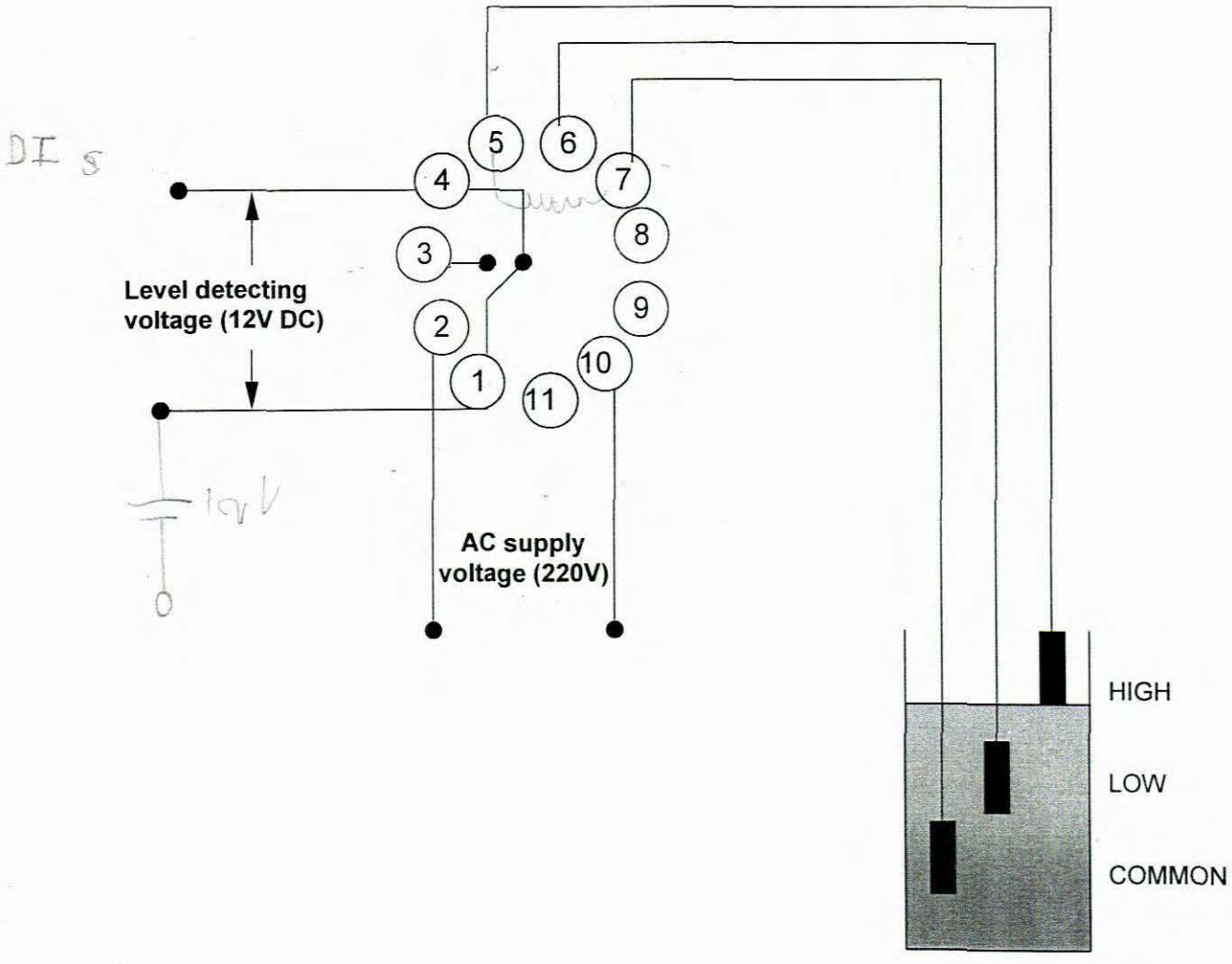


Figure 5.4. Wiring Diagram of LV1 Level Controller for Level Sensors.

- **Filling Mode**

When using the relay in filling mode the relay will energize if the level is below the LOW level probe. The relay will remain energized until the level reaches the HIGH level probe and the relay will de-energize.

- **Emptying Mode**

When the level reaches the HIGH level probe the relay will energize and will remain in this state until the level falls below the LOW level probe, when the relay will deenergize. In applications where only one level is monitored, contacts 5 and 7 should be bridged and used as the common probe and contact 6 should be used as a level sensing probe. This connection is used in the catchpots and hoopers only since they only deal with high or low levels. The figure below shows the scheme of how the signals from the level sensors are sent to the DAQ card.

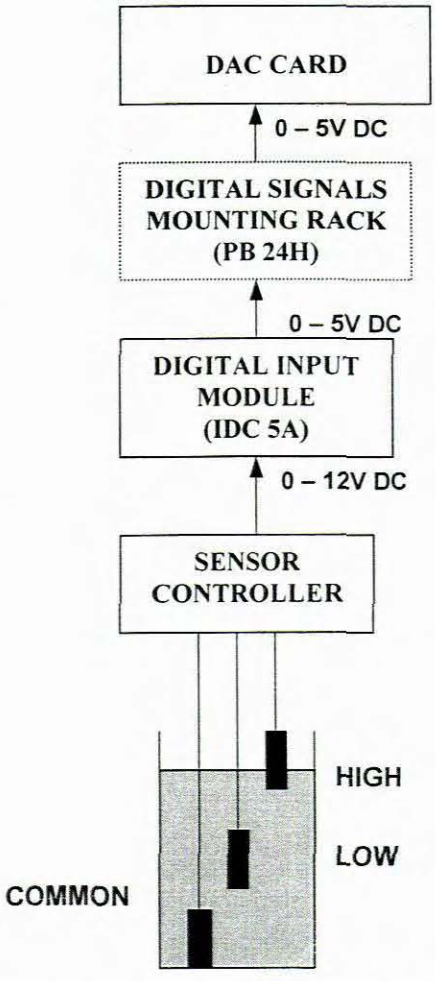


Figure 5.5. A Scheme Showing How Level Sensors Send Signals to the Digital Input of the DAQ Card.

5.3.4.8 Solenoid Valves (Bulletin 8320)

The Bulletin 8320 solenoid valves are three-way electrically controlled valves rated at 9W maximum at 220V AC voltage input. The Bulletin 8320 can be used with any of the following fluids; water, air, neutral gas and oil. The solenoid valves are switched ON/OFF by the computer signal 5V through the OAC 5A relay modules that switch on and off 220V from the supply. Solenoid valves are normally closed. If a solenoid valve is energized, ON air under pressure is allowed to flow between the inlert point (3) to the outlet point (1), which in turn closes the pneumatic (diaphragm) valve. If the solenoid valve is de-energized, OFF the pressurized air is blocked, the flow is between the inlet (3) and the vent (2) thus opening the diaphragm valve. The diagram below Fig.5 6 indicates this operation

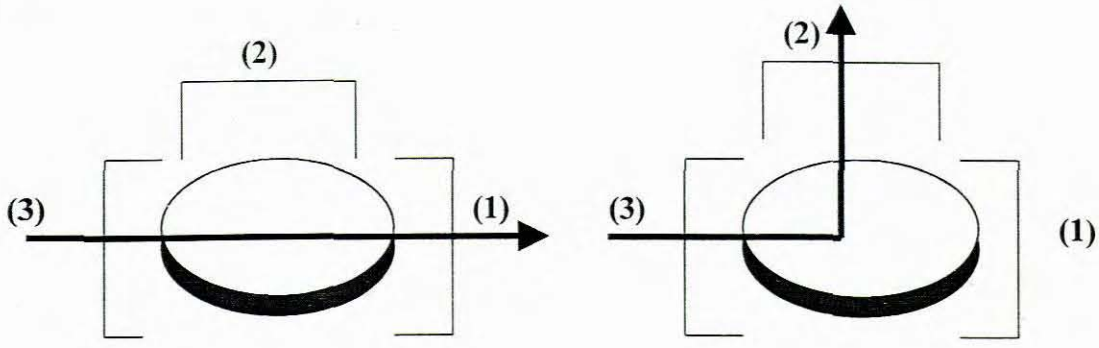


Figure 5.6. Diagrams Showing Energized and De-energized Solenoid Valves.

5.3.4.9 Diaphragm Valves

The diaphragm valves are normally open. On receiving air under pressure from the solenoid valves they close to block liquid flow on their flow pipe.
NOTE: Care should be taken during the startup of the plant. The solenoid valves should be all on to make sure that the diaphragm valves are closed.

5.3.4.10 Power Supplies

5V external DC power supply are provided for use with the digital IO mounting rack (PB 24H). This power supply has the following specifications:

- 220V AC input voltages
- 10 – 13% of the operating frequency (47 – 63Hz)
- 10 – 15% tolerance of the input voltage (230 – 240V)
- Output ripple of 5 – 15V units, 5.0mV peak-peak voltage maximum
- Overvoltage protection circuit (crowbar) used to prevent damage to sensitive loads such as TTL logic circuits.
- Operating temperature of 25 – 65°C and
- Isolation breakdown voltage of 2500V AC and power dissipation of 3 –9W.

A 12V external DC power supply is used to drive the signals from the level sensors and another also for use with the increased current adapter (PB 38X). For the level sensors 12V is switched ON or OFF depending on the status of the LV1 level detector to drive the input module. This 12V fed to the digital input module is then converted to a 5V signal for the PC. The specifications for this power supply are the same as for the 5V DC power supply above.

5.3.5 Manual Operation

The plant can be controlled manually by switching solenoid valves in a required sequence. This involves switching of relevant valves for different flows or streams depending on the requirements. Manual operation can be achieved by the control from the PC using LabVIEW functions or using physical opening and closing of valves controlled by manual switches. Manual/automatic control card in a portable casing is mounted inside the control box with sixteen switches each corresponding to a particular solenoid valve. The switches are numbered on the casing. Streams for measurement of pH and conductivity can also be manually controlled using relevant solenoid valve. Manual mode of operation can be used for shutdown purposes (testing, maintenance or troubleshooting) or emergency. The control switches are a three-pole single throw providing three possible control positions (Fig. 8). Position 0 is the manual OFF operation, at this position the corresponding solenoid valve switched off completely. Position 1 is for manually switching on of the solenoid valve. On position 2 the PC controls the corresponding solenoid valve and this provides the automatic control mode of operation. Depending on the PC control program a valve can be switched on or off. Though the possibility of self-run program and manually run program are designed, an operator has only control rights to self-run program. This program allows only the operator to either start or stop the program. All other control access possibilities are withheld from the operator using password control.

The manual control card also provides the solenoid status indicators. The control card status indicators are 220V LEDs. These indicators only determine if a solenoid valve is on or off but does not distinguish between manually ON or automatic ON. This means that as long as the solenoid valve is on the status indicator will come on. To detect whether the solenoid valve is automatic ON or manual ON one has to observe the switch position or use the status indicator on the mounting rack. If the mounting rack status indicator is on, then it means that the solenoid valve is operated automatically (Fig.5.7).

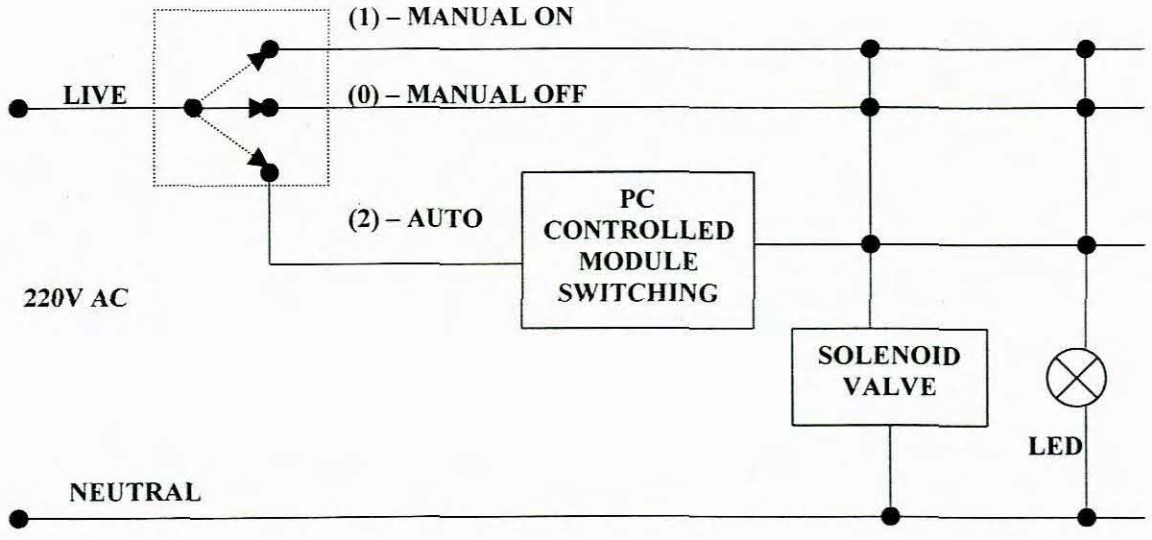


Figure 5.7. Manual / Automatic Operation Selection.

5.3.5.1 Computer Manual Operation

Though the computer control according to the design is an automatic control with the operation on this mode it is possible to control the plant manually. With the plant on automatic control solenoid valves can be switched ON or OFF using a program that is not self-running (sequence driven). The program allows automatic and manual control. On manual control sequencing can be done by switching solenoid valves using LabVIEW control functions on the control panel of the computer monitor.

5.3.5.2 Test of the Digital Section Instrumentation

The digital section instrumentation has been tested in the laboratory successfully by acquiring digital levels using level sensors. Different operating conditions were tested on emptying and filling modes of the level sensor controller and successful results were obtained.

5.4 CONCLUSION

In the data acquisition and instrumentation chapter the interfacing of the instrumentation hardware and association of this hardware with software used as a controlling tool is given. Firstly analog signals obtained from the pH and conductivity sensors are discussed as they play the measure role in determining salt concentration of the input and the output streams. The interfacing of these signals to the computer system which does control analysis and calculations for the control variable is discussed. Digital signals as used for control of solenoid valves (ON/OFF switching) and their interface with the computer to achieve the fully automated real time implementation of the control system is also discussed.

Different operation modes of the plant are also discussed, the manual control which deals with physical control of switches by the operator and also computer control which can be divided into two operating modes, self-running sequencing or manual control using control software (labVIEW).

The use of instrumentation from different manufacturers has proved very successful. Taking into account the fact that these devices are bought off-shelf and very cheap is a great financial benefit to the whole design. These devices have also worked very well with the control software, LabVIEW. The main benefit in terms of control software is the fact that the DAQ card (PC30GA_ADV) has its control functions that are designed based on LabVIEW. This made interfacing very easy unlike having to develop software functions for the DAQ card that would have to be linked to LabVIEW, which could be very tedious. All the control instrumentation is realized using instrumentation available in South Africa.

Chapter Six

Implementation of Data Acquisition and Control Algorithms and Programs

---

This chapter describes how the results from the previous chapters are implemented to develop a combined software and hardware automated system for acquiring of data and control the process by a personal computer. In this section also a discussion on how the software control of the plant is achieved, this includes analysis and calculations involved. Some control actions have to be taken based on these calculations and this is how the instrumentation, sensors and controlling software interface.

The chapter aims at arriving at two goals:

- acquire data, analyze it and store data that needs to be stored and
- to provide necessary control actions to the plant.

The question that this part of the dissertation tries to answer is "*how do we arrive at our main goal of minimum startup time and the optimal control of the process*?" with the developed instrumentation and the control software to be used. The three following statements gives the answer:

- Write a program for data logging (acquisition) of variables of pH and conductivity.
- Write a program that uses the acquired pH and conductivity readings to automatically optimize the plant performance and

- Then finally interface data acquisition and the optimization programs on the plant instrumentation units.

Interactions between data acquisition (DAQ) board and the pH and conductivity sensors allow calculations to be done based on the real values for the minimum time calculation. pH and conductivity values are acquired from the plant to the computer, from the received real values analysis are done, set-points are then set. If need be a new control action is sent through the DAQ system back to the plant.

6.1 ALGORITHMS FOR MEASUREMENT AND ACQUIRING OF ANALOG AND DIGITAL VALUES

The developed DAQ system provides capability of automatic or manual control. On automatic mode the plant can be controlled using a set of switches connected in the control box. In the automatic mode the plant is run by the computer based on the developed programs. The control programs are written using LabVIEW software. The switching modules is currently having sixteen switches for solenoid valves control of the cation system. Switches used are three-way switches that provide manual OFF, manual ON and automatic positions. On automatic mode the task of switching ON or OFF is provided by the computer system, thus the program will give the switch status.

To achieve the two goals of control and analysis two software packages are used LabVIEW and EDR software packages. LabVIEW is the controlling software working as the backbone of the system and EDR software provides certain functions that are developed specifically for the PC30 DAQ boards. The EDR is a controlling software developed for use with PC30-series DAQ boards but compatible with LabVIEW. LabVIEW will take care of all calculations and analysis after receiving data from the PC30GA_ADV that has been collected using the EDR functions. LabVIEW is a National Instruments product and EDR is an Eagle Technology product.

6.1.1 Measurements and Acquiring of Analog Values

Two streams are currently being measured and monitored for the cation section of the pilot plant to perform sodium (Na) concentration meausurement to be used with the optimizing of the process. pH and conductivity of both cation input and cation output streams are monitored. The input stream is the feed solution of the cation load and the product stream is the cation load output. The stream selection is based on the selection valves, V18 and V19 as show in Fig. 4.1. To monitor the input feed, valve (V19) is open and valve (V18) is closed. Readings are recorded for a period of five minutes for each stream. Before changing over a flush period of five seconds is allowed to lapse to rinse the previous stream contents on the pipes.

6.1.1.1 pH/Conductivity Technique for Measurement of the Salinity (Na Concentration in the solution)

Conductivity measurement is very often used for measurement of concentrations of different salts because of:

1. Conductivity is a linear function of concentration over the range of interest – at low concentrations
2. The conductivity of the acids is much higher than the conductivity of the salts of the same concentration.
3. The series of test have to be performed to establish the feasibility and the useful operating ranges.

The following method for determining of salts and acid mixtures is used:

1) Determination of the H^+ concentration from measurement of pH.
2) Determination of the part of solution conductivity K_H (Algorithm. 6.1) contributed by the H^+ ions using the experimental curves K_H versus C_H (Table 6.1) for hydrogen.
3) Determination of the solution total conductivity K_T from the measured conductivity.
4) Determination of the conductivity of the solution contributed by the sodium ions – K_{Na}.

$$K_{Na} = K_T - K_H \tag{6.1}$$

5) Determination of the solution concentration contributed by the sodium (Na) from the experimental curve K_{Na} versus C_{Na} (Table 6.2). The last concentration is used for control and model calculation.

This method is possible to be used as the concentration of the solution determined by two kinds of ions Hydrogen (H^+) and sodium (Na^+) ions, which take part in ion exchange (Fig. 6.1).

Concentration [g/l]	Measured pH	H+ Counductivity (KpH) [uS/cm]	CpH Conc. H+	CH* Conc. H+	INCREMENTS [ml]
0.3733804	2.06	4200	114.815	0.008710	50 HCl
0.187902	2.30	2100	199.526	0.005012	50 HCl + 50 water
0.125268	2.46	1400	288.403	0.003467	50 HCl + 2x50 water
0.093951	2.55	1000	354.813	0.002818	50 HCl + 3x50 water
0.0751535	2.62	850	416.869	0.002399	50 HCl + 4x50 water
0.062634	2.68	750	478.630	0.002089	50 HCl + 5x50 water
0.0536915	2.73	610	537.032	0.001862	50 HCl + 6x50 water
0.0469755	2.77	530	588.844	0.001698	50 HCl + 7x50 water
0.041756	2.80	480	630.957	0.001585	50 HCl + 8x50 water
0.0375804	2.84	420	691.831	0.001445	50 HCl + 9x50 water

Table 6.1. Experimental Data used for Calculation of pH Factor of the Final Concentration.

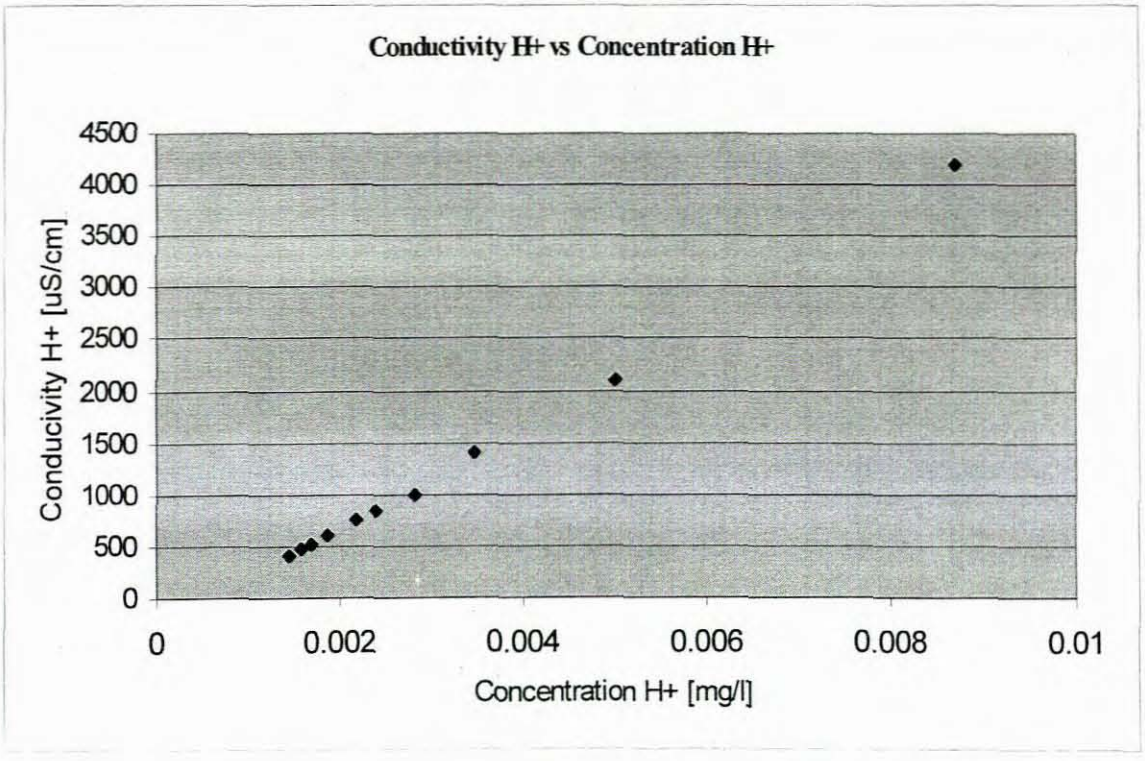


Figure 6.1. Experimental Data used for Calculation of pH Fractions of the Final Concentration.

Concentration [N]	CNa Concentration [g/l]	pH (NaCl)	KNa Conductivity [uS/cm]	INCREMENTS [ml]
0.01108	0.64819	6.66	1218	50 NaCl
0.00554	0.32410	6.04	680	50 NaCl + 1x50 water
0.00369	0.21587	6.46	380	50 NaCl + 2x50 water
0.00277	0.16205	5.87	340	50 NaCl + 3x50 water
0.002216	0.12964	5.78	260	50 NaCl + 4x50 water
0.001846	0.10799	5.68	215	50 NaCl + 5x50 water
0.001585	0.09272	5.61	180	50 NaCl + 6x50 water
0.001385	0.08102	5.50	160	50 NaCl + 7x50 water
	6.482		12180	

Table 6.2. Experimental Data used for Calculation of Conductivity Factor of the Final Concentration.

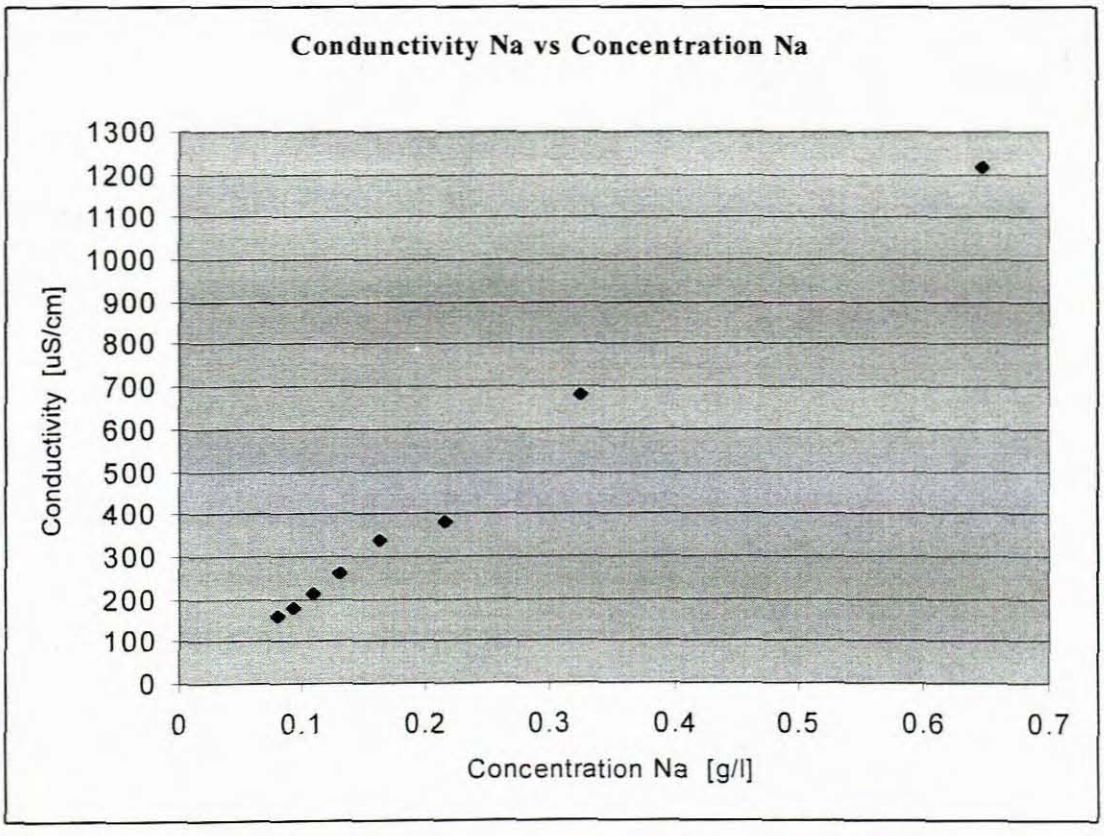


Figure 6.2. Sodium Conductivity versus Sodium Concentration Curve.

6.1.1.2 Procedure for Determining Gradients for Calculation of y-value

The procedure for calculation of gradients for calculation of the conductivity and concentration of Na^+ and H^+ in the solution is based on experiment and graphical considerations. It consists of the steps:

1. Read the constant from the pH probe.
2. Read the constant from the conductivity probe.
3. Calibration pH.
4. Calibration conductivity.

   Put probe into 0.01NaCl and adjust to 336.4 [μS/cm]
5. Draw graph for conductivity,
   - Pore 100ml NaCl – 0.01N – measure conductivity.
   - Calculate the slope of the graph.

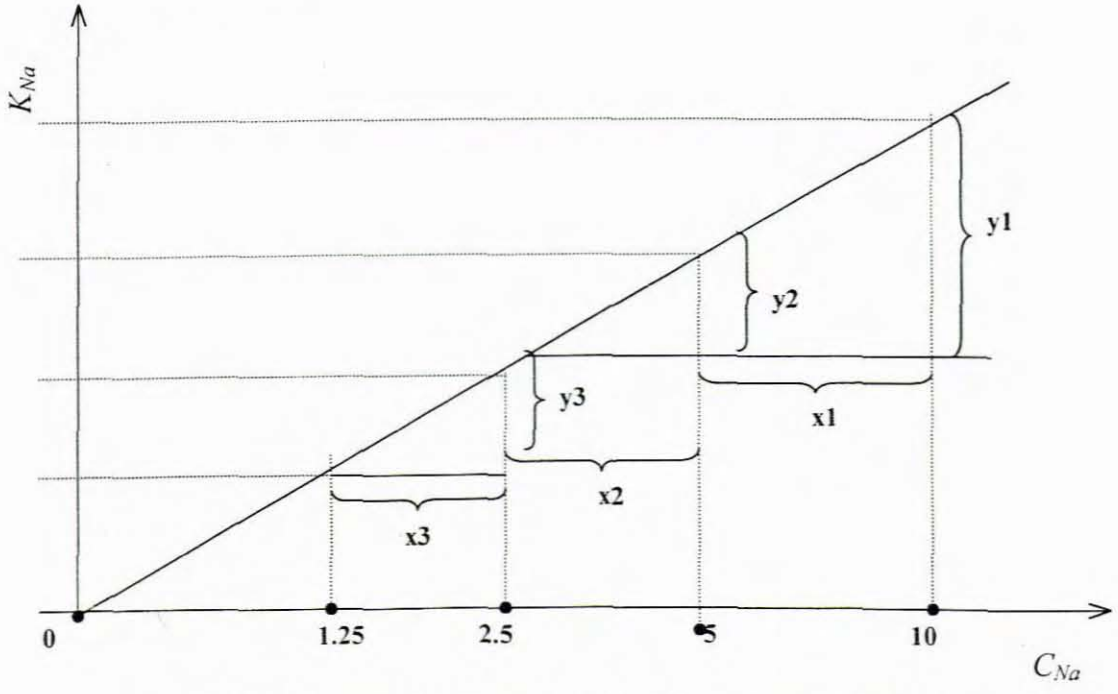


Figure 6.3a. Method for Determining a Gradient. Coefficient for Sodium.

$$\Delta k^*_{Na}1 = \tan\frac{y1}{x1} = \frac{conductivity\,(0.01) - conductivity\,(0.025)}{7.5} \tag{6.2a}$$

$$\Delta k^*_{Na}2 = \tan\frac{y2}{x2} = \frac{conductivity\,(0.05) - conductivity\,(0.025)}{2.5} \tag{6.2b}$$

$$\Delta k^*_{Na}3 = \tan\frac{y3}{x3} = \frac{conductivity\,(0.025) - conducivity\,(0.0125)}{1.25} \tag{6.2c}$$

$$\Delta k^*_{Na} = \frac{\Delta k^*_{Na}1 + \Delta k^*_{Na}2 + \Delta k^*_{Na}3}{3} \tag{6.3}$$

- Calculate more points from the graph for conductivity NaCl from 0 till 20.
- Draw the graph – using 1cm concentration and 1cm per conductivity
- Divide concentration axis with Δt = 0.2mm and calculate conductivity NaCl from formula

$$K_{NaCl} = \Delta k^*_{Na} \times concentration\ Na \tag{6.4}$$

- Introduce values of conductivity NaCl and concentration NaCl into two separate files.

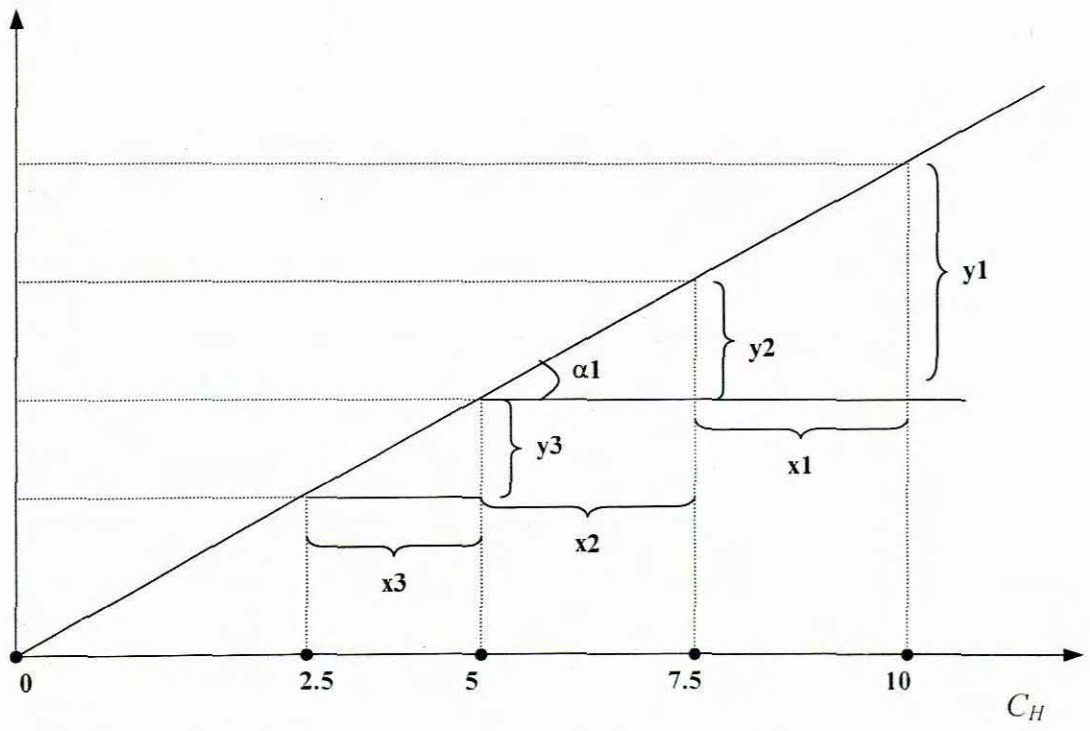


Figure 6.3b. Graph for Determining a Gradient Coefficient for H^+ Conductivity.

$$m1_{Na} = \tan\frac{y_1}{x_{1=5}} = (10,5) \tag{6.5a}$$

$$m2_{Na} = \tan\frac{y_2}{x_{2=2.5}} = (10,7.5) \tag{6.5b}$$

$$m3_{Na} = \tan\frac{y_3}{x_{3=2.5}} = (5,2.5) \tag{6.5c}$$

$$m = \frac{m1_{Na} + m2_{Na} + m3_{Na}}{3} \tag{6.6}$$

The real calculations according to the above procedure are given in tables 6.3 and 6.4 respectively:

		CALCULATION OF THE COEFFICIENT [bpH]	
4200	0.0087		
2100	0.005		
1400	0.0035		
1000	0.0028		
850	0.0024		
750	0.0022		
610	0.0019		
530	0.0017		
480	0.0016		
420	0.0015		
y1 =	3720	k*(H)1 = tan (y1/x1) =	-1.18415
x1 =	0.0071		
y2 =	270	k*(H)2 = tan (y2/x2) =	6.161163
x2 =	0.0006		
y3 =	60	k*(H)3 = tan (y3/x3) =	1.03297
x3 =	0.0001		
		k*(bpH) = [k*(H)1+k*(H)2+k*(H)3]/3 =	2.003329

Table 6.3. *Calculation of the pH Gradient Coefficient – using Experimental Data.*

CALCULATIONS OF THE COEFFICIENT [bNa]

1218	0.6482
680	0.3241
380	0.21606
340	0.16205
260	0.12964
215	0.10803
180	0.0926
160	0.0810

y1 =	1003
x1 =	0.54017

k*(Na)1 = tan (y1/x1) = 0.1425

y2 =	125
x2 =	0.05402

k*(Na)2 = tan (y2/x2) = -5.662

y3 =	55
x3 =	0.0270

k*(Na)3 = tan (y3/x3) = -1.478

k*(bNa) = [k*(Na)1+k*(Na)2+k*(Na)3]/3 = -2.332

Table 6.4. Calculation of the Sodium Gradient Coefficient – using Experimental Data.

6.1.1.3 Measurement of Conductivity

The conductivity meter has the following scales of measurement:

- Range conductivity-meter 0 – 500 [μS/cm]
  4 – 20mA

This means that for 0 μS/cm measurement in the solution the output reading from the probe is 4mA and for 500 μS/cm the probe gives 20mA. A formula is therefore required to calculate the range for the full scale and be used in the calculation procedure.

- Input range for the PC71 card 0 –20mA
- Output range from the PC71card 0 – 10V.

Since the PC30GA_ADV (DAQ) card accepts only voltages for its analog inputs (measured values). It means that another calculation must be performed to determine the corresponding voltage outputs from the PC71 that will be sent to the DAQ card.

Algorithm 6.1:

1) Formula for Calculating the Total Conductivity

$$K_T = m_i Imeasured - m_i 4 = m_i (Imeasured - 4). \quad (6.7)$$

When reading *Imeasured* = 4mA from the probe output, then

$K_T = m_i(4\text{mA} - 4\text{mA}) = m_i\,(0) = 0$

2) Calculating of the coefficient (m_I) for the current range

$$m_I = \frac{range\ conductivity}{range\ current} = \frac{(500-0)\mu S/cm}{(20-4)mA}$$
$$= \frac{500}{16}\left[\frac{\mu S/cm}{mA}\right]$$
$$= 31.25\left[\frac{\mu S/cm}{mA}\right] \quad (6.8)$$

Then

$$K_T = 31.25 \times (Imeasured - 4)mA \quad (6.9)$$

3) Calculating of the coefficient (m_V) for the voltage range

Since the PC71 card has a range of $0-10V$ output range for every 0 – 20mA inputs, then,

- Range for the conductivity-meter 4 – 20 mA corresponds to 2 – 10V.
- Then

$$K_T = m_V Vmeasured - m_V \cdot 2 = m_V(Vmeasured - 2) \quad (6.10)$$

$$m_V = \frac{(500-0)\mu S/cm}{(10-2)V} = \frac{500}{8}\left[\frac{\mu S/cm}{V}\right]$$
$$= 62.5\left[\frac{\mu S/cm}{V}\right] \quad (6.11)$$

Therefore

$$K_T = 62.5\left[\frac{\mu S/cm}{V}\right](Vmeasured - 2)[V] \quad (6.12)$$

- **Procedure for Determining Inputs to the DAQ card**

1. Measure conductivity from the solution.
2. Get the corresponding current value from the conductivity meter.
3. Convert the current value to voltage (PC71 – current to voltage converter).
4. Subtract 2V from the output voltage from the card (Vmeasured).
5. Multiply the new voltage value by 62.5 to receive K_T (total conductivity in μS/cm).

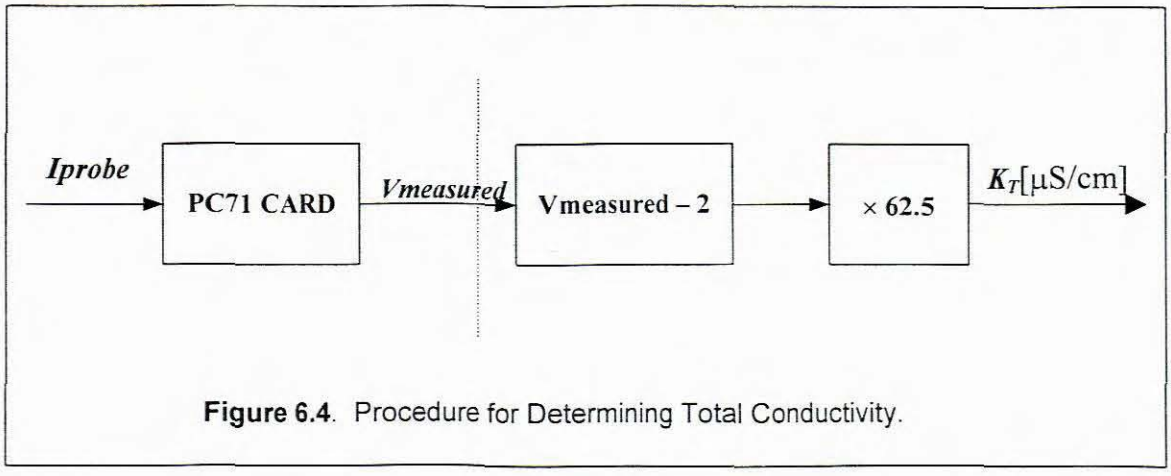


Figure 6.4. Procedure for Determining Total Conductivity.

6.1.1.4 Measurement of pH

The procedure for measuring pH is the same as for measuring conductivity the only difference will be the coefficient values in the calculation procedure.

- Range pH-meter 0 – 14 pH
  4 – 20mA

The same interface card (PC71) is used for converting current values from the pH-meter to the DAQ card.

- Input range for the PC71 card 0 –20mA
- Output range from the PC71card 0 – 10V.

Algorithm 6.2:

1) Formula for Calculating Total pH

$$pH_T = (m_{pH} Imeasured - m_{pH} 4)[pH]$$

$$pH_T = m_{pH}(Imeasured - 4)[pH] \qquad (6.13)$$

2) Calculating of the coefficient (m_{pH}) for the current range

$$m_{pH} = \frac{range\ pH}{range\ current} = \frac{(14-0)pH}{(20-4)mA} = \frac{14}{16}\left[\frac{pH}{mA}\right] = 0.875\left[\frac{pH}{mA}\right] \qquad (6.14)$$

$$pH = m_{pH}(Imeasured - 4)[pH]$$

3) Calculating of the coefficient (m_v) for the voltage range

- Range for the pH-meter 4 – 20 mA corresponds to 2 – 10V.
- Then

$$m_{pH} = \frac{(14-0)pH}{(10-2)V} = \frac{14}{8}\left[\frac{pH}{V}\right] = 1.750\left[\frac{pH}{V}\right] \tag{6.15}$$

Finally

$$pH = m_{pH}\left[\frac{pH}{V}\right](Vmeasured - 2)[V] = 1.75(Vmeasured - 2)\left[\frac{pH}{V}\right][V] \tag{6.16}$$

- **Procedure for Determining Inputs to the DAQ card**

1. Measure pH from the solution.
2. Get the corresponding current value from the pH-meter.
3. Convert the current value to voltage (PC71 – current to voltage converter).
4. Subtract 2V from the output voltage from the card (Vmeasured).
5. Multiply the new voltage value by 1.75 to receive *pH* (total pH).

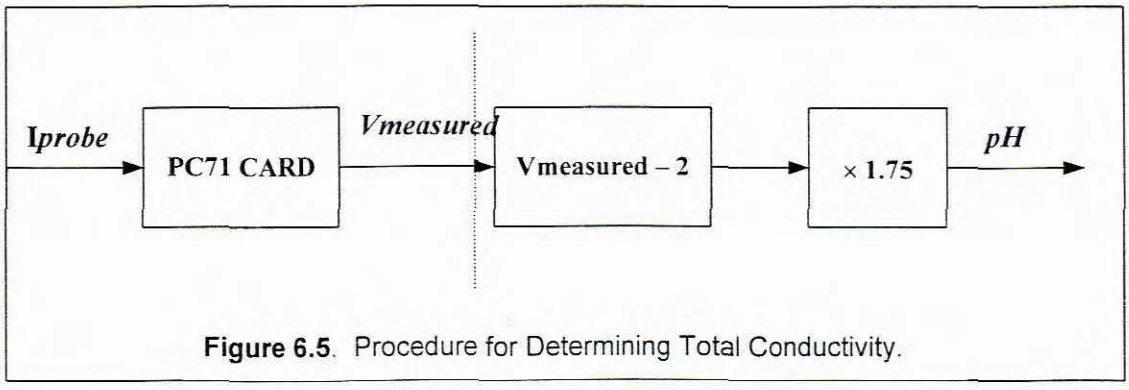


Figure 6.5. Procedure for Determining Total Conductivity.

6.1.1.5 Procedure for Determining Input (y_f) and Output (z) Concentration

Then aim of this procedure is to calculate the final concentration used for model and control calculations, as per step (5) of the pH/conductivity technique above. The final concentration equation is given by:

$$y = \frac{C_{Na}}{C_{Na} + C_{pH}} \equiv \frac{[mg/l]}{[mg/l]+[mg/l]} \tag{6.17}$$

The value of y can then be used for the determination of the measured input concentration (y_f) of the process (ion exchange, cation load column), for the model. This value can also be used for determination of the measured concentration at the output of the column (z).

Algorithm 6.3:

1. Get the pH value from Fig.6.4
2. Determining C_{pH} $[eq/l]$ by raising 10 to power pH value (10^{pH}) and take the inverse of this value.

$$C_{pH} = \frac{1}{10^{pH}}[eq/l] \tag{6.18}$$

3. Multiply this value by b_{pH}, the gradient as determined from experimental data (Table 6.2) to get K_{pH} $[\mu S/cm]$. This value will be used for two calculations.

$$K_{pH} = C_{pH} \times b_{pH}[\mu S/cm] \tag{6.19}$$

4. Multiply the K_{pH} value by 0.1 to receive K_{pH} in $[mg/l]$.
5. Multiply the K_{pH} from step (4) by 6.6 to receive this value in $[mg/l]$. This value is now ready for determination of the final concentration.
6. Get K_T value from Fig. 6.5.
7. Determine conductivity sodium (K_{Na}) by subtracting K_{pH} from (6) from K_T value.

$$K_{Na} = K_T - K_{pH}[\mu S/cm] \tag{6.20}$$

8. Determine sodium concentration, C_{Na} by dividing the K_{Na} $[\mu S/cm]$ value by the gradient (b_{Na}) calculated from experimental data (Table 6.1). This C_{Na} is in $[g/l]$.

$$C_{Na} = b_{Na} \times K_{Na}[g/l] \tag{6.21}$$

9. Multiply the C_{Na} value in step (8) by 1000 above to convert it to $[mg/l]$.
10. Add this value to the K_{pH} [mg/l] value calculated in step (5) to get the denominator of the y equation, (6.17).

$$C_{Na} + C_{pH} \tag{6.22}$$

11. Finally divide C_{Na} by the denominator (6.22) in step (10) to find y.

6.1.1.6 Block Diagram for the Procedure for Determining Input (y_f) and Output (z) Concentration

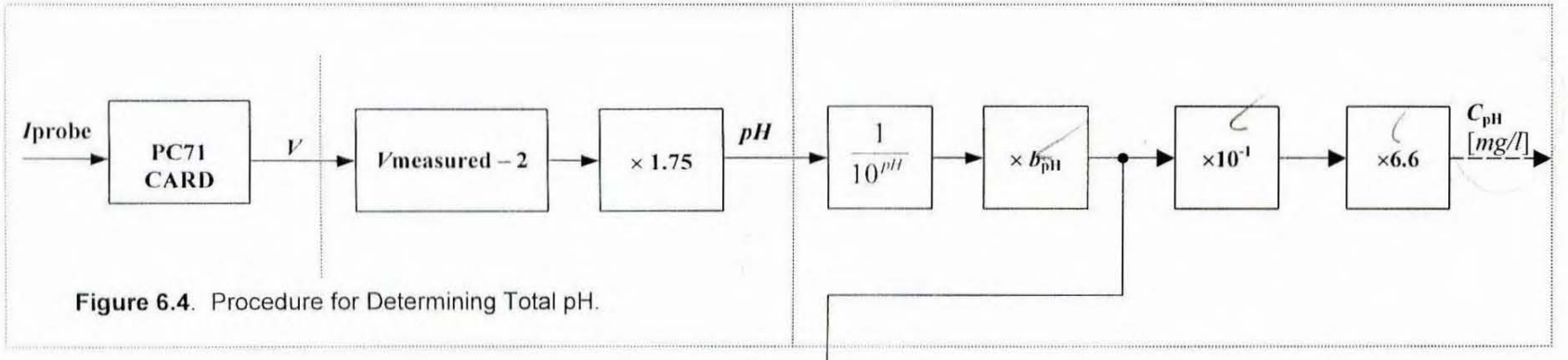


Figure 6.4. Procedure for Determining Total pH.

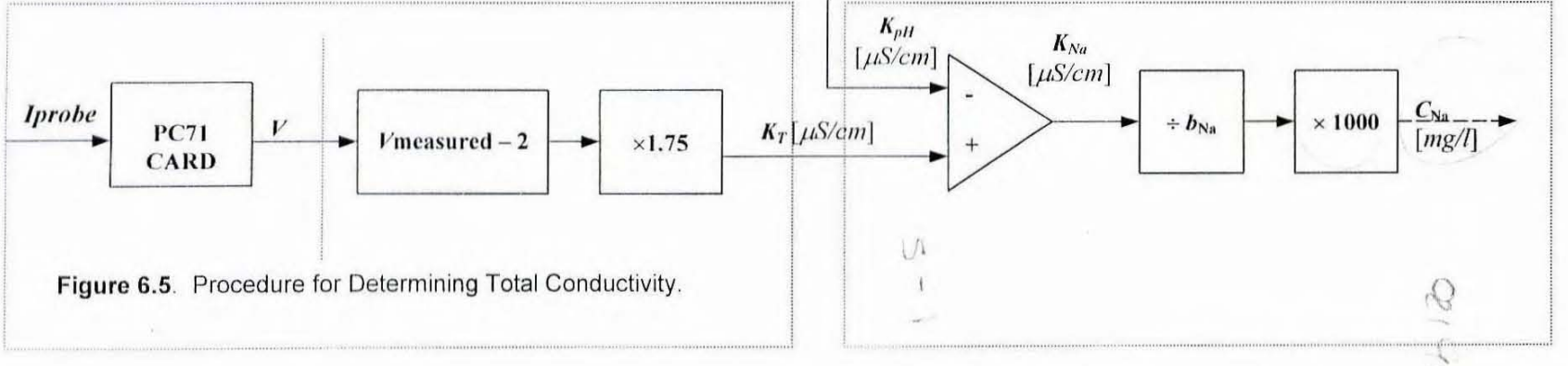


Figure 6.5. Procedure for Determining Total Conductivity.

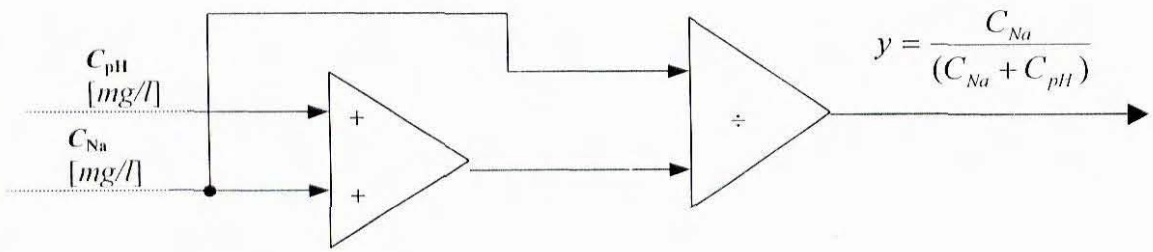


Figure 6.6. Procedure for Determining Final Concentration from the Measured pH and Conductivity.

6.1.1.7 Digital Control of the Analog (pH and conductivity) Measurements

This section controls the switching sequence for input or output measurements of the analog readings from the pH and conductivity probes. A required stream will be run for five minutes before switching over to the next. The algorithm below gives the order of events. The program is included in APPENDIX B.

Algorithm 6.4:

1) Select the input stream to the measuring tank by opening input stream valve (V19).
2) Measure and acquire pH and conductivity of the input stream for the set time.
3) Wait for the set time to lapse.
4) Close the tank input stream valve.
5) Select the output stream to the measuring tank by opening output stream valve (V18).
6) Allow for 30 seconds to lapse to flush out the previous stream.
7) Measure and acquire pH and conductivity of the output stream for the set time.
8) After the set time has lapsed allow for 30 seconds.
9) Check for stopping condition.
   - If FALSE go back to step 1.
   - If TRUE stop the program.

6.1.2 Measurements and Acquiring of Digital Values

Level sensors are connected at different strategic point for monitoring of levels on the top catchpots, hoopers and feed tanks as shown in Fig, 4.1 in chapter four. For the level sensors in the feed tanks all the three probes are connected in order to monitor both high and low levels. The level sensors in the hoopers and top catchpots have two bridged probes in order to monitor either a high or a low level condition. The basic structure for acquiring these values is indicated below Fig. 6.7.

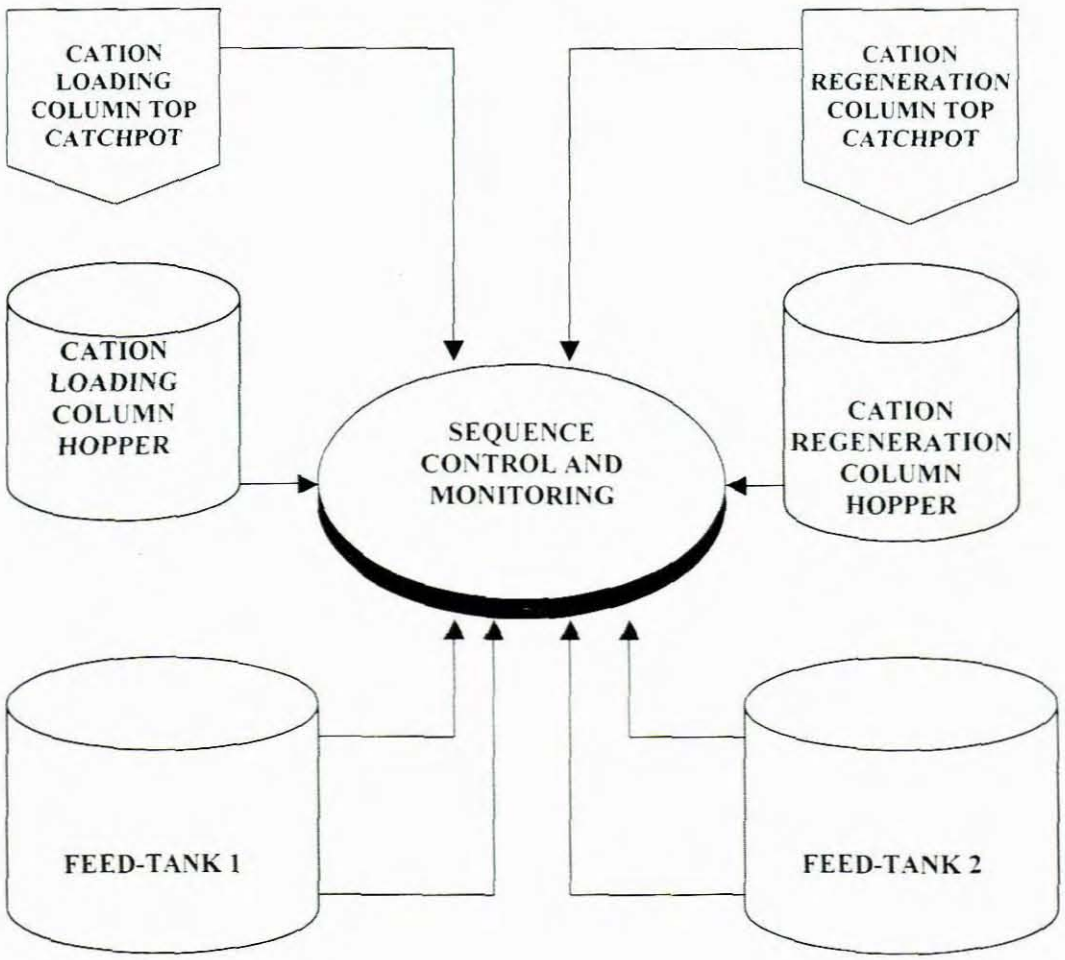


Figure 6.7. Measuring and Acquiring of Digital Values.

Two high level sensors are placed in the highest level of these vessels, one on the cation loading top catchpot and another one on the cation regeneration top catchpot. These signals indicate if the resin can be entered to the column or not. The level sensors at these two vessels are set to give a signal if a specified high level is reached. If a signal is received from the sensors, this indicates that the top catchpot is full and resin must not be transported into this column. These sensors are wired to indicate only one level, a high level, Table. 6.4. The high level signal received from these sensors determines if the top catchpot is holding enough resin, i.e., if a top catchpot cannot hold extra resin, a high level signal is given to indicate that the vessel is full.

Another set of two level sensors is placed in the lowest levels of the hoopers (APPENDIX A), one is for the cation loading hooper and another one for the cation regeneration column hooper. These sensors are connected to indicate only low levels (Table.6.4). When a signal is received from these sensors it means that the up-flow period in progression and no resin can be entered into either loading or regeneration columns. These sensors will go off once the level is lower than their position, this will happen during pull-down period.

And finally a set of two level sensors is connected such that two levels (high and low) can be detected. These level sensors are placed in the two feed tanks. They are for indicating if there is enough feed in each tank. The high levels from the tanks will indicate that the tank is full and no more feed must be entered. A low-level signal from any of these sensors will mean the tank is empty and more feed is required, Fig. 4.1.

6.1.2.1 Experiments for Different Mode of Operation of Level Sensors

The LV1 offers fail-safe control and monitoring of high and/or low levels of conductive liquids. The unit can be configured for "filling or "emptying applications by means of rear mounted DIP switches.

In applications where only one level is monitored, contact 5 and 7 (Table 6.6) should be bridged and used as the common probe and contact 6 should be used as the level-sensing probe. For sensoring of both high and low level like in the feed tanks, all of the three probes are used with contact 7 as a common probe (Table 6.6).

FILLING: The relay will energize if the level drops below the low-level probe. The relay will remain energized until the level reaches the high-level probe, when the relay will de-energize.

EMPTYING: When the level reaches the high level probe the relay will energize and remain in this state until the level falls below the low level probe, when the relay will de-energize.

The in or out condition in the table refers to the probe being in water or not. The 1 and 0 at the output refers to the high or low condition of the output.

	COMMON 7	LOW 6	HIGH 5	OUTPUT
Emptying 1+3	In	In	Out	1
	In	In	In	0
	In	In	Out	0
	In	Out	Out	1
Emptying 1+4	In	In	Out	0
	In	In	In	1
	In	In	Out	1
	In	Out	Out	0
Filling 1+3	In	In	Out	0
	In	In	In	1
	In	In	Out	1
	In	Out	Out	0
Filling 1+4	In	In	Out	1
	In	In	In	0
	In	In	Out	0
	In	Out	Out	1

Table 6.5. Experimental Data Obtained using Level Sensors for Two-Level *Condition*.

	Common (7+5)	Low/High	Output
Emptying 1+3 (high level)	In	Out	1
	In	In	0
Emptying 1+3/4 (low level)	Invalid		
Emptying 1+4 (high level)	In	Out	0
	In	In	1
Filling.1+3 (low level)	In	In	1
	In	Out	0
Filling 1+3/4 (high level)	Invalid		
Filling 1+4 (low level)	In	In	0
	In	Out	1

Table 6.6. Experimental Data Obtained using Level Sensors for only one Level Condition.

6.1.2.2 The Program for Acquiring of Digital Values

Currently six digital input lines are used for acquiring of digital signals from the plant. These digital signals are acquired at different parts of the plant as indicated in the block diagram Fig.6.7. A program to acquire these signals is given in APPENDIX B. The program acquires these signals and then switches a corresponding valve as per requirement.

The program in conjunction with the control sequence program monitors the resin to be fed to the loading column after regeneration by monitoring the level sensor status. If pull-down period is on for either regeneration or loading columns, low level sensors (hoopers – regeneration or loading) should come on immediately if the level drops below a set point (Fig.4.1). This allows resin to be fed into any column at that moment if the top catchpot has enough resin to be sent.

If there is enough resin in the in any of the top catchpot the high level sensors should come on immediately when a set-point for high level is reached. This signal is then sent to the sequence program to stop any transportation of resin to these catchpots.

The program also monitors the status of the feed tanks. If any of the tanks is full the program from detecting a high level coming on should shut the inlet valve to that tank. If the feed has been used up the low level sensors in any of the tank that has reached low level will give a signal and this will be used as an indication to the program to open the inlet valve to fill up that particular tank again.

6.2 MODELLING AND PARAMETER ESTIMATION

6.2.1 Theoretical Model Calculation

Some parameters must be determined from theoretical values and some from experimental data for the identification problem. Parameters with values to be determined from experimental data were obtained from the previous data. And then with the availability of that data the theoretical variables were then determined, A, B, $B1$, W matrices were determined.

6.2.1.1 Calculations of Parameters for the Theoretical Model

$N = 8$, number of stages.

$y_1, y_2, y_3, \ldots y_8$, mole fractions of the sodium (Na) in liquid phase (states).

F_R, rate of resin in the column (control)

T, length of the up-flow time (real input)

Calculations of T from the mass balance expressed by the volume of the resin in the stage from equation (3.80).

$$F_R T = hd$$

$$T = \frac{hd}{F_R} \qquad (3.80)$$

where, d – is the fraction of the resin holdup which is moved from one stage to another,

h – resin holdup calculated or given,

H – resin holdup calculated or given.

Load Column dimensions: Outside diameter = $4dm$ (400mm)

Inside dimension = $3.8dm$ (380mm)

Regeneration Column dimensions Outside diameter = 1.6*dm* (400mm)

Inside dimension = 1.4*dm* (1.4mm)

Both Columns Depth = 0.5*m* = 5*dm*

F_L = 2000 dm^3/h (measured)

F_R = I2.63

H =37.86

h =28.43

$\overline{C}$ = 1.5 or 1.8

C = O.05N

a_n ,n=$\overline{1,8}$

b_n, n=$\overline{1,8}$

6.2.1.2 Matlab Program for Calculation

The Matlab program for calculating the remaining model variables is included in APPENDIX B.

6.2.1.3 Simulink Program for Simulation

The developed model is simulated using Matlab Simulink and this program is also included in APPENDIX B.

6.2.2 Parameter Estimation

6.2.2.1 HIQ Program

A program for parameter estimation is attached in APPENDIX B. This program is writtem usin HIQ program.

6.3 OPTIMAL CONTROL CALCULATIONS

6.3.1 Steady State and Dynamic Solution Programs

Steady state program has been developed using LabVIEW programming package for determination of the control value at steady state (F_R^s). The algorithm for this program is given is chapter four, (Algorithm 4.3).

The Dynamic model forms the second sublayer of solving the optimization problem. It deals with maintaining the optimal values as calculated from the first sublayer (steady state model). Due to disturbances (changing of salt concentration of feed water) that occur during the exchange these values cannot be maintained for a long period. For this reason new optimal values have to be calculated based on the disturbance values and this is known as repetitive optimization.

The dynamic model program has been developed using Matlab based on Algorthim 4.4. Both these programs are included in APPENDIX B.

6.4 SEQUENCE CONTROL

- Calculate a new set-point (y^s) based on the calculated salt concentration
- Perform optimization computation
- Calculate up-flow periods (T^S and T^{SR}) for the load and regeneration columns based on the optimal values
- Use these optimal up-flow values to initiate appropriate control action (if needed)

A sudden change in the feed concentration results in an immediate compensation in upflow time, based on the measured values of pH and conductivity, the question that may come to mind is what happens to the old value that was stored in the program. Do we terminate and introduce the new value immediately or do we carry on until that period has lapsed and then introduce the new value. This has more further problems in that if we do not change the value what about the new feed concentration we are treating currently.

This problem is solved by the condition:

- Is sodium input feed concentration greater that 0.1 of the previous one?
  - ✓ If this is true then one needs to start a new optimal control calculation.
  - ✓ If this condition is not true the current value is equated to the previous.

The program is developed using LabVIEW with EDR functions for driving the IO board. This program is also included in APPENDIX B.

6.5 CONCLUSION

Analog and digital algorithms to be used for developing control programs and calculation of the feed salt (NaCl) concentration have been developed. Determination of feed salt concentration directly affects the optimal control since the main object is centred around the amount of salt in the feed water.

A program for acquiring of pH and conductivity values has been developed and it proved to be successful. Digital values acquiring program has been developed and has been successfully tested. Data acquiring runs were done in the plant and were successful.

Theoretical programs were tested in the laboratory and they all proved to be successful. HIQ program for parameter estimation and Simulink program for simulation of the process model have been successfully tested. A Matlab program for calculating dynamic model values has also been develop and it also produced positive results.

Chapter Seven

Conclusion

Fresh water like all other natural resources is becoming a scarce commodity due to growth in population and industrial needs, sources of recoverable water with each requiring different degree of treatment for potable use have been identified sea water, natural precipitation underground water and reclaimer sewage plant discharge

Water upgrading and recovery can be divided into two major categories:

- systems that provide a desalination function
- systems that provide a tertiary treatment function

In considering any of the above options, the costs of the process involved in the recovering water is a major determining factor. The emphasis of this research work is on the reuse of domestic water supplies, starting with a secondary sewage effluent as a feed to the cation column and producing water of a potable quality. The ion exchange (IX) is a very convenient chemical process for wastewater desalination, this has been established from previous studies. Ion exchange process is further suited for recovery and reuse of industrial effluent as equally important as to water recovery, for the following:

- desalination of hard brackish water
- combined neutralisation and desalination of acidic effluents, such as acid mine drainage, acid mine tailings etc.
- removal of organics and colloidal particulates.

7.1 PROBLEMS SOLVED IN THE DISSERTATION

The progress in computational methods for solving optimization problems leads to the possibility to develop dynamic optimal control for chemical processes and realize them to the real plants.

This work concerns the development of methods, algorithms and programs for modelling and dynamic optimal control design and implementation of the ion exchange process. By using detailed model and decomposition approach two types of optimal controls are developed:

- Startup of the ion exchange process
- Optimal control in the presence of slowly varying disturbances

From experimental studies it is found that not the exact optimal control profiles developed, but the concepts revealed from the optimization are important for improving and optimizing the dynamic operation of the ion exchange column.

7.1.1 Hardware Structure

- The designed instrumentation is broadly divided into digital and analog section. The digital control has two responsibilities controlling of the flows (including for analog measurements and sequential control) and acquiring of level sensors from the plant.
- The instrumentation has been kept at very low cost as per requirements but still achieving the best possible results.
- Instrumentation bought from different vendors has been successfully interface and developed. Run tests have been performed on the pilot plant and successful results were achieved.

7.1.2 Data Acquisition

There are two sets of data values that are acquired, the analog values for analog (pH and conductivity) measurements and another set for digital values acquired from the level detectors in the plant. Together these data sets are used for calculation of optimal control values.

- LabVIEW programs are developed for calculation of optimal parameters to be used with the developed model.
- LabVIEW software is used with PC30GA_ADV (IO board), PC38 X (current module), IDC5A (digital input modules), OAC5A (digital output modules), PB24 (digital modules mounting rack), electro-LV1 Level Controller, ASCO diaphragm valves and Bulletin 8320 solenoid valves and 5V DC International Power (power supply), to acquire real time digital data to and from the developed pilot plant for acquiring digital values. This also provides the control sequence to the pilot plant.
- LabVIEW software is also developed to work with PC71 (current-to-voltage converter), (pH sensor and meter), CONDUCTIVITY 8225 (conductivity sensor and meter) and 12V DC International Power (power supply) to solve the problem of acquiring analog data (pH and conductivity measurements) from the plant.

7.1.3 Modelling and Parameter Estimation

The following are the problems the dissertation solved in addressing the global aim of the study undertaken:

- Operating parameters (control variables) are identified for the ion exchange process (as used for water desalination) as an object of control.
- Model parameters are then identified for developing a mathematical (theoretical) model and estimated using experimental data received from the previous

investigation on the process. Method for algorithm and program are developed for parameter estimation using gradient procedure.

- The mathematical model is then transformed into a discrete one to be used with the digital computer for achieving optimization requirements.

7.1.4 Optimal Control Strategy

- Optimal control strategy is proposed on the basis of multilayer control and disturbance analysis.
- The structure consists of self-organization, adaptation, optimization and direct control layers.
- The problem for optimization is then decomposed according to the frequency of the disturbance. A decomposition method is used for solving steady state subproblems of the optimization layer.
- Two subproblems are formulated steady state optimization that is solved by steady state model and steady state stabilization solved by a dynamic model.
- Matlab programs are developed for solving the controller linearization problem of the dynamic model.

7.1.5 Minimum Time Problem

- Minimum startup time is formulated on basis of minimum time requirement for the process to reach steady state.
- A method for optimization of minimum startup time period is developed based on Lagrange funcionals of optimality. The algorithm for the method is developed.

7.1.6 Optimal Control

- A two-layer control method is developed as a base for optimal control strategy in the presence of varying disturbances. The control strategy is based on the following three factors:
    - the goal of the process,
    - type of the process and
    - the used technology.
- Lagrange functionals are again used as a solution for the two-layer optimal control problem.

7.1.7 Sequential Control

- The control sequence is proposed on the basis of analysis of process flows.
- Program in LabVIEW is developed to realize the control sequence.
- All programs are then linked together in order to realize the optimal control strategy.

7.2 BENEFITS OF THE ON-LINE OPTIMIZATION

Benefits of process optimization come from improved plant and business performance. In general terms, the revenues come from improved yields of valuable products, reduced energy consumption, and higher processing rates either through increased capacity of existing equipment or longer stream factors. Optimization may also influence reduction in a number of other operating costs including maintenance, equipment wear and staff utilization.

The engineering benefits come from improved process troubleshooting and assistance in making quicker and more accurate decisions. This ultimately relates to improved process operations. There is also an interaction with process design of new units. The knowledge that comes from computer optimization can have a bearing on equipment sizing and upon assumed capacity factors.

7.3 FUTURE DEVELOPMENTS OF METHODS AND APPLICABILITY

The future outlook for application of systems for control and online optimization for control of industrial process is bright. The technical concepts are fundamental to progress. All parties taking parts in the building of such systems such as technology, people, computers, measurements, incentives, limitations are continuously improved and developed with the years (Harrison and Straut, 1978).

The overall shape of process control systems is becoming more generalized. Rather than appearing as a complicated set of loops on a process and instrument drawing with hundreds of set-point input values it can be conceived as a general functional block, representing the computer. From this perspective optimizing control becomes a broad, complex technology. It is an evolving philosophy for bridging the gap between the plant equipment and the desire of people. It is impinging on the work interface of the process operators, instrument people, plant engineers and corporation management. We are entering an era of interesting digital automation and control.

7.3.1 The Future Development of the Proposed Structure

- Development of the anion loading and regeneration columns.
- Introduction of measurement of concentration in every stage.
- Introduction of fully decentralized control.
- Development of method for algorithm and programs for on-line estimation of the model.
- Connection of estimation and optimal control problem solution.
- Remote control of the process from the Department of Electrical Engineering.
- Development of practicals for students in control subjects.

7.3.2 Application of Developed Method Algorithm and Programs

1. In water purification plants.
2. In mining industry for extraction of metal ions.
3. In sugar and pharmaceutical industry.
4. For control purposes in education of chemical and electrical engineering students.
5. As an application plant for different control strategies.

7.4 PUBLICATIONS IN CONNECTION WITH THE DISSERTATION

1. Dube, N., R.Tzoneva. Method for start-up optimal control of technological processes. *Proc. of the International Conference AFRICON'99*, 1999, 517 – 520.
2. Dube, N., R.Tzoneva. Method for optimal control of ion exchange process for desalination of water. *Proc. of the 3 rd International Conference on Control Theory and Application, CDROM, ISBN:981-04-4794-9*, 2001.

References

References

ATHANS, M. 1971: On the design of PID using Optimal Linear Regulator Theory. Automatika, vol. 7, pp. 643 – 647.

AMUNDSON, N.R. 1966: Mathematical Methods in Chemical Engineering Matrices and Their Applications. New York: Englewood Cliffs, Prentice Hall.

APPLEBAUM, S.B. 1968: Demineralisation by Ion Exchange. New York: Academic Press.

ARDEN, T. 1968: Water Purification by Ion exchange. London: Butterworths.

BARSA, A.; DE RE, G & FOSCOLO, P.O. 1980: Numerical Simulation of an Ionic Exchange Bed. In Proceedings of the 7th International Symposium on Fresh Water(?), vol. 2, pp. 13 – 23. Amsterdam.

BECERRA, V. & ROBERTS, P. 1996: Dynamic Integrated System Optimization and Parameter Estimation for Discrete Time Optimal Control of Nonlinear Systems. International Journal of Control, vol. 63, no. 2, pp. 257 – 281.

BELTER, P.A. & SPEAKER, M. 1967: Controlled-Cycle Operations Applied to Extraction Processes. I &EC Process Design and Development, vol. 6, no. 1, pp. 36 –40.

BOSTON, J.F. & SULLIVAN, S.L. JNR. 1972: An improved Algorithm for Solving Mass Balance Equations in Multistage Separation Processes. Canadian Journal of Chemical Engineers, vol. 50, pp. 663.

BOX, G.E. & DRAPER, N.R. 1987: Empirical Model Building and Response Surfaces. New York: Wiley.

BOYD, G.E.; ADEMSON, A.W. & MYERS, S.L. 1974: Kinetics of Ion Exchange Adsorption Solution Processes. AIChE (American Institute of Chemical Engineers), vol. 69, pp.2836 – 2848.

BROGAN, W.L. 1974: Modern Control Theory, 3rd Edition. New Jersey: Prentice Hall Inc.

BUCKELY, P. 1964: Techniques of Process Control. New York: Wiley.

References

BUNZL, K. 1995: Calculation of the Helfferich Number to Identify the Rate-Controlling Step of Ion Exchange for a Batch Process. Industrial Engineering and Chemical Research, vol. 34, pp. 2584 – 2587.

BUTTLER, J.N. 1964: Ionic Equilibrium, a Mathematical Approach. Class Catalogue, no. 65/1841.

CADZOW, J.A. & MARTENS, H.R. 1970: Discrete Time and Computer Control Systems. New York: Prentice-Hall, Englewood Cliffs.

CARNAHAN, B.; LUTHER, H.A; & WILKES, J.O. 1969: Applied Numerical Method. New York: John Wiley and Sons.

CARSLOW, N.S. & JOLGER, J.C. 1959: Conduction of Heat in Solids. Oxford Clarendon Press.

CELIKOVSKY, S. 1993: On the Stabilization of the Homogeneous Bilinear Systems. System Control Letters, vol. 21, pp. 503 – 510.

CLOETE, F. & STREAT, M. 1963: A New Continuous Solid-Fluid Contacting Technique. Nature, vol. 200, pp. 1199 – 1200.

COLEMAN, T.; BRANCH, M.A. & GRACE, A. 1999: Optimization Toolbox for use with MathLab. User Guide, Version 2. Math Works Inc.

COULSON, J.M. & RICHARDSON, J.F.; BACKHURST, J.R. & HARKER, J.H. 1997: Chemical Engineering. Volume 1: Heat Flow, Heat Transfer and Mass Transfer. Oxford: Butterworth-Heinemann Ltd. (Pergamon Press).

COULSON, J.M. & RICHARDSON, J.F.; BACKHURST, J.R. & HARKER, J.H. 1978: Chemical Engineering Volume 2: Unit Operations, 3rd Edition. United Kingdom: Headington Hill Hall. (Pergamon Press).

CRAIG, W.M.; HAINES, A.K.; NICOL, D.I.; OLËN, C.M.; DOUGLAS, M.G. & LOUW, G.D. 1978: The Design and operation of Continuous Ion-Exchange Demonstration Plant for Recovery of Uranium. Journal of .South African Instr. Min. Metall, pp. 316 – 324.

References

CRANK, J. 1956: The Mathematics of Diffusion. New York: Oxford University Press.

DAVIS, M.E. 1984: Numerical Methods and Modeling for Chemical Engineers. New York: Wiley.

de GRAYTEZ, W. 1991: Ion Exchangers. New York:

de SILVA, F.J. 1995: Essentials of Ion Exchange. Paper presented on Ion Exchange/Resin, delivered at the Water Quality Association Annual Convention and Exhibition, Nashville, Tennessee. http://www.wqa.org/Technical/essentials-of-ionexchange, (March 15).

DEKKER, M. 1966: Ion Exchange. Editor, MAZINSKY, JA. New York.

DENBIGH, K.1955: The principles of Chemical Equilibrium. New York: Canbridge University Press.

DESHPANDE, P. 1995: Prospects for Advanced Controls in Desalination Plants. Proceedings of IDA World Congress on Desalination and Water Sciences, Abuphabi, pp. 189 – 203.

DESHPANDE, P.B. & CHEN, L.C. 1993: Real Time Silmulation and Advanced Process Control, Present Status and Future Trends. Proceedings of the 19th Annual Advanced Control Conference. Indiana: W.Lafayette.

DODDS, R.; HUDSON, P.I.; KERSHENBAUM, L. & STREAT, M. 1973: The Operation and Modelling of a Periodic, Countercurrent Solid-liquid Reactor. Chemical Engineering Science, vol. 28, pp. 1233 – 1248.

DORF, R.C. 1998: Modern Control Systems, 8th Edition, (BISHOP, R.H.). Addison Wesley.

DORFNER, K. 1972: Ion Exchangers. Properties and Applications. Michigan: Ann Arbor.

DUDAS, J. 1995: Applications of Ion Exchange. http://www.rpi.edu/dept/chem-eng/Biotech-Enviro/DUDAS/application. Applications of Ion Exchange.

References

DYER, A., HUDSON, M.J. & WILLIAMS, P.A. 1995. Ion Exchange Process: Advances and Applications. In Proceedings of the IEX'95, Wrecxham, no.196. UK: North East Wales Institute. ROYAL SOCIETY OF CHEMISTRY, 1997.

EAGLE TECHNOLOGY, 1999: EDR: Software Developers Kit for Eagle Technology Boards. User Manual for DOS and WINDOWS.

EAGLE TECHNOLOGY, 1996a: User Manual for the PC30F and PC30G Series Boards.

EAGLE TECHNOLOGY, 1996b: PC71, 0 to 20mA Current to Voltage Converter Module. For the PC 30xx Series/ PC 126 Data Acquisition Board.

EDGAR, T. & SEBORG, D. 1980: Feed-forward Control. AIChE, vol. 26, pp. 26 – 31.

FIEG, G.; WANZY, G. & KRUSE, C. 1993: Experimental and Theoretical Studies of the Dynamics of Startup and Product Switchover Operations of Distillation Columns. Chemical Engineering Processes, vol. 32, pp. 283 – 280.

FINDEISEN, W. 1980: Control and Coordination in Hierarchical Systems. Chichester, West Sussex: Wiley.

FISHER, W.; DOHERTY, M. & DOUGLAS J. 1984: An Evolution and Hierarchical Procedure for Optimization of Preliminary Process Design. Presented at an Annual Meeting of AIChE, Paper 104c. San Francisco.

FLEMING, C.A. & NICOL, M.J. 1983: A Comparative Study of Kinetic Models for the Extraxtion of Uranium by Strong base Anion Exchange Resins. Journal of South African. Instr. Min. Metall, February, pp. 89 – 99.

FORD, M. & KELLY, R.J. 1985: NIMCIX Absorption Columns for the recovery of Uranium at Vaal Reefs South Africa. Proceedings of the International Conference, MINTEK 50, pp. 511 – 518.

FORD. M.A. 1984: Council of Mineral Technology (MINTEK). The Simulation and Process Design of NIMCIX Contactors for the Recovery of Uranium. International Conference on Ion Exchange Technology, July, pp. 669 – 678. University of Cambridge.

References

FOUTCH, G.Z.; KAR, S & HIGH, M. 1995: Membrane Theory. Ultrapure Water Journal Workshop Series. Philadelphia.

FOUST, A.S.; WENZEL, L.A.; CLUMP, C.W.; MAUS, L. & ANDERSEN, L.B. 1980: Principles of Unit Operations, Second Edition. New York: John Wiley and Sons.

FRANKS, R. 1972: Modeling and Simulation in Chemical Engineering. New York: Wiley.

FRECMAN, R. & KOKOTOVIC, P. 1995: Optimal Nonlinear Controllers for Feedback Linearizable Systems. In Proceedings of the American Control Conference, vol. 4, pp. 2722 – 2726.

FUNKE, J.W. & COOMBS, P. 1973: Water and Effluent Management in the Electroplating and Anodizing Industry. Technical Guide, K29. National Institute for Water Research. Council for Scientific and Industrial Research, Pretoria, South Africa.

GENESIO, R & TESI, A. 1988: The Output of SISO Bilinear Systems. IEEE Transport and Automatic Control, vol. 33, pp. 950 – 952.

GEORGE, P.; ROSS, J & PRATER, J. 1968: Byproduct uranium Recovery with New Ion Exchange Technique. Mining Engineering, vol. 20, pp. 1 – 5.

GIDDEY, T.B.S. 1979: Ion Exchange for the Desalination and tertiary Treatment of Wastewater. PhD submitted to the University of Cape Town.

GLASKI, F.A. & DRANOFF, J.S. 1963: Ion Exchange Kinetics: A Comparison of Models. AIChE, vol. 9, no. 3, pp. 426 – 431.

GLIMSHAW, R.W. & HARLAND, C.E. 1975: Ion Exchange: Introduction to Theory and Practice. Chemical Engineering Society. London.

GLUECKAUF, E. 1955: Theory of Chromatography: Part 10. Formulae for Diffusion into Spheres and Their Application to Chromatography. Faraday Society, vol. 51, pp. 1540 – 1551.

GOVIND, R. & POWERS, G. 1982: Control System Synthesis Strategies. AIChE (American Institute of Chemical Engineers), vol. 28, no. 60.

References

GREIG, J.A. 1996: Ion Exchange Developments and Applications. In Proceedings of IEX'96, no. 182. United Kingdom: Nutrasweet Kelco Co.

GRÜNEWALD, M. & SCHMIDT-TRAUB, H. 1999: Absorption in Multistage Fluidized Bed Column. Chemical Engineering Technology, vol. 22, no. 3, pp. 206 – 209.

GUERRA, R & MORALES, J. 1995: Some Results About Nonlinear Observers for a Class of Bilinear Systems. Proceedings of the American Control Conference, Seattle, pp. 1643 – 1644.

HAMMOND, P. & WILLIAMS, B. 1994: On Determining a Dynamic Model of a Binary Distillation Column using a Computer Program. Advances of Chemical Engineering, London, pp. 1 – 22.

HARRISON, T. & STRAUT, T. 1978: Minicomputers in Industrial Control. ISA. Pittsburg: Pa, pp. 208.

HASKINS, D. 1983: Restraints on Entire Plant Optimization. Chemical Engineering Progress, vol. 79, no. 6.

HELFFERICH, F. 1962: Ion Exchange. New York: McGraw Hill.

HELFFERICH, F. & PLESSET, M.S. 1958: Ion Exchange Kinetics: A Nonlinear Diffusion Problem I. Chemical Physics, vol. 28, no. 3, pp. 418 – 424.

HENRY, B.A. 1982: Continuous Countercurrent Ion Exchange for Desalination and Tertiary Treatment of Effluents and Other Brackish Waters. Water Science Technology, vol. 14, pp. 535 – 552.

HENSON, M. & SEBORG, D. 1991. A Critical of Exact Linearization Strategies for Process Control. Process Control, vol. 1, pp. 122 – 139.

HIGGIN, J. 1984. Process Control No Longer Separate from Simulation Design. Chemical Engineering News, vol. 62, no. 14.

HIMMELBLAU, D. & BISCHOFF, K. 1968: Process Analysis and Simulation: Deterministic Systems. New York: John Wiley and Sons.

References

HÖEGFELD, E. 1991: The Höegfeld Three-Parameter Model. A Useful Approach for Summarizing Data on Ion Exchange. Proceedings of the International Conference on Ion Exchange, pp. 19 – 24.

HOLLAND, C.& LIAPSIS, A. 1983: Computer Methods for Solving Dynamic Separation Problems. New York: McGraw Hill.

HORN, F.J.M. January 1967: Periodic Countercurrent Processes. I&EC Process Design and Development (Industrial Engineering Chemical Process Design and Development), vol. 6, no. 1, pp. 30 – 35.

HORN, F.J.M. & LIN, R.C. 1967: Period Processes: A Variational Approach, I&EC Process Design and Development (Industrial & Engineering Chemical Process Design and Development), vol. 6, no. 1, pp. 21 – 30.

HULL, R., SCHUMACHER, D & QU, Z. 1995: Design and Evaluation of Robust Nonlinear Missile Autopilots from a Performance Perspective. In Proceedings of the American Control Conference, vol. 1, pp. 189 – 193.

HUNTER, P.R. 1987: Automated Process Control Systems. Concepts and Hardware, 2nd Edition. New Jersey: (A Division of Simon & Schuster). Prentice Hall Inc.

IDA, 1997: Measurement, Modeling, Identification and Optimized Controller Parameter Settings of MSF Desalination Units. World Congress of Desalination and Water Reuse, pp. 52 – 81.

ISIDORI, A. 1995: Nonlinear Control Systems. London: Springer.

JOHANSSON, R. 1993: System Modeling and Identification. New York: Prentice Hall.

JOHNSON, C.D. 1993: Process Control Instrumentation Technology, 4th Edition. New Jersey: Prentice Hall Inc.

JONES, L.D. 1991: Electronic Instruments and Measurements, 2nd Edition. New Jersey: Prentice Hall Inc.

JUNKINS, J.L. 1991: An Introduction of Optimal Estimation of Dynamic Systems. Alphen Aan Den Rijn: SIJHOFF and NOORDHOFF INTERNATIONAL.

References

KATAOKA, T; YOSHIDA, H & OZASA, Y. 1977: Breakthrough Curve in Ion Exchange Column-Particle Diffusion Control. Journal of Chemical Engineering Japan, vol. 10, no. 5, pp. 385 – 390.

KATOKA, T. & YOSHIDA, R. 1975: Resin Phase Mass Transfer in Ion Exchange between Different Ions Accompanied by Resin Volume Change. Journal of Chemical Engineering Japan, vol. 8, no. 6, pp. 451 – 456.

KELLER, M. 1992: Basic Ion Exchange for Residential Water Treatment. Sybron Chemicals Inc.

KELLY, R.M. 1995: General Processing Considerations. In Handbook of Separation Process Technology. Edited by ROUSSEAU. New York: John Wiley and Sons.

KILKAS, A.C. & HUTCHISON, H.P. 1980: Process Optimization using Linear Models. Computers and Chemical Engineering, vol. 4, pp. 39 – 48.

KIRK, D.E. 1970: Optimal Control Theory. An Introduction. Edited by NEWCOMB, R.W. New Jersey: Prentice Hall Inc.

KIRK-OTHMER ENCYCLOPEDIA. 1995. Encyclopedia of Chemical Technology. Volume 14: (Imaging Technology – Lanthanides), 4th Edition, executive editor KROSCHWITZ, J.I, edited by HOWE-GRANT. M.

KIRMAN, D. 1995: Mass Transfer Principles. In Handbook of Separation Process Technology, Edited by ROUSSEAU, R. New York: John Wiley and Sons

KODITSCHEK, D. & NARENDRA, K. Stability of Second Order Bilinear Systems. IEEE Transport and Automatic Control, vol. AC-28, pp. 987 – 989.

KOH, J.; WANG, N. & WANKET, P. 1995: Ion Exchange if Phenylalanine in Fluidizes Expanded Beds. Industrial Engineering and Chemical Research, vol. 34, pp. 2700 – 2711.

KORNGOLD, E. &. VOLSI, D. 1992: Water Desalination by Ion Exchange, Hollow Fibres. http://www2.uwin.siu.edu:4001/usr/local/data/wrsic/1992.txt_13475683_1992.

KORNGOLD, E. &. VOLSI, D. 1991: Water Desalination by Ion Exchange, Hollow Fibres. Desalination DSLNAH, vol. 84, pp. 123 – 135.

References

KOZIETULSKI, M. 1981: Two Layer Implementation of Repetitive Dynamic Optimization (Static-Optimally Aided Control). Archivum Automatiki I Telemeckaniki, vol. 26, no. 4, pp. 511 – 520.

KRAAIJEVELD, G.H. 1991: Wesselingh Ion Exchange Kinetics - The Maxwell_Stefaan Description Process. The International Conference on Ion Exchange, pp. 13 – 18.

KRAVARIS, C. & KANTOR, J. 1990b: Geometric Methods for Nonlinear Process Control. I: Controller Synthesis. Industrial Engineering and Chemical Research, vol. 29, pp. 2295 – 2310.

KRAVARIS, C. & KANTOR, J. 1990a: Geometric Methods for Nonlinear Process Control. II: Background. Industrial Engineering and Chemical Research, vol. 29, pp. 2295 – 2310.

KRENER, A. 1984: Approximate Linearization by State Feedback and Coordinate Change. Systems and Control Letters, vol. 5, pp. 181 – 185.

KRIVOSHEEV, V. & TORGSHOV, A. 1999: The Optimal Control of Processing Systems by Economical Criteria as Applied to Distillation. Proceedings of the 14th IFAC World Congress, Beijing, vol. F, pp. 229 –234.

KUNIN, R. 1972: Ion Exchange Resins. Edited by KRIGER, R.E. New York.

KUNIN, R. 1971: Elements of Ion Exchange. Edited by KRIGER, R.E. New York.

KUNIN, R. 1958: Ion Exchange Resins. 2nd Edition, New York: John Wesley and Sons.

KUO, J. & DAVID, M. 1963: Single Particle Studies of Cation Exchange in Packed Beds: Barium ion-Sodium ion System. AIChE, vol. 9, no. 3, pp. 365 – 370.

LATOUR, P.R. June 1979: Online Computer Optimization 1: What it is and where to do it. Hydrocarbon Processing, pp. 49 – 63.

LAPIDUS, L. 1962: Digital Computation for Chemical Engineers. New York: McGraw Hill.

LEVENSPIEL, O. 1962: Chemical Reaction Engineering. New York: John Wiley and Sons.

References

LEFKOWITZ, 1966: Multilevel Approach to Control System Design. Transcript of ACME, Journal of BE, no. 6.

LEWIS, K.A. & TUCKER, P. 1986: Computer Method for Simulating Relative Density Distribtion Analysis with Application to Gravity Separations. Transcripts from Instr. Min. Met. Section C, 95, pp. 35 – 40.

LI, P.; FLENDER, M.; WONZY, G. & FIEG, G. 1999: Development and Implementation of Dynamic Optimal Operation Policies for Distillation Columns. Proceedings of 14th IFAC World Congress, Beijing, vol. N[1], pp. 331 – 336.

LIPTAK, B.G. 1999: Instrument Engineer's Handbook. Process Control, 3rd Edition. CRC Press LLC.

LIOR, N. 1995: Measurement and Instrumentation in Desalination. Proceedings of IDA World Congress on Desalination and Water Sciences, Abu Dhabi, pp. 229 –248.

LOVLAND, J. 1968: Industrial Engineering Chemical Process Design and Development, vol. 7, pp.65.

LUYBEN, W.L. 1990: Process Modeling, Simulation and Control for Chemical Engineers, 2nd Edition. McGraw Hill Publishing Co.

LUYBEN, W. 1973: Process Modeling, Simulation and Control for Chemical Engineers. New York: McGraw Hill.

MATJOBA, I. 1997: Use of Continuous Distillation Columns for Batch Separation. Transcript from Industrial Chemical Engineering, vol. 75, Part A, no. 9, pp. 609 – 619.

MANKAT, P. 1974: Separation Science, vol. 9, no. 2, pp.85.

MARCEL DEKKER, INC. 1992: Encyclopedia of Chemical Processing and Design. Volume 27: (Hydrogen Cyanide – Ketones Dimethyl, Acetone). Executive editor, McKETTA, J.J., associate editor, CUNNINGNGHAM, W.A.

MARCHELLO, J.M. & DAVIS, M.W. 1963: Theoretical Investigation of Agitated Ion Exchange Beds. Industrial Engineering Chemical Fundamental, vol. 2, no.1, pp. 27 – 31.

MARINSKY, J. 1966: Ion Exchange. Volume 1. Edited by Marcel Dekker.

References

MARTINOLA, F. 1974: The Lewatit Ion Exchange in Fluidized Beds. Canadian Journal of Chemical Engineering, vol. 52, no. 1, pp. 25 – 27.

McCABE, W.L. & THIELE, E.W. 1925: Graphical Design of Fractionating Columns. Industrial Engineering and Chemical Research, vol. 17, pp. 605.

McGOVAN, wes. 1988: Water Processing. Water Quality Association.

McGRAVEY, F.X. & FIDHER, S.A. 1986: Measurements and Control in Ion Exchange Installations, chapter 2.8, pp. 404 – 427. In Measurements and Control in Water Desalination, edited by N. Lior. Amstserdam: Elsevier.

McGRAW-HILL ENCYCLOPEDIA. 1992: Encyclopedia of Science and Technology, vol. 9, 4th Edition.

MESAROVIC, M.D. 1970: Theory of Hierarchical, Multilevel Systems. New York: Academic Press.

MOHLER, R. 1973: Bilinear Control Processes, New York: Academic Press.

MONTGOMERY, J.M. 1985: Water Treatment Principles and Design. John Wiley and Sons.

MORARI, M.; ARKUN, J. & STEPHANOPOULOS, G. 1980: Studies in the Synthesis of Control Structures for Chemical Process. Part I: Formulation of the Problem. Process Decomposition and the Classification of the Control Tasks. Analysis of the Optimizing Control Structures. AIChE, (American Institute for Chemical Engineers) vol. 26, no. 2, pp. 220 – 232.

MORARI, M. & STEPHANOPOULOS, G. 1980: Studies in the Synthesis of Control Structures for Chemical Precess. Part II: Structural Aspect and the Synthesis of Alternative Feasible Control Schemes. AIChE, (American Institute for Chemical Engineers) vol. 26, no. 2, pp. 232 – 247.

MORARI, M. & STEPHANOPOULOS, G. 1980: Studies in the Synthesis of Control Structures for Chemical Process. Part III: Optimal Selection of Secondary Measurements within the Framework of State Estimation in the Presence of Persistent Unknown

References

Disturbances. AIChE, (American Institute for Chemical Engineers) vol. 26, no. 2, pp. 247 – 260.

MORBIDELLA, A., SEWIDA, A., STORTI, G. & CARRAS, 1982: Simulation of Multi-component absorption beds. Model Analysis and Numerical Solution. Industrial Engineering Chemical Fundamentals, vol. 21, no. 2, pp. 123 – 131.

NACHODI, F.C. 1956: Ion Exchange Technology. Academic Press Inc.

NATIONAL INSTRUMENTS CORP. 1998a: LabVIEW Basics I. National Instrument Corp. http://natinst.com.

NATIONAL INSTRUMENTS CORP. 1998b: DAQ. SCC Series User Manual. National Instrument Corp., June. http://natinst.com.

NATIONAL INSTRUMENTS CORP. 1999: DAQ. PCI-E Series User Manual. National Instrument Corp., July. http://natinst.com.

NEUMAN, C.P. & BARADELO, C.S. 1979: Digital Transfer Functions for Microcomputer Control, IEEE (Institute of Electrical and Electronic Engineers) Transport Systems, Man., Cybernetics, SMC-Y, no. 12, pp. 856.

NICULESCU, S.; DION, J. & DUGARD, L. 1995: Stabilization Criterion for Bilinear Systems with Delayed State and Saturating Actuators. Proceedings of IFAC Conference: System Structure and Control, Nautles, France, pp. 272 – 277.

NORTON, J.P. 1986: An Introduction to Identification. London: Academic Press, Harcourt Brace Javanovic Publishers.

OSBORN, G.H. 1961: Synthetic ion Exchangers. London: Chapman Hall.

OSMONICS INC., March 1992: Methods of Water Purification. National Development. http://www.osmonics.com.

OWENS, L.D. 1985: Practical Principals of Ion Exchange Water Treatment. Tell Oaks Publishing.

PALM III, W.J. 1983: Modeling, Analysis and Control of Dynamic Systems. John Wiley and Sons Inc.

References

PIERRE, A.D. 1969: Optimization Theory with Applications. New York: John Wiley and Sons, Inc.

PEEL, R.G. & BENEDEK, A. 1981: A Simplified Driving Force Model for Activated Carbon Adsorption (Quadratic Driving Force Model). Canadian Journal of Chemical Engineering, vol. 59, pp. 688 – 692.

PHILBRICK, F.A.; HOLMYARD, E.J. & PALMER, W.G. 1949: A Textbook of Theoretical and Inorganic Chemistry. London: J.M. Dent and Sons.

PLESSET, M.S.; HELFFERICH, F. & FRANKLIN, J.N. 1958: Ion Exchange Kinetics: A Nonlinear Diffusion Problem II. Particle Diffusion Controlled Exchange of Univalent and Bivalent ions. Chemical Physics, vol. 29, no. 5, pp. 1064 – 1069.

POPCHEV, I. & TSONEVA, R. 1993: Design of Optimal Sensitive Control of Interconnected Systems. In Proceedings of the 12th IFAC World Congress, vol. 6, pp. 330 – 306.

POPCHEV, T. & TSONEVA, R. 1990: Synthesis of Two Layer Control of Interconnected Systems with Time Delays in the Presence of Slowly Varying Disturbance. Proceedings for the, Tollin: XI World Congress IFAC, vol. 2, pp. 25 – 31.

PROJECT, 1982: Ion Exchange for the Desalination and Tertiary Treatment of Effluents, vol. 1 – vol. 5. University of Cape Town (UCT): Department of Chemical Engineering.

QUINN, J. 1990: Stabilization of Bilinear Systems by Quadratic Feedback Controls. Journal of Mathematical Analysis and Application, vol. 75, pp. 66 – 80.

RAMASAMY, S.; DESHPANDE, P.; TAMBE, S. & KULKARNI, B. 1995: Identification and Advances Controls of MSF Desalination Plants. Proceedings of IDA World Congress on Desalination and Water Sciences, Abu Dhabi, pp. 189 – 203.

RANDALL, E.W. 1984: A microprocessor-Based Data Monitoring and Control System for a Continuous Ion Exchange Plant. Desalination, vol. 49, pp. 169 – 184, Amsterdam: Elsevier Science Publishers B.V.

References

RAO, M.G. & DAVID, M. 1964: Single Particle Studies of Ion Exchange in Packed Beds: Cubic Ion-Sodium Ion System. AIChE, vol. 9, no. 3.

REMCO ENGINEERING. 1998: Environmental Water System and Controls De-ionization Systems. http://www.remco.com/

ROBERTS, P. 1999: Stability Properties of an Iterative Optimal Control Algorithm. Proceedings of 14th IFAC Congress, China, vol. F, pp. 269 – 274.

ROBERTS, P.D. & LIN, J. 1991: Potential for Hierarchical Optimizing Control in the Process Industry. Proceedings of the 1st European Control Conference, Grenoble, France, vol. 1, pp. 213 – 217.

ROSEN E.M. 1980: Steady State Process Simulation: A State of the Art Review, in Computer Applications to Chemical Engineering (R.G. SQUIRES and G.V REKLAITIS, Editions); ACS Symposium Series, vol. 124, American Chemical Society, Washington DC.

ROSENBROCK, H.H. 1974: Computer-Aided Control System Design. London: New York Academic Press.

ROSENBROCK, H.H. 1970: State Space and Multivariable Theory. London: Nelson.

ROSENBROCK, H.H. & STORY, C. 1966: Computational Techniques for Chemical Engineers, pp. 189 – 208. Pargamon Press.

ROUSSEAU, R.W. 1995: Handbook of Separation Process Technology. New York: John Wiley and Sons.

ROY, W. 1981: Advanced Process Control. New York: McGraw Hill.

RPI. 1995: Applications of Ion Exchange. http://www.rpi.edu/dept/chem-eng/

RPI. 1995: Applications of Ion Exchange. http://www.rpi.edu/dept/chem-eng/Biotech-Enviro/IONEX/biochem3.htm – /biochem4.html.

RUCHENSTEIN, E; VAIDYANATHAN, P. & YOUNGQUIST, G. 1971: Sorption of Solids with Bi-disperse Pore Structures. Chemical Engineering Science, vol. 26, pp. 1305 – 1318.

References

RYAN, E & BUCKINGHAN, J. 1983: On Asymptotically Stabilizing Feedback Control of Bilinear Systems. IEEE Transport and Automatic Control, vol. AC-28, pp. 863 – 864.

SCHARZENBACH, J. 1996: Essentials of Control. Eddison Wesley Longman.

SCHMUCKLER, G., NATIV, M. & GOLDSTSEIN, S. 1976: The Theory and Practice of Ion Exchange. Society of Chemical Industry, pp. 171. London.

SCHLOGE, R. & HELFFERICH, F. 1957: Comment on the Significance of Diffusion Potentials in Ion Exchange Kinetics. Chemical Physics, vol. 26, no. 1, pp. 5 – 7.

SERCHSEN, E.; MACHIETTO, S.; STUART, G. & SKOGESTAD, S. 1994: Optimal Control and On-Line Operation of Reactive Batch Distillation. Comut. Chemical Engineering, vol. 20, pp. 1492 – 1498.

SHACHAM, M. 1984: Decomposition of Systems of Nonlinear Algebraic Equations. AIChE, vol. 30, no. 1, pp. 92 – 98.

SHELLEY, S. December 1997: Ion Exchange Curb water and Chemical Use. Chemical Engineering, pp. 117 – 118.

SHINSKEY, F. 1977: Distillation Control. New York: McGraw Hill.

SIEMENS, PAZO. 1999: Liquid Analysis. Catalog PA 20, pp. 2/4 – 2/5.

SINNOT, R.K. 1997: Chemical Engineering. Volume 2: Chemical Engineering Design. Energy Balances, 2nd Edition. Oxford: Butterworth-Heinemann Ltd. (Pergamon Press).

SKOGESTAD, S.; HALVORSEN, I; LARSSON, T & GOVATSMARK, M. 1999: Plant-Wide Control: The Search for the self-optimizing Control Structure. Proceedings of the 14th IFAC World Congress, vol. N, China, pp. 325 – 330.

SLATER, M.J. 1992: Ion Exchange Advances. In Proceedings of IEX'92. Applied Science, Elsevier.

SLATER, M.J. 1991: Principles of Ion Exchange Technology. Butterworth – Heinemann, Ltd.

SLATER, M.J. February 1974: Continuous Ion Exchange in Fluidized Beds. The Canadian Journal of Chemical Engineering, vol. 52, no. 2, pp. 43 – 51.

References

SMITH, B.D. 1963: Design of Equilibrium Stage Processes. New York: McGraw Hill.

SMITH, G.D. 1965: Numerical Solution of Partial Differential Equation. New York: Oxford University Press.

SMITH, R.G. & DRANOFF, J.S. 1964: Film Diffusion Controlled Kinetics in Binary Exchange. Industrial Engineering Chemical Fundamentals, vol. 3, no. 3, pp. 195 – 201.

SOROUSH, M. 1995: Nonlinear Output Feedback Control of a Polymerization Reactor. In Proceedings of the American Control Conference, vol. 4, pp. 2672 – 2676.

STEPHANOPOULOS, G.1984: Chemical Process Control. New York: Prentice Hall, Englewood Cliffs.

STEPHANOPOULOS, G. 1982: Synthesis of Control Synthesis for Chemical Plants. Proceedings of the International Symposium on Process Systems Engineering. Kyoto.

STREAT, M. 1995: "The Waters were made Sweet." Advances in Ion Exchange Technology. Industrial Engineering and Chemistry Research, vol. 34, pp. 2841 – 2848.

STREAT, M. & CLOETE, F.L.D. 1995: Ion Echange. Handbook of Separation Process Technology. Editor, ROUSSEAU, R. New York:John Wiley and Sons.

STREAT, M. 1976: The Theory and Practice of Ion Exchange. An International Conference held at Churchill College. University of Cambridge: Society of Chemical Industry.

STROZ, M & GILLES, E. 1995: I/O Linearization of Chemical Processes within a plant-wide Quality Control framework. In Proceedings of American Control Conference, vol. 4, pp. 235 – 239.

SUGIE, T., SIMIZU, K. & IMURA J. 1993: H_∞ Control with exact Linearization and Its Application to Magnetic Levitation Systems. In Proceedings of 12th IFAC World Congress, vol. 4, pp. 363 – 366.

SUNG, E., DAC HAN, C. & RHEE, H. 1979: Optimal Design of Multistage Adsorption Bed System. AIChE, vol. 25, no. 1, pp. 87 – 100.

SYBRON CHEMICALS INC. 1992: Cation Exchange Countercurrent Regeneration.

References

SYBRON CHEMICALS INC. 1992: http://www.ionexchange.com/ionexchange/press.html. Sybron Chemical Inc

TAMURA, H. 1975. Decentralized Optimization for Distributed Lag Models of Discrete Systems. Automatica, vol. 11, pp. 593 – 602.

TEO, K.L.; GOH, C.J. & WONG, K.H. 1991: A Unified Computational Approach to Optimal Control Problems. London: Longman.

TETRA. 1991a: CCIX™ Principles of Operation. http://tetratec.com. CCIX™ Continuous Ion Exchange technology.

TETRA. 1991b: Applications and Industries. http://tetratec.com/Hpage-02.htm.

TETRA. 1991c: CCIX™ Systems Design. http://tetratec.com/Hpage-04.htm

THURSTON, C.W. June 1980: Computer-aided Design of Distillation Column Control, Part 1 and Part 2. Hydrocarbon Processing, pp. 65 – 77.

TOMICH, J.F. 1970: A New Simulation Method for Equilibrium Store Processes. AIChE, vol. 16, pp. 228.

TREYBAL, R.E. 1986: Mass-Transfer Operations. Third Edition, International Edition, McGraw-Hill Inc.

TSONEVA, R. & POPCHEV. R. 1995: Minimum Time Sensitive Control of Nonlinear Time Delay Systems. In Proceedings of International Conference on Systems, Analysis, Control and Design, Methodologies. Examples, vol. 2, pp. 189 – 197.

TURNER, J.C.R.; CHURCH, M.R.; JOHSON, A & SNOWDON, C. 1966: An Experimental Verification of the Nerst-Planck Model for Diffusion in an Ion Exchange Resin. Chemical Engineering Science, vol. 21, pp. 217 – 325.

ULLMAN'S ENCYCLOPEDIA. 1989: Encyclopedia of Industrial Chemistry. Volume A14: (Immmobilized Biocatalysts – Isoprene), 5th Edition. Edited by ELVERS, B.; HAWKINS, S; RAVENSCROFT, M & SCHULZ, G.

References

UNBAHANEN, H & RAO, G. 1997: Continuous Time Approach to System Identification: A Tutorial in Preprints of 11th IFAC Symposium on System Identification, vol. 3, pp. 1023 – 1049.

UNBAHANEN, H & RAO, G. 1990: Continuous Time Approach to System Identification – A Survey. Automatica, vol. 26, pp. 23 – 35.

van WINKLE, M. 1967: Distillation. New York: McGraw Hill.

VASSILIADIS, V.; PANTELIDES, C. & SARGENT, R. 1994: Solution of a Class of Multistage Dynamic Optimization Problems 1. Problems without Path Constraints. Industrial Engineering and Chemical Research, vol. 33, pp. 2111 – 2122.

VERMEULEN, T. 1955: Theory Irreversible and Constant-Pattern Diffusion. Industrial Engineering Chemical, vol. 45, pp. 1664 – 1670.

WATER QUALITY ASSOCIATION. 1995. Essentials of Ion Exchange. http://www.wqa.org/Technical/essentials-of-ionexchange.html.

WEATHERLY, L.R & TURNER, J. 1976: Ion Exchange Kinetics – Composition between a Macroporous and Gel Resin. Transcript from Industrial. Chemistry, vol. 54, pp. 89 – 94.

WESTPHAL, L.C. 1995: Sourcebook of Control Systems Engineering. Commissioned by Technical Communications. Chapman and Hall.

WIERZCHOWSKI, S. 1995: Application of Ion Exchange. RPI, http://www.rpi.edu/dept/chem-eng/Biotech-Enviro/IONEX/biochem3.htm.

XIN, J. & GUIHUA, L. 1996. The Simulation of Continuous Countercurrent Ion Exchange Process. Proceedings of the International Conference on Ion Exchange. London, pp. 633 – 636.

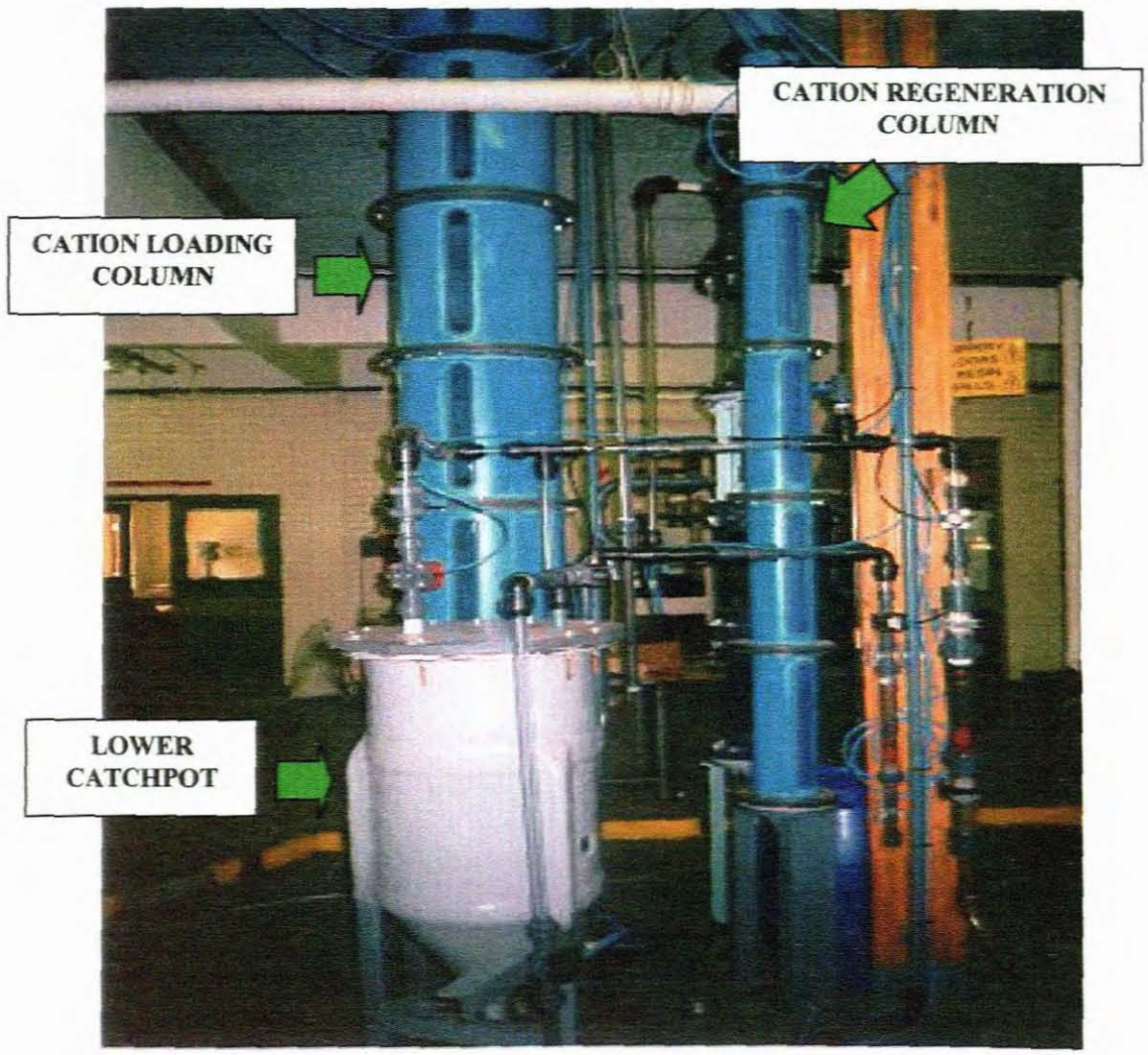
YU, W. & LIU, D. 1999: Time Variant Parameter estimation of a Nonlinear System using a Quasi-Newton Method. Proceedings of 14th world Congress of IFAC, China, vol. H, pp. 55 – 60.

APPENDICES

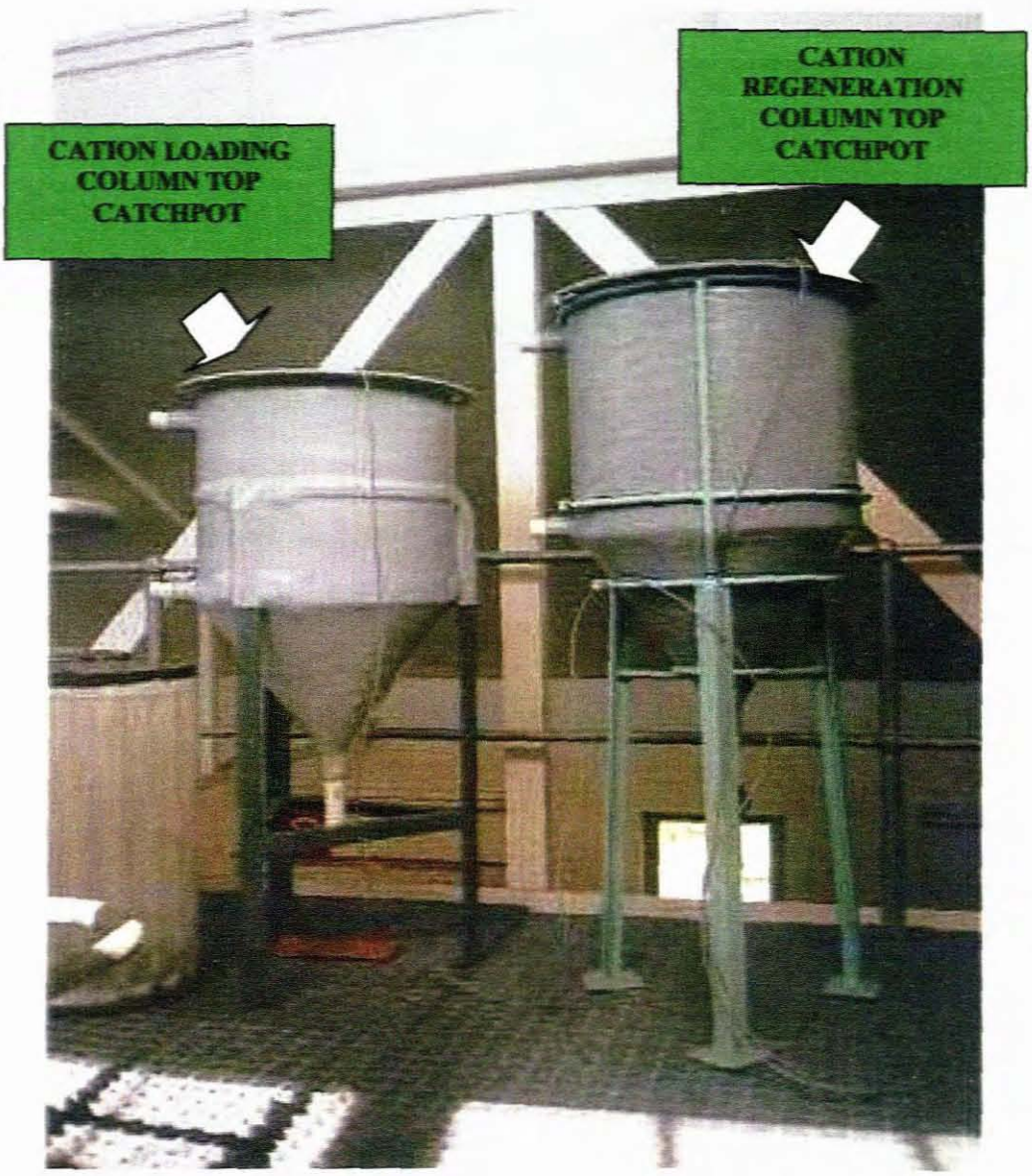
APPENDIX A

Pictures of the CCIX Pilot Plant built in the Department of Chemical Engineering – Peninsula Technikon (SA).

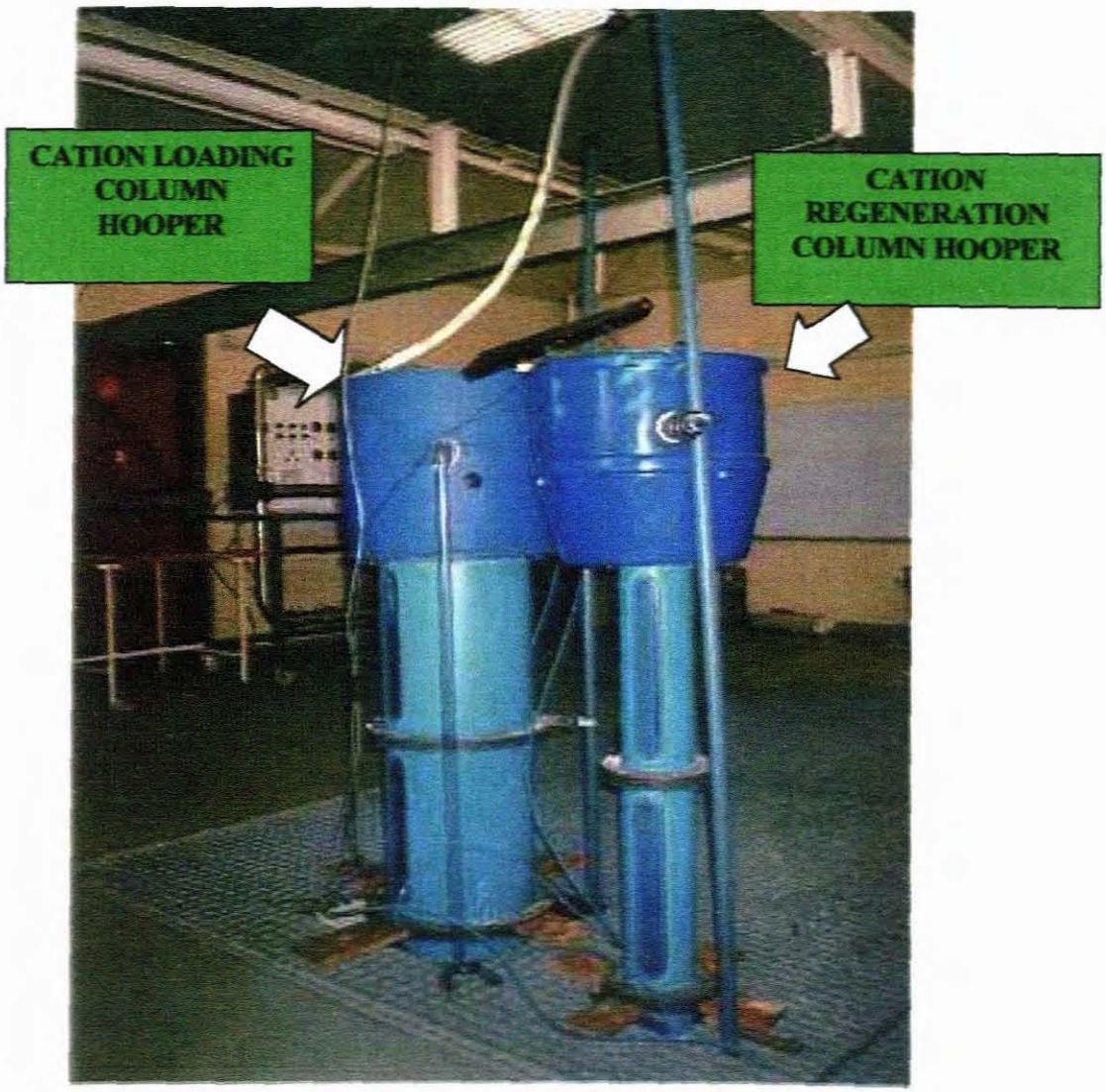
Picture 0. Cation Loading and Regeneration Columns for CCIX Plant as in Chem. Eng. Department of Peninsula Technikon.



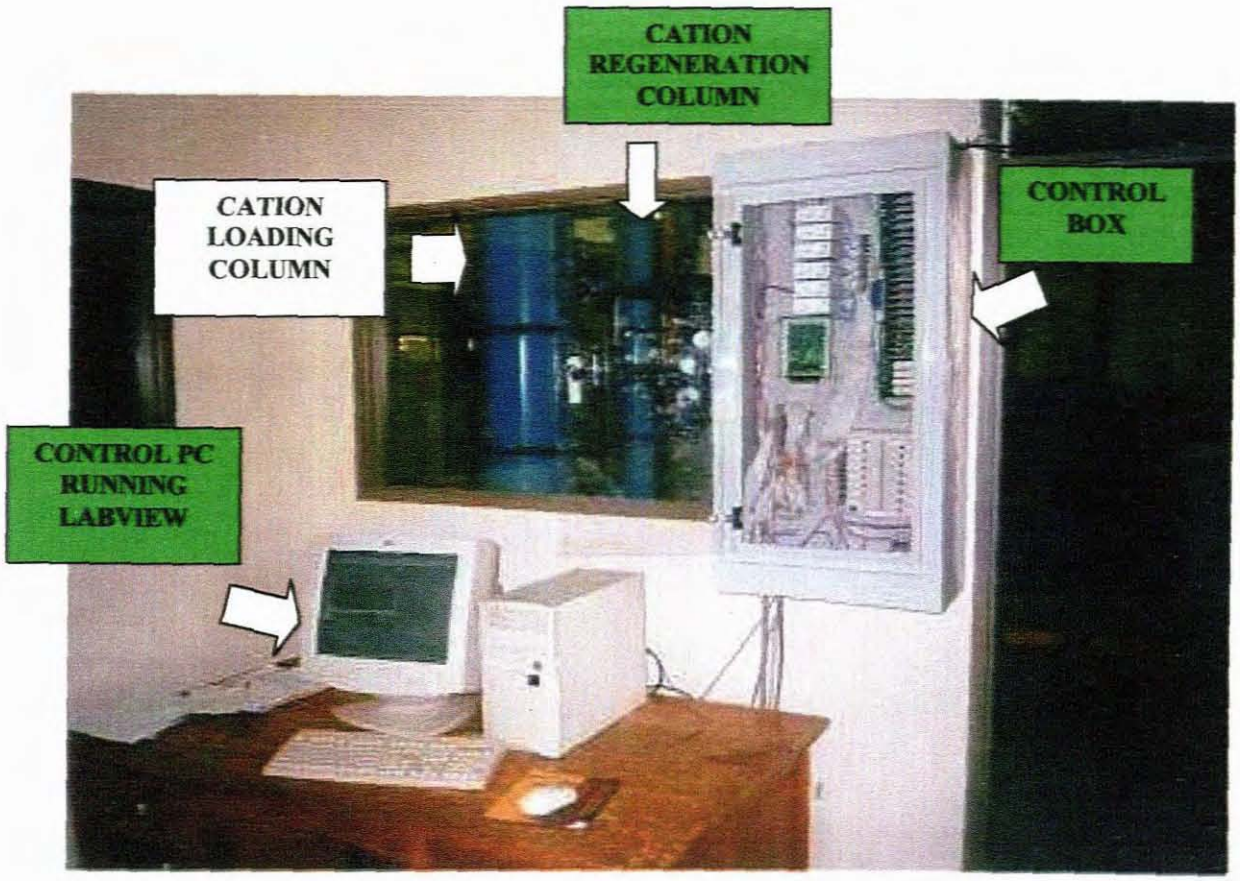
Picture 1. Cation Loading and Regeneration Columns for CCIX Plant as in Chem. Eng. Department of Peninsula Technikon.



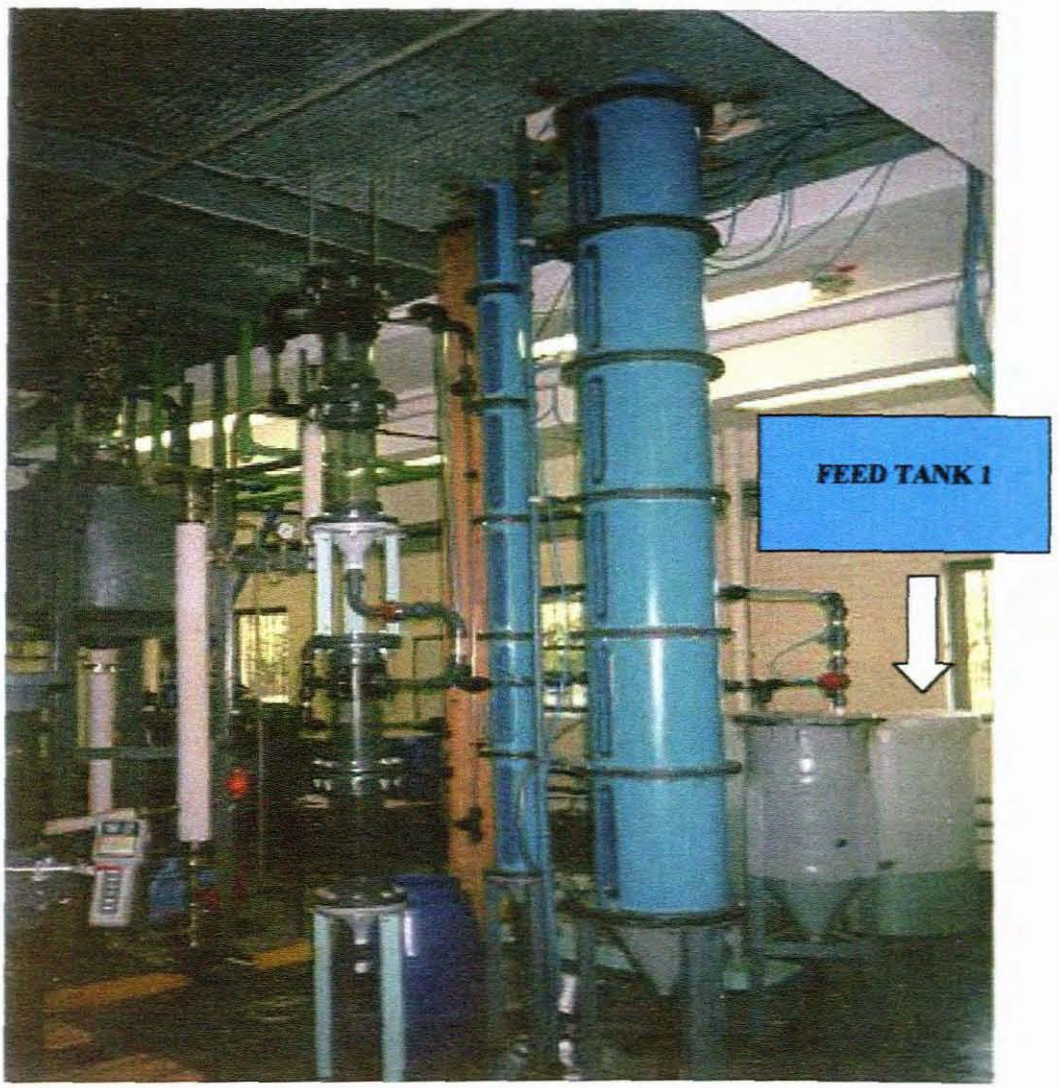
Picture 2. Cation Loading and Regeneration Top Catchpots for CCIX Plant as in Chem. Eng. Department of Peninsula Technikon.



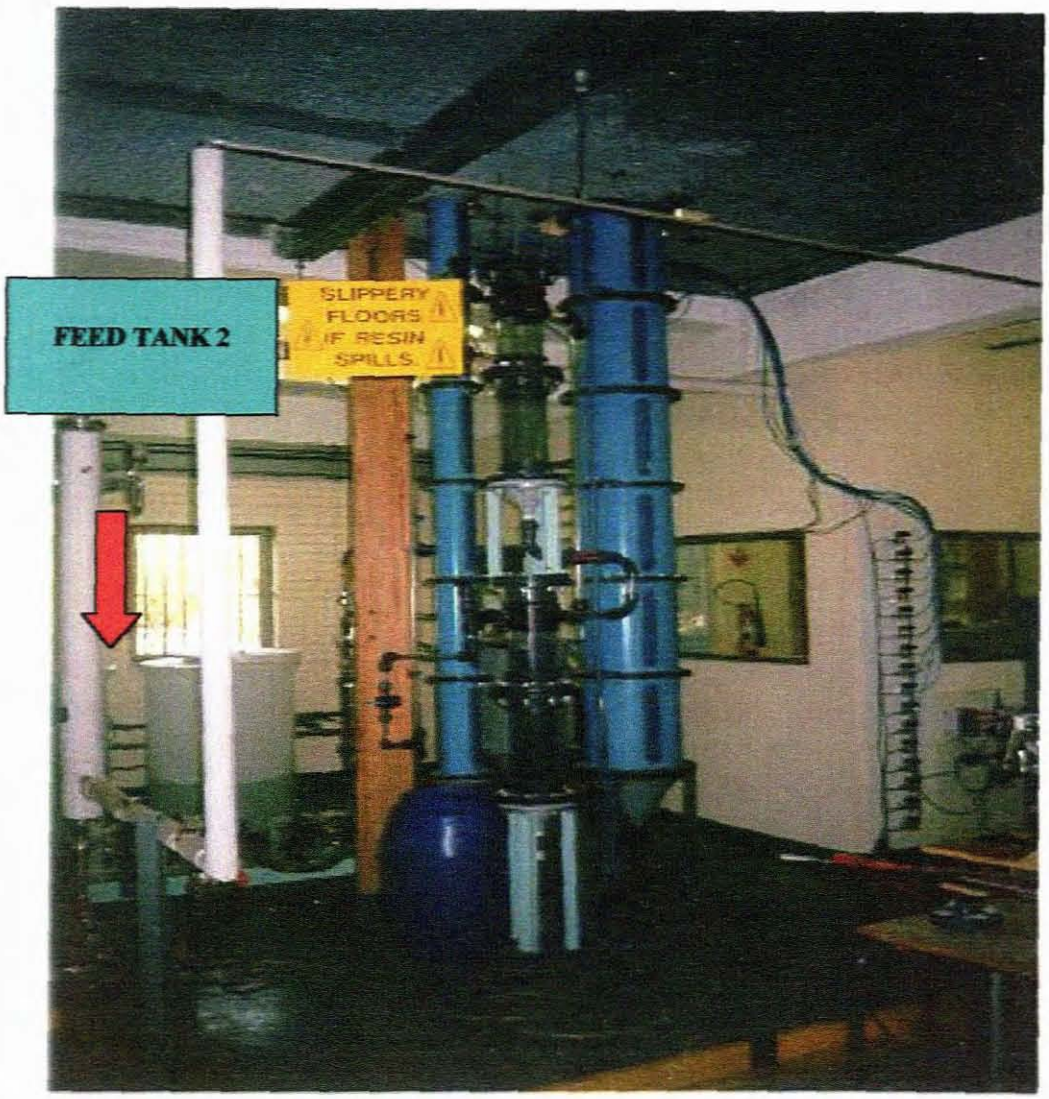
Picture 3. Cation Loading and Regeneration Hoopers for CCIX Plant as in Chem. Eng. Department of Peninsula Technikon.



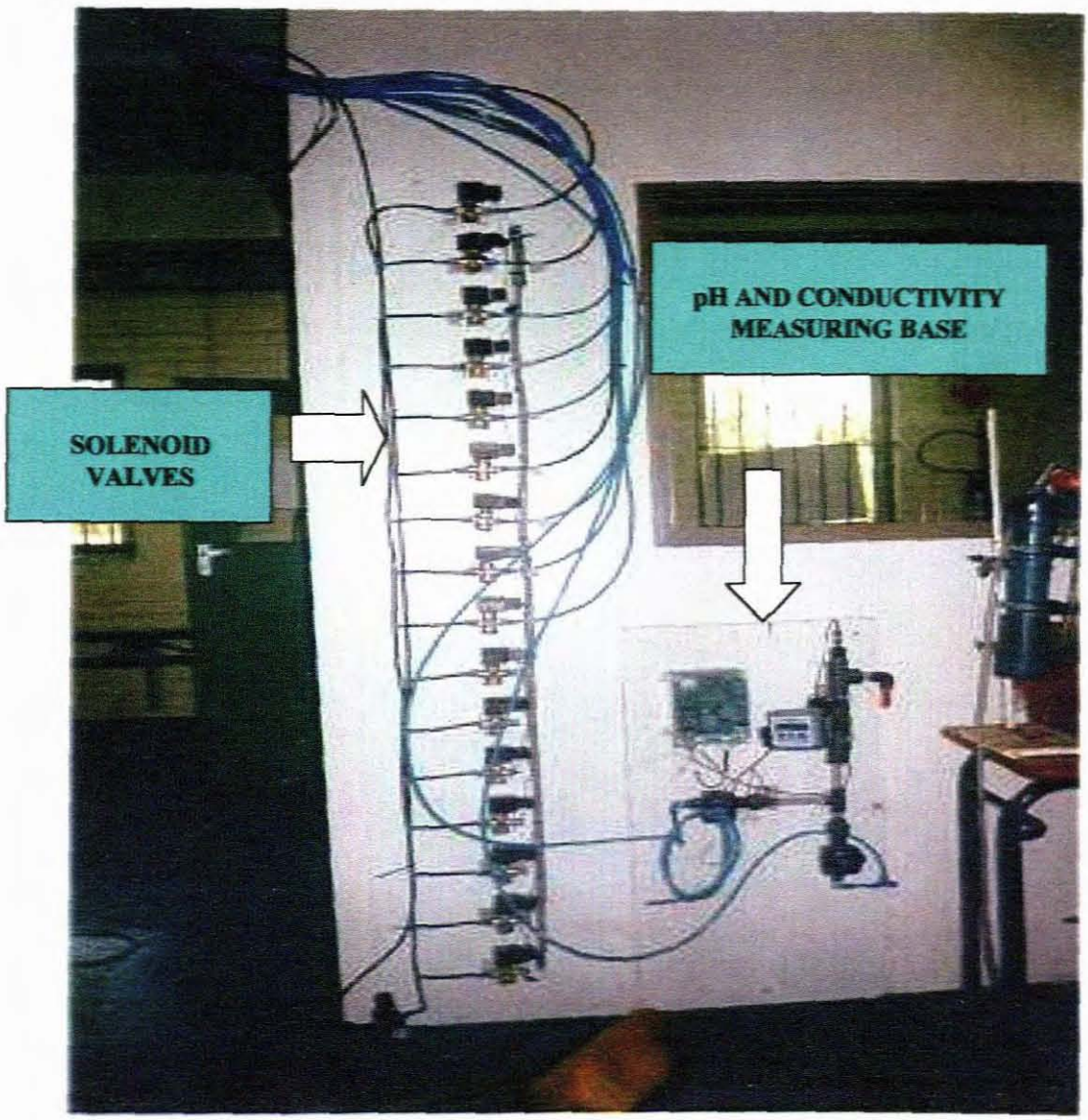
Picture 4. The Control Room for the CCIX Cation Loading and Regeneration Column Plant in Chem. Eng. Department of Peninsula Technikon.



Picture 5a. The Feed Tanks for the CCIX Cation Loading and Regeneration Column Plant in Chem. Eng. Department of Peninsula Technikon.



Picture 5b. Feed Tank 2 of Cation Loading and Regeneration Columns for CCIX Plant as in Chem. Eng. Department of Peninsula Technikon.



Picture 6. Cation Loading and Regeneration Control Solenoids for CCIX Plant as in Chem. Eng. Department of Peninsula Technikon.

APPENDIX B

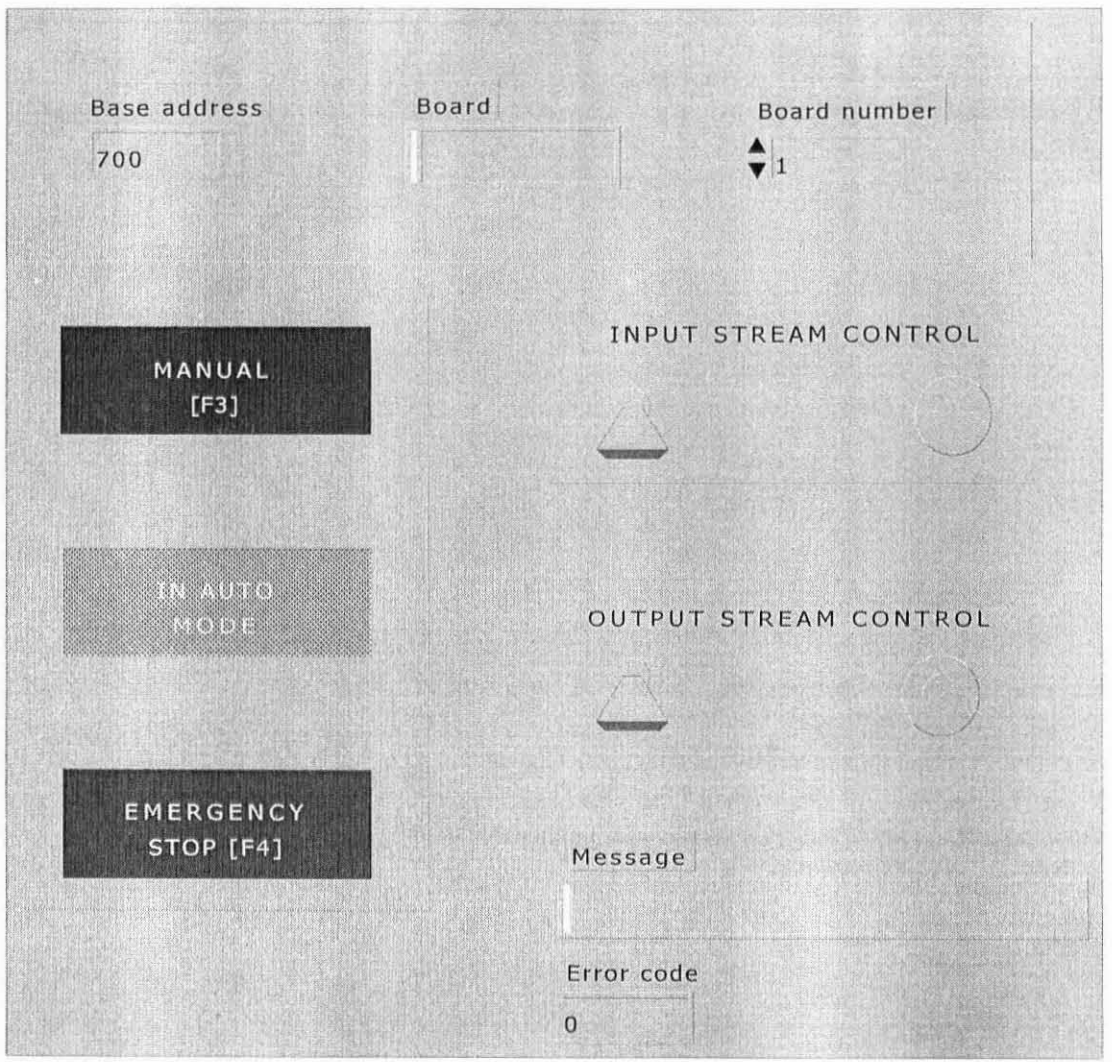
Optimal Control Programs

1. **pH and Conductivity Measurements Program (Analog Values) – LabVIEW Program**

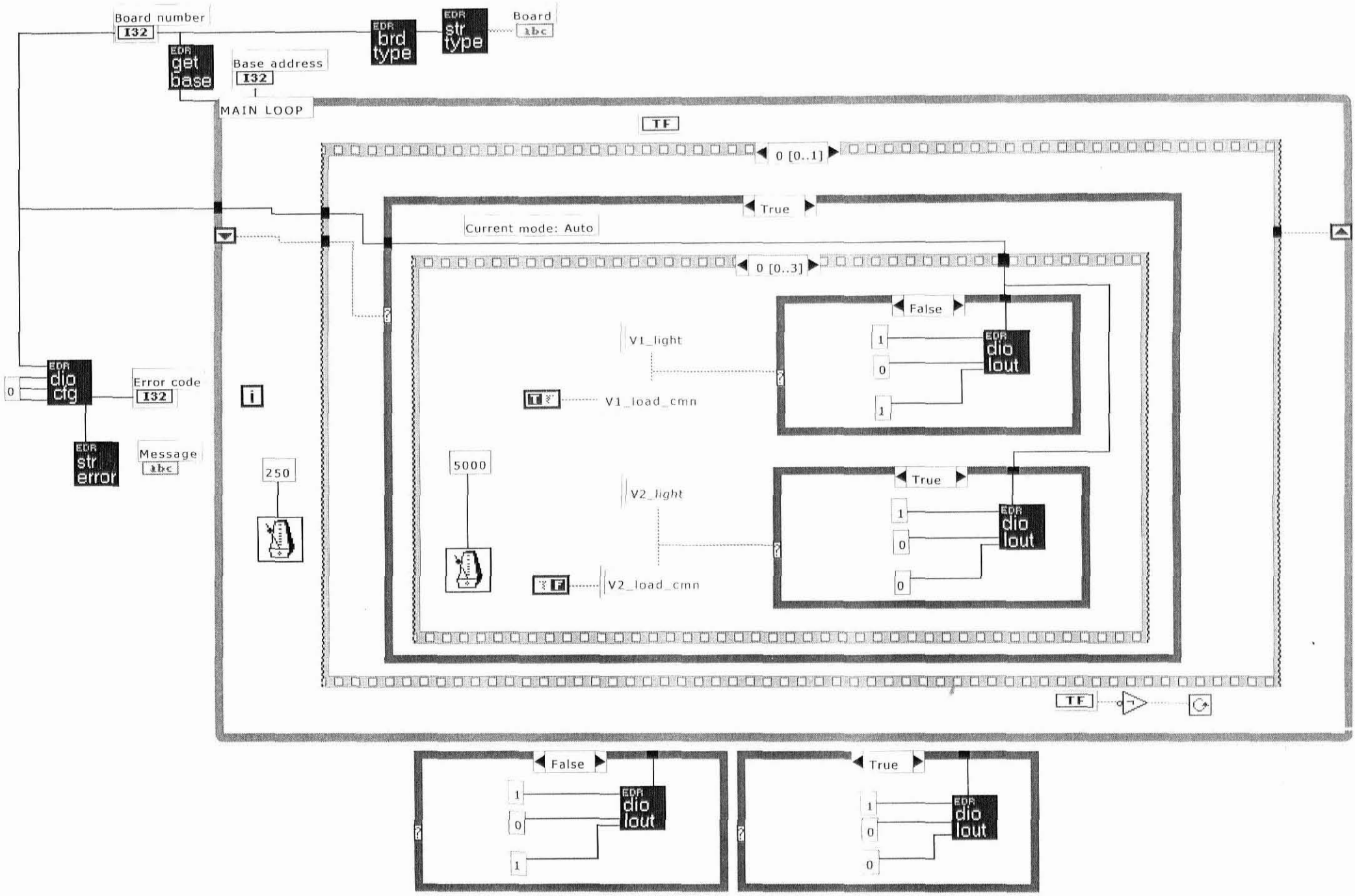
Connector Pane

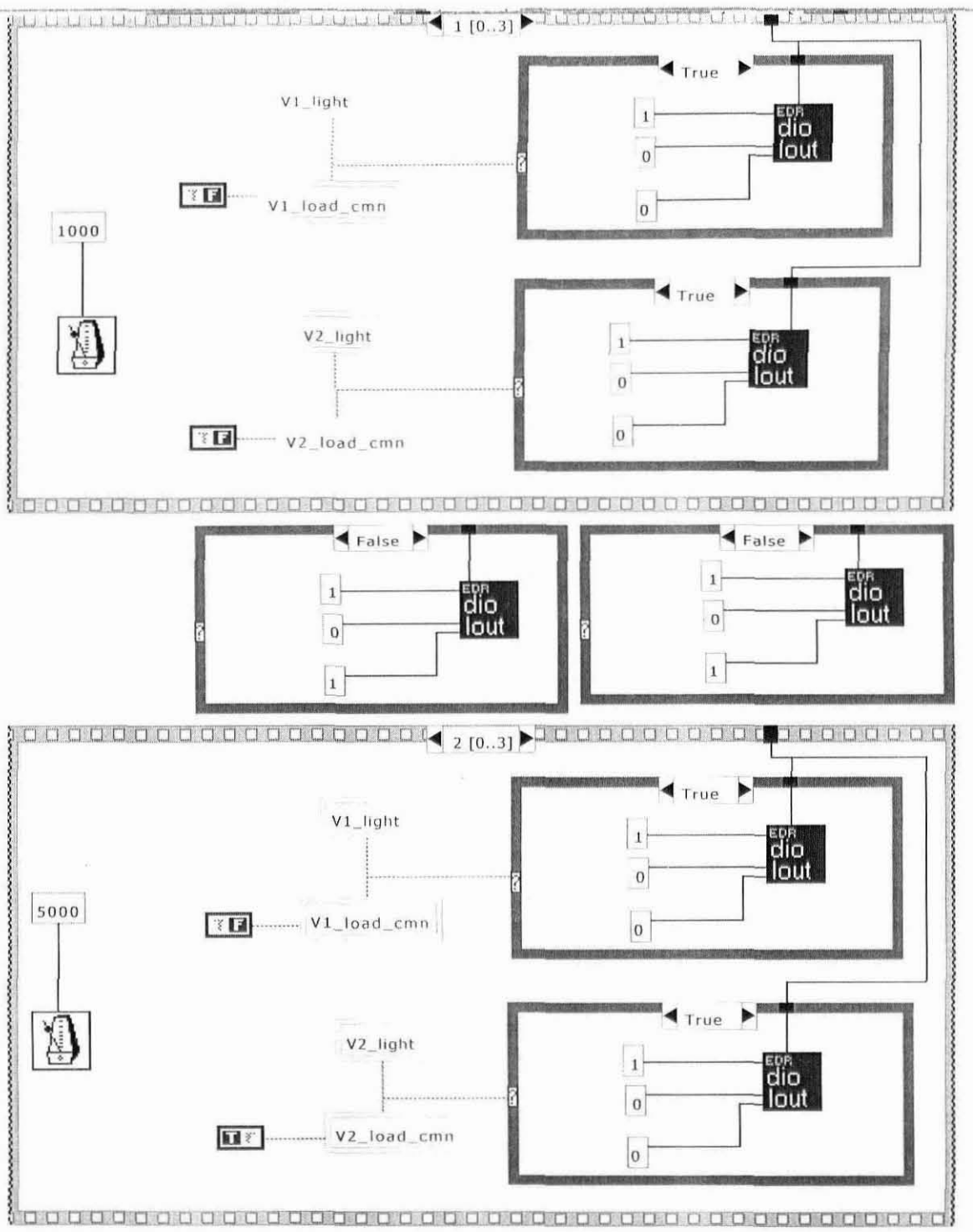
analog_control_001.vi

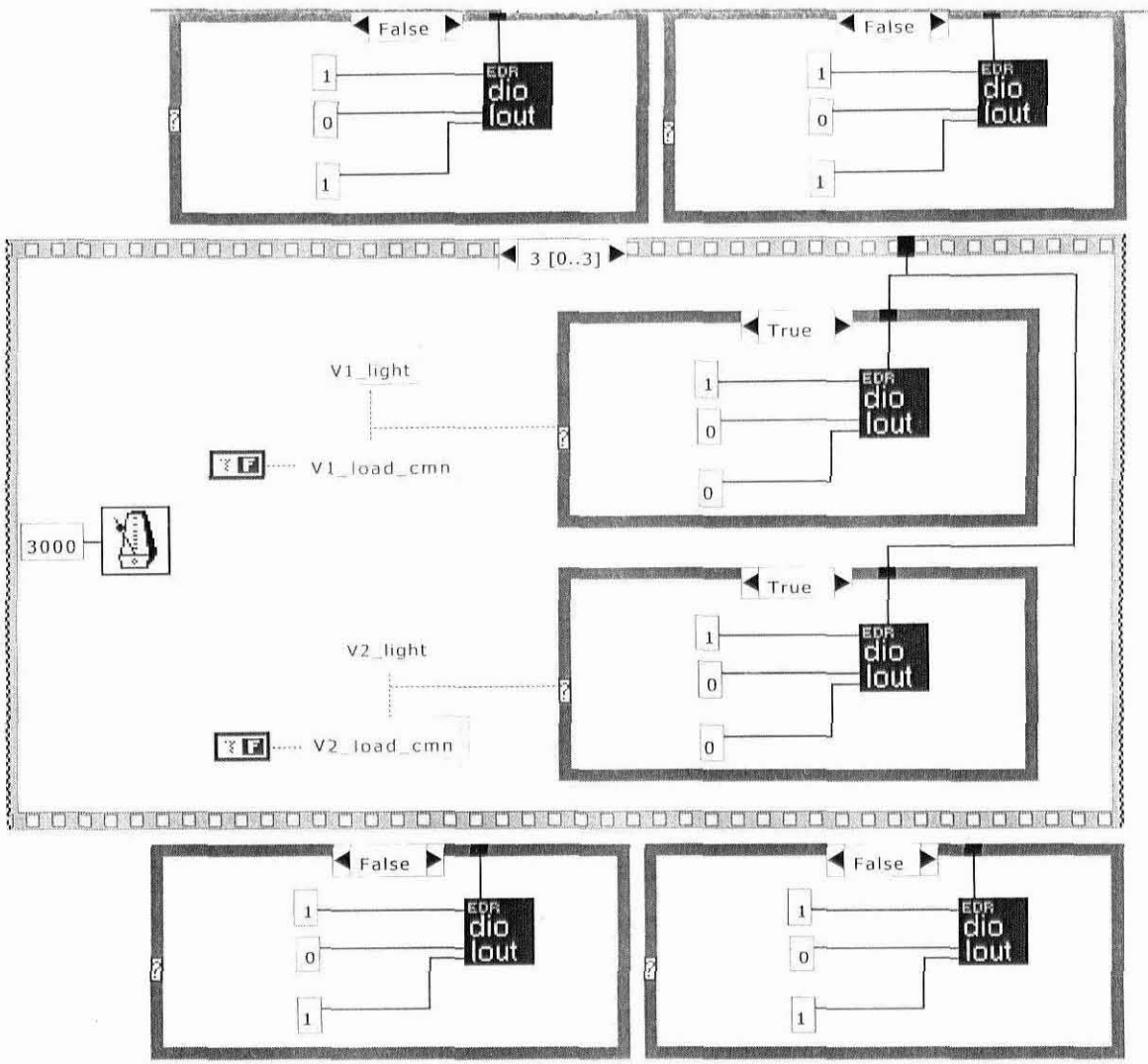
Front Panel

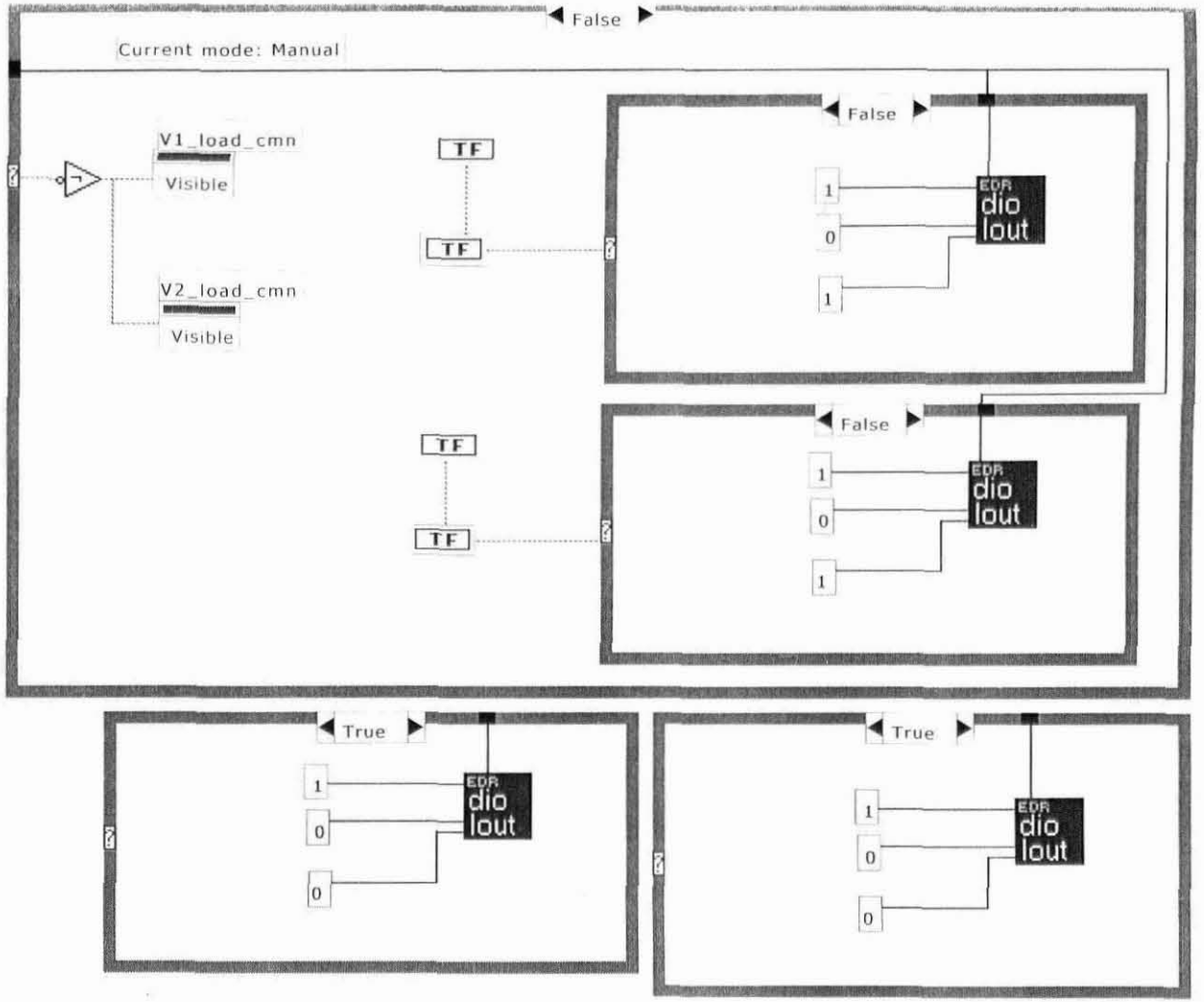


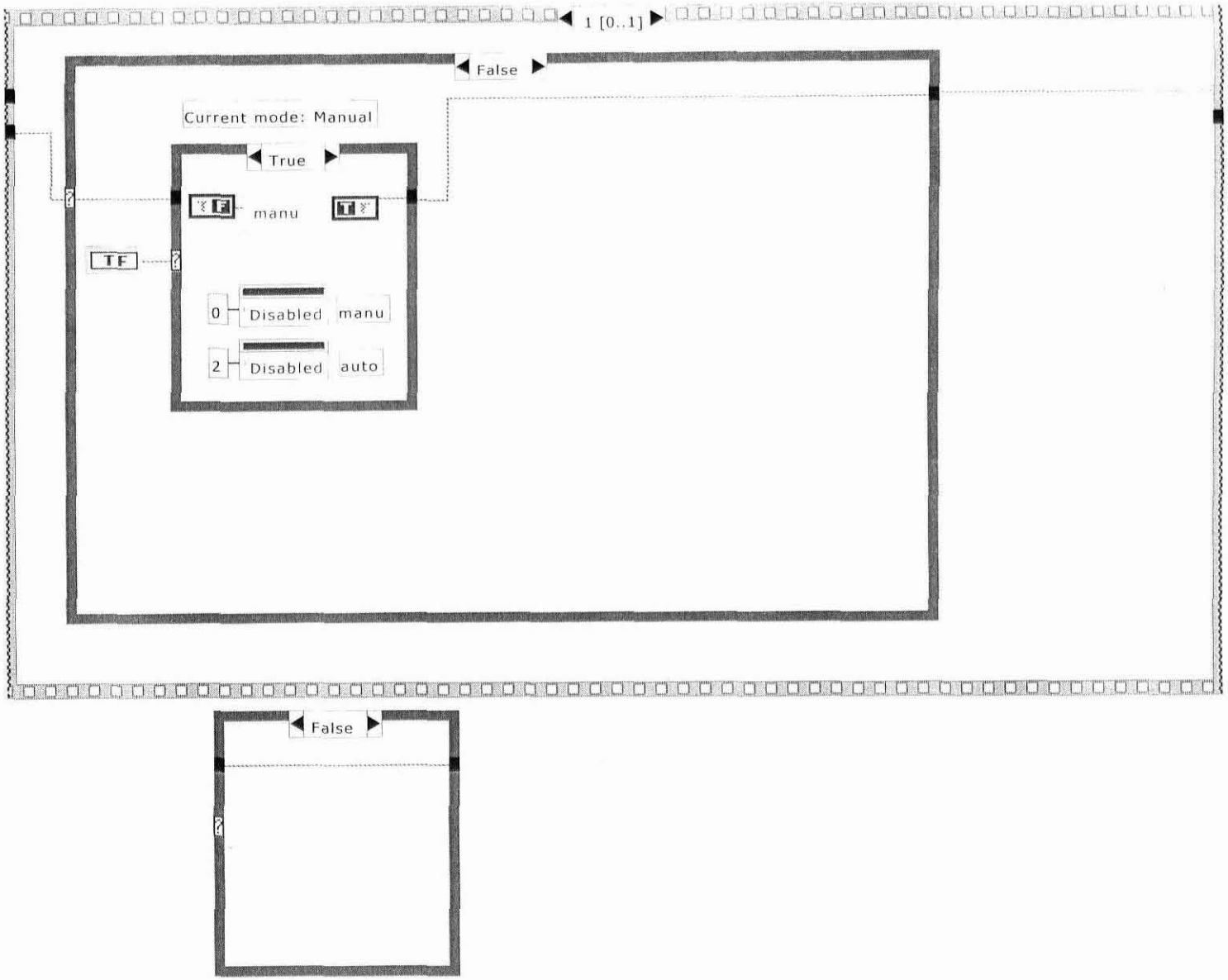
Block Diagram



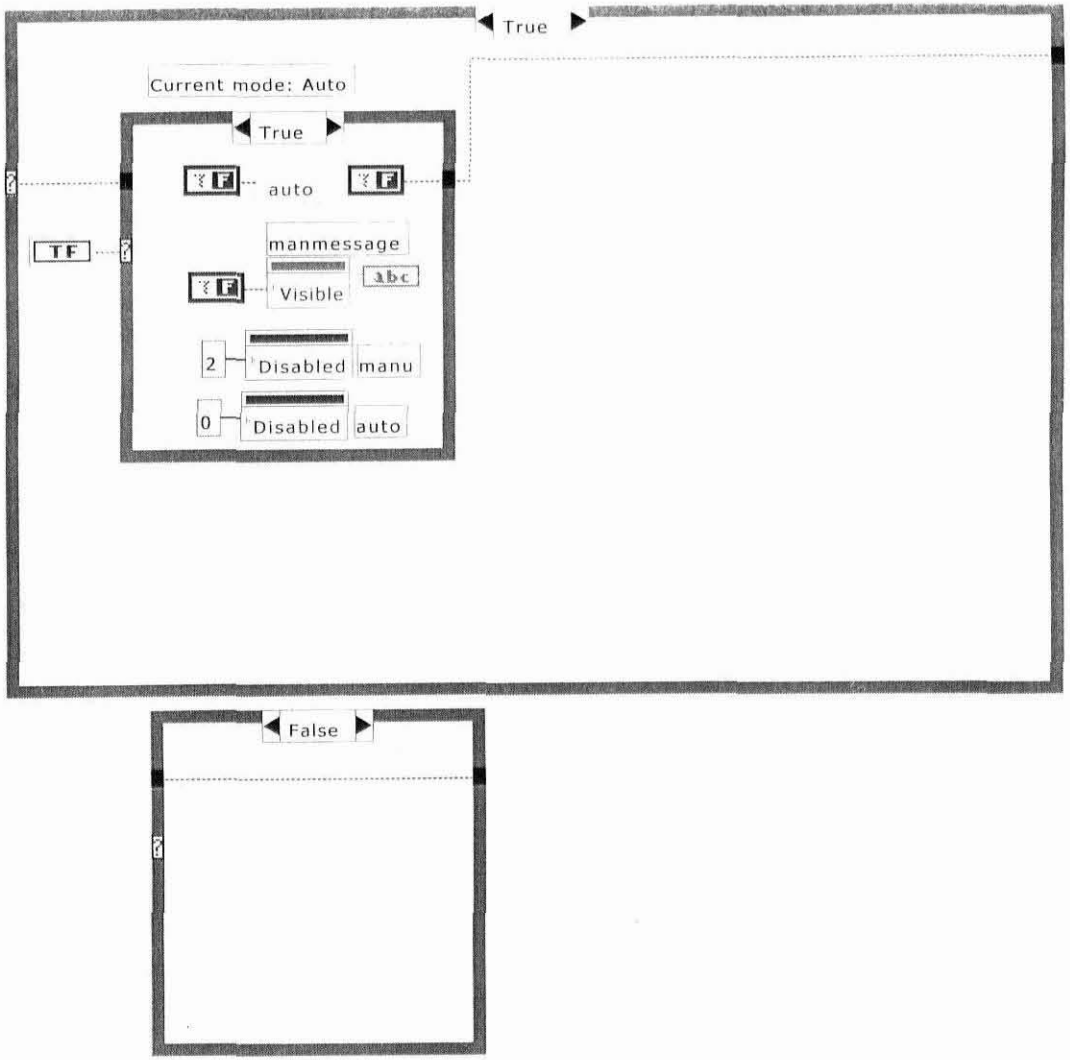

1 [0..3]
True
V1_light
1
0
0
EDR
dio
lout
F
V1_load_cmn
1000
True
V2_light
1
0
0
EDR
dio
lout
F
V2_load_cmn
False
1
0
1
EDR
dio
lout
False
1
0
1
EDR
dio
lout
2 [0..3]
True
V1_light
1
0
0
EDR
dio
lout
5000
F
V1_load_cmn
True
V2_light
1
0
0
EDR
dio
lout
T
V2_load_cmn


False
1
0
1
EDR
dio
lout
False
1
0
1
EDR
dio
lout
3 [0..3]
True
V1_light
1
0
0
EDR
dio
lout
V1_load_cmn
3000
True
V2_light
1
0
0
EDR
dio
lout
V2_load_cmn
False
1
0
1
EDR
dio
lout
False
1
0
1
EDR
dio
lout


False
Current mode: Manual
V1_load_cmn
Visible
V2_load_cmn
Visible
TF
TF
False
1
0
1
EDR
dio
lout
TF
TF
False
1
0
1
EDR
dio
lout
True
1
0
0
EDR
dio
lout
True
1
0
0
EDR
dio
lout


1 [0..1]
False
Current mode: Manual
True
F
manu
T
TF
0
Disabled
manu
2
Disabled
auto

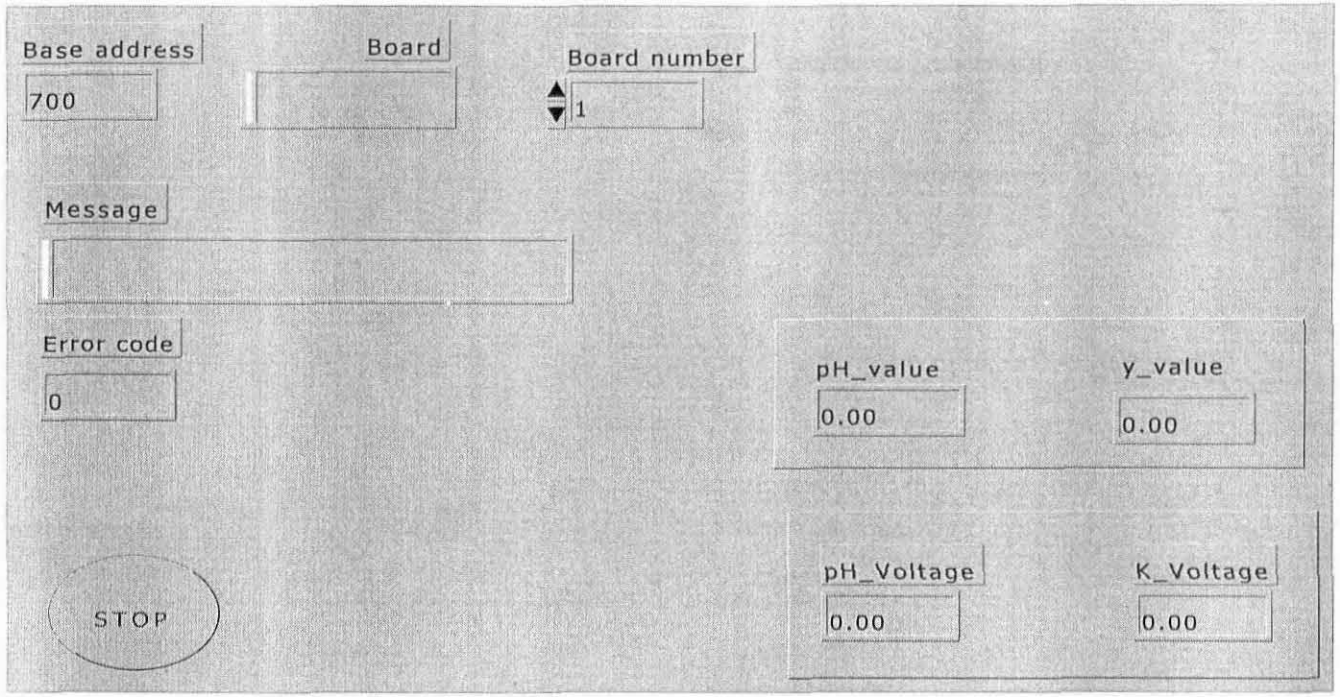
False


True
Current mode: Auto
True
auto
TF
manmessage
Visible
abc
2
Disabled
manu
0
Disabled
auto
False

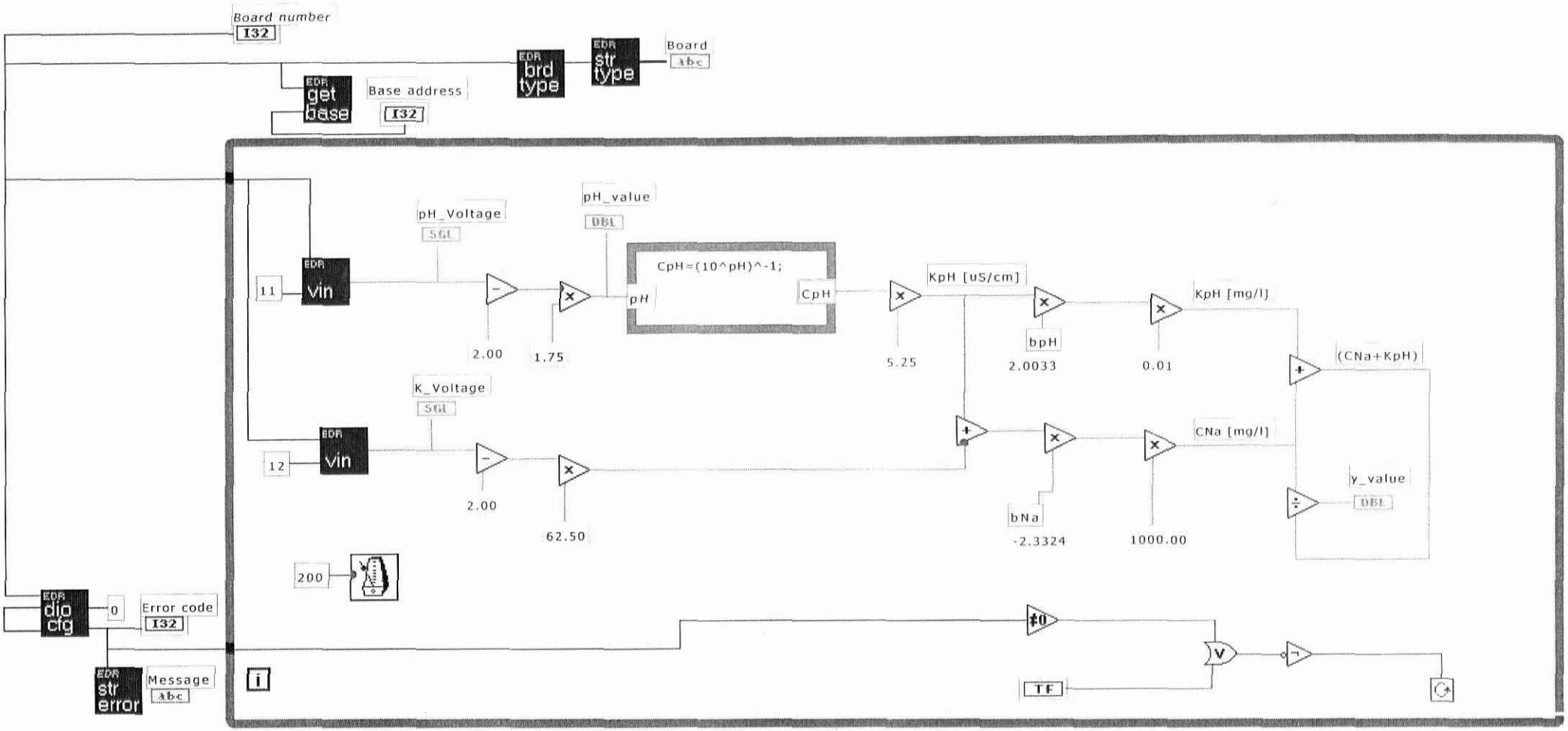
Connector Pane

y_value_calculation_001.vi

Front Panel



Block Diagram



APPENDIX B

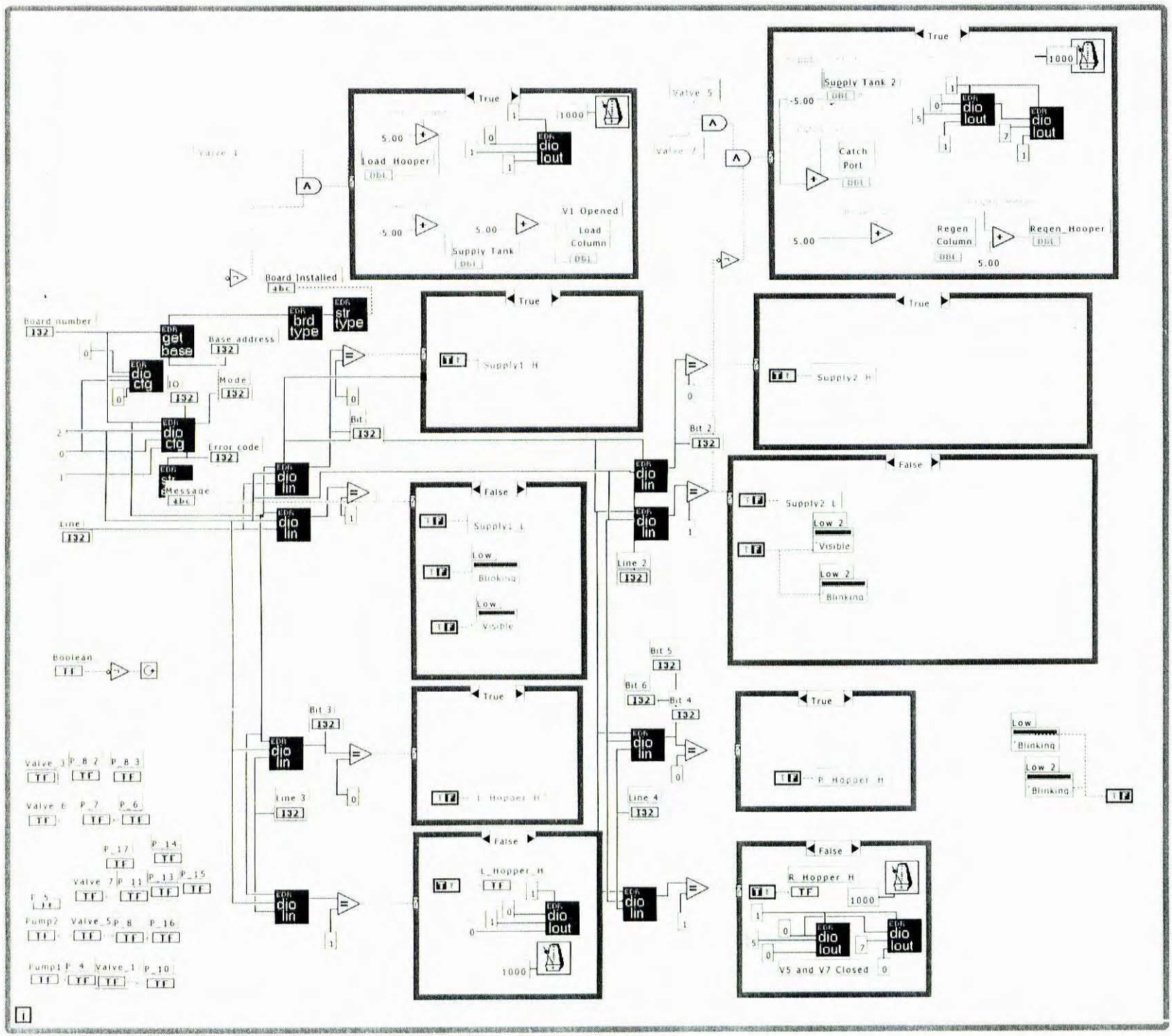
---

2. Digital Values Control Program – LabVIEW Program

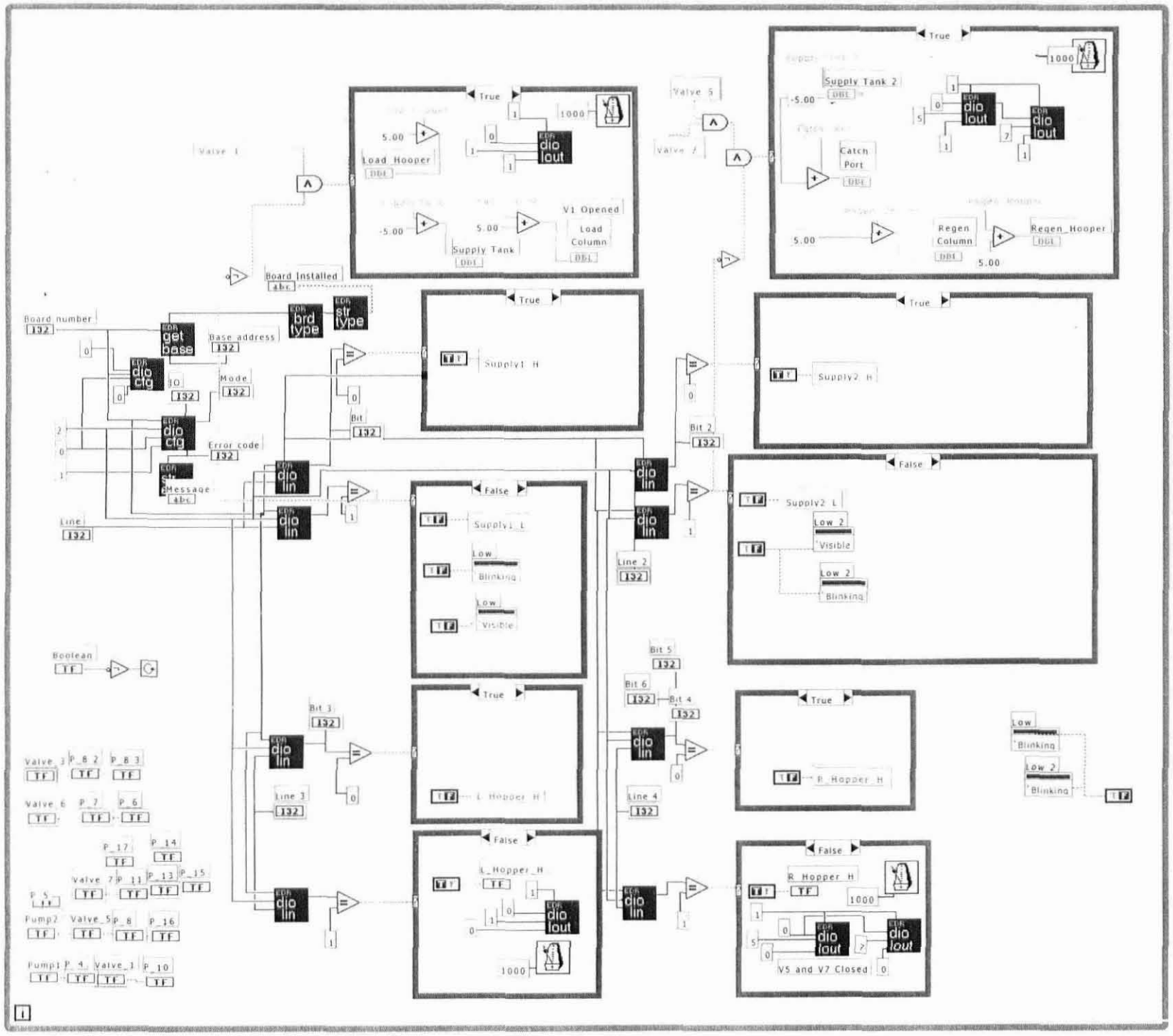
Connector Pane

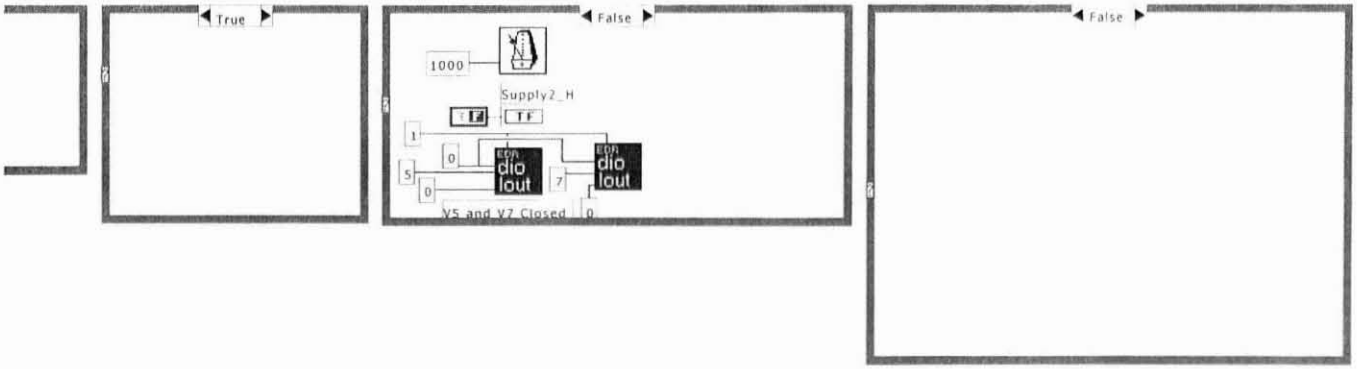
digital_values_control_001.vi

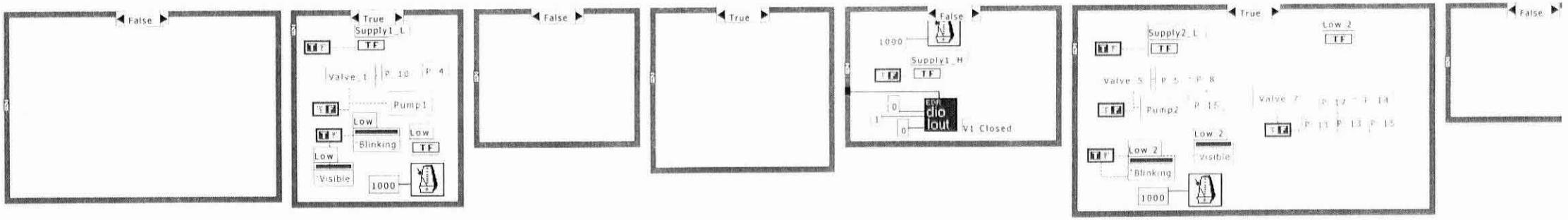
Block Diagram



Block Diagram




True
False
1000
Supply2_H
TF
1
0
5
0
7
0
EDR
dio
lout
EDR
dio
lout
V5 and V7 Closed
False


False
True
Supply1_L
TF
Valve_1
P_10
P_4
Pump1
Low
Blinking
Low
TF
Low
Visible
1000
False
True
False
1000
Supply1_H
TF
0
1
dio
out
0
V1 Closed
True
Supply2_L
TF
Low_2
TF
Valve_5
P_5
P_8
Pump2
P_15
Valve_7
P_12
P_14
P_11
P_13
P_15
Low_2
Visible
Low_2
Blinking
1000
False

APPENDIX B

---

3. Theoretical Model Parameter Calculations – Matlab Program

```
%Diameter of the load column inside
dl = 3800
Tl = 1.5*h
FR = h*dl/Tl
t1 = 0:0.1:15
tt = length(t1)
%
%input in the work space
FR = FR*ones(tt,1);
length(FR)
yf = yf*ones(tt,1);
length(yf)
A = [t1',FR]
B = [t1',yf]
```

```
.% Script file for Matlab used for simulation of the model in Simulink
% Parameter calculations
l1 = 27.635
l2 = 27.6350
l3 = 24.6731
l4 = 24.2401
l5 = 23.8220
l6 = 23.3390
l7 = 23.1823
l8 = 22.2846
%
m1 = 0.0158
m2 = 0.0166
m3 = 0.0185
m4 = 0.0188
m5 = 0.0191
m6 = 0.0194
m7 = 0.0195
m8 = 0.0201
%
k1 = 0.01
k2 = 0.01
k3 = 0.01
k4 = 0.01
k5 = 0.01
k6 = 0.01
k7 = 0.01
k8 = -0.15

m12 = 0.0193
m23 = 0.0207
m34 = 0.0214
m45 = 0.0223
m56 = 0.0230
m67 = 0.0237
m78 = 0.0243
%
% concentration of NaCl in the waste water is considered as an input disturbance.
yf = 1
%
%0.5 stage_volume
h = 28.43
H = 37.86
d = 0.67
K = 50
```

APPENDIX B

---

4. Simulation of the Model – HIQ Program

```
    zt = z';
    eps = zbar-zt;
    delta_J = eps*eps';
    dJdp  = (delta_J-J)/delta;
    //
    //calculation 0f the derivative of the criterion towards pi
    //
    //form the vector
    dJdpv[i]= dJdp;
    p[i]=p[i] - elta;
end for;
//
//caculation of the gradients for the fastest descent method on calculation of the weighted //sum
s1 = dJdpv*dJdpv';
s = sqrt(s1);
//
//calculation of the directions of the gradient
dp = -alfa*dJdpv/s;
//calculation of the improved estimate
p = p+dp;

//check for the optimal solution
//calculation of the norm of dp
//
dnorm = norm(dp,<L2>);
//
//check termination
if dnorm <= eps then
    [t,y] = ODEIVP(fcty,y0,0,K,1);
    exit while;
    else j = j+1;
end if;
   end while;
//
   g = createGraph(tout,y[*,1]);
        addplot(g,tout,y[*,2]);
        addplot(g,tout,y[*,3]);
        addplot(g,tout,y[*,4]);
//
 g1 = creategraph(tout,y[*,5]);
      addplot(g1,tout,y[*,6]);
      addplot(g1,tout,y[*,7]);
      addplot(g1,tout,y[*,8]);
```

```
k5 = b6 - b5;
k6 = b7 - b6;
k7 = b8 - b7;
k8 = -b8;
//
//forming vector of parameters P
p = {v:l1,m12,m1,k1,l2,m23,m2,k2,l3,m34,m3,k3,l4,m45,m4,k4,l5,m56,m5,k5};
p = {v:p,l6,m67,m6,k6,l7,m78,m7,k7,l8,m8,k8};

//solution of the model equation with initial value of parameters
j = 1;
while j <= M do
        [tout, y] = ODEIVP(fcty,y0,0,K,1);
//
// plotting of a graph
g = createGraph(tout,y[*,1]);
    addplot(g,tout,y[*,2]);
    addplot(g,tout,y[*,3]);
    addplot(g,tout,y[*,4]);

 g1 = creategraph(tout,y[*,5]);
        addplot(g1,tout,y[*,6]);
        addplot(g1,tout,y[*,7]);
        addplot(g1,tout,y[*,8]);
//
for k=1 to K+1 do
     z = y[t,8];
end for;
//
zt = z';
eps = zbar - zt;
J = eps*eps';
//
for i = 1 to m do
     p[i] = p[i]+delta;
     [t,y] = ODEIVP(fcty,y0,0,K,1);
     for k = 1 to K+1 do
          z = y[t,8];
     end for;
//
```

```
M = 500;
delta = 0.01;
epsilon = 0.001;
alfa = 0.05;
m = 31;
K = 50;
N = 8;
y0={v:1.1,1.1,1.1,1.1,1.1,1.1,1.1,1.1};
//
H=37.86;
h=28.43;
T = 1.5;
d= 2/3;
FL=2000;
FR =(h*d)/T;
yf =1;
//
//Introduction of the vector of the measured data for the time points
for k=1 to K+1 do
    zbar =1.5*exp(-0.1*k);
end for;
l1=FL/(H+a1*h);
l2=FL/(H+a2*h);
l3=FL/(H+a3*h);
l4=FL/(H+a4*h);
l5=FL/(H+a5*h);
l6=FL/(H+a6*h);
l7=FL/(H+a7*h);
l8=FL/(H+a8*h);
//
m12 = a2/(H + a1*h);
m23 = a3/(H + a2*h);
m34 = a4/(H + a3*h);
m45 = a5/(H + a4*h);
m56 = a6/(H + a5*h);
m67 = a7/(H + a6*h);
m78 = a8/(H + a7*h);
//
m1 = a1/(H + a1*h);
m2 = a2/(H + a2*h);
m3 = a3/(H + a3*h);
m4 = a4/(H + a4*h);
m5 = a5/(H + a5*h);
m6 = a6/(H + a6*h);
m7 = a7/(H + a7*h);
m8 = a8/(H + a8*h);
//
k1 = b2 - b1;
k2 = b3 - b2;
k3 = b4- b3;
k4 = b5 - b4;
```

```
// HiQ PROGRAM
//
//model
function fcty(t,y)
project variable_p, FR, yf;
//
//t-real scalar for the time
//y-real vector
ydot[1]= p[1]*yf -p[1]*y[1]+p[2]*FR*y[2]-p[3]*FR*y[1]+ p[4]*FR;
ydot[2]= p[5]*y[1]-p[5]*y[2]+p[6]*FR*y[3]-p[7]*FR*y[2]+p[8]*FR;
ydot[3]= p[9]*y[2]-p[9]*y[3]+p[10]*FR*y[4]- p[11]*FR*y[3]+p[12]*FR;
ydot[4]= p[13]*y[3]-p[13]*y[4]+p[14]*FR*y[5]-p[15]*FR*y[4]+p[16]*FR;
ydot[5]= p[17]*y[4]-p[17]*y[5]+p[18]*FR*y[6]-p[19]*FR*y[5]+p[20]*FR;
ydot[6]= p[21]*y[5]-p[21]*y[6]+p[22]*FR*y[7]-p[23]*FR*y[6]+p[24]*FR;
ydot[7]= p[25]*y[6]-p[25]*y[7]+p[26]*FR*y[8]-p[27]*FR*y[7]+p[28]*FR;
ydot[8]= p[29]*y[7]-p[29]*y[8]-p[30]*FR*y[8]+p[31]*FR;
return ydot;
//
//ydot real vector of the derivatives;
end function;
//
//THE BEGINNING OF THE MAIN PROGRAM
project variable_p,FR,yf;
//
a1=1.2;
a2=1.4;
a3=1.6;
a4=1.8;
a5=2.0;
a6=2.2;
a7=2.4;
a8=2.6;
//
b1 = 0.08;
b2 = 0.09;
b3 = 0.10;
b4 = 0.11;
b5 = 0.12;
b6 = 0.13;
b7 = 0.14;
b8 = 0.15;
//
```

APPENDIX B

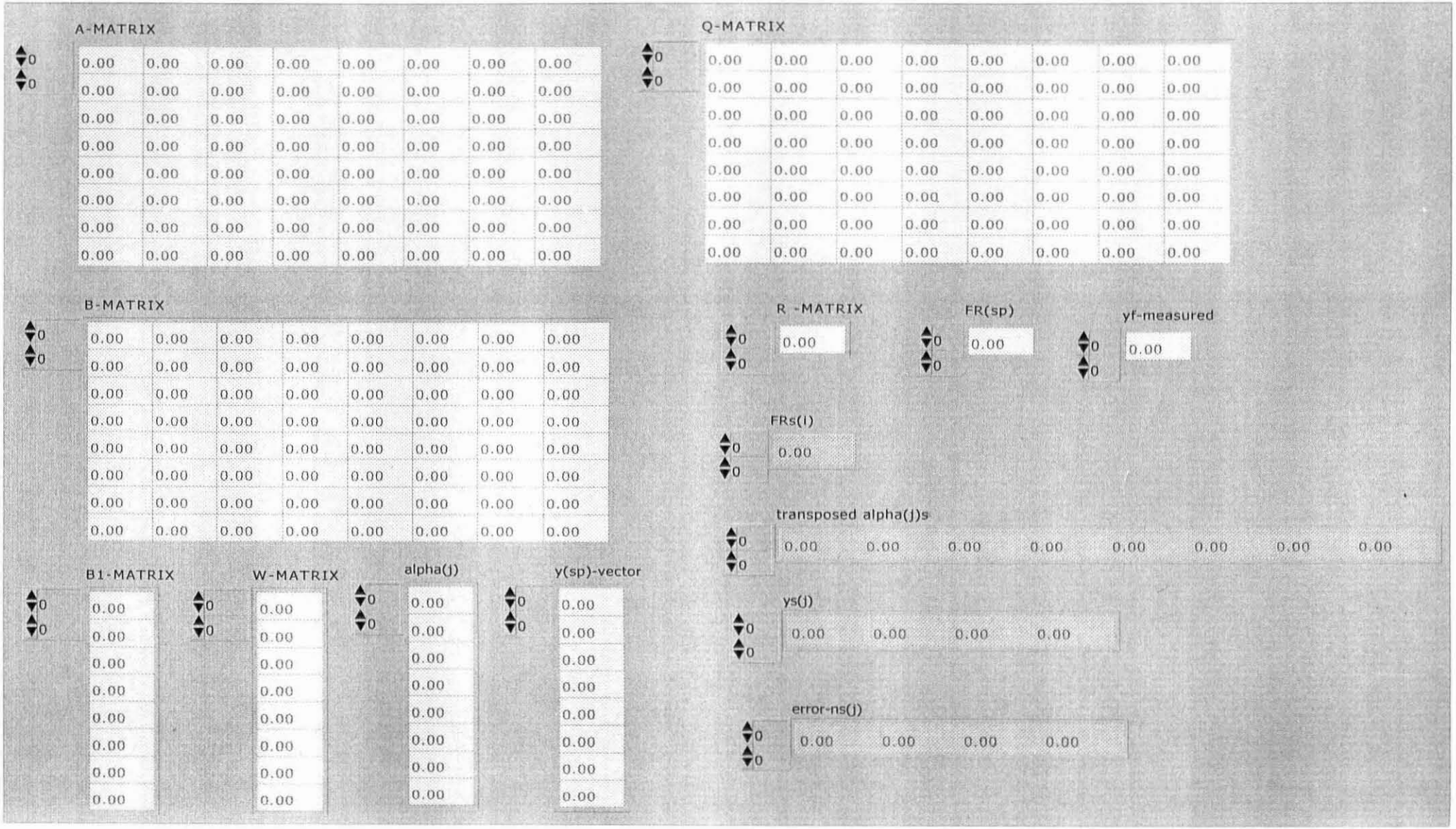
---

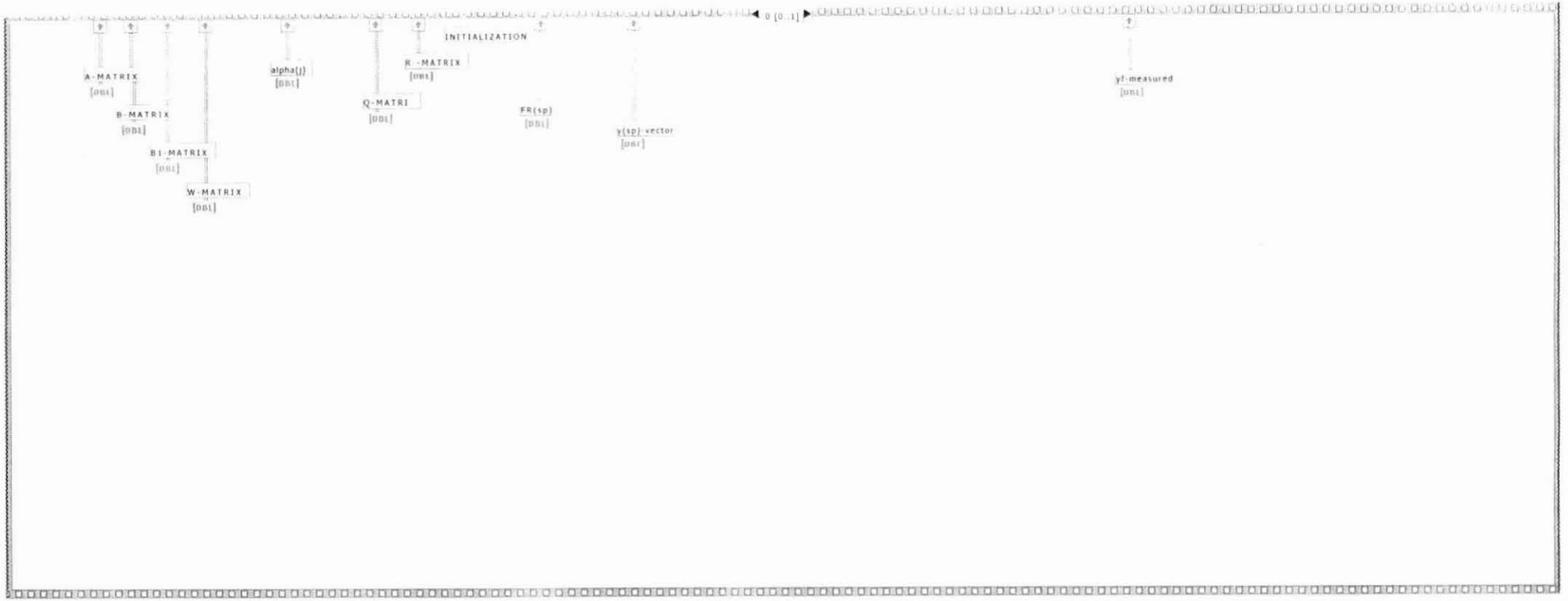
5. Steady State Model Program – LabVIEW Program and (Matlab Trial Program)

Connector Pane

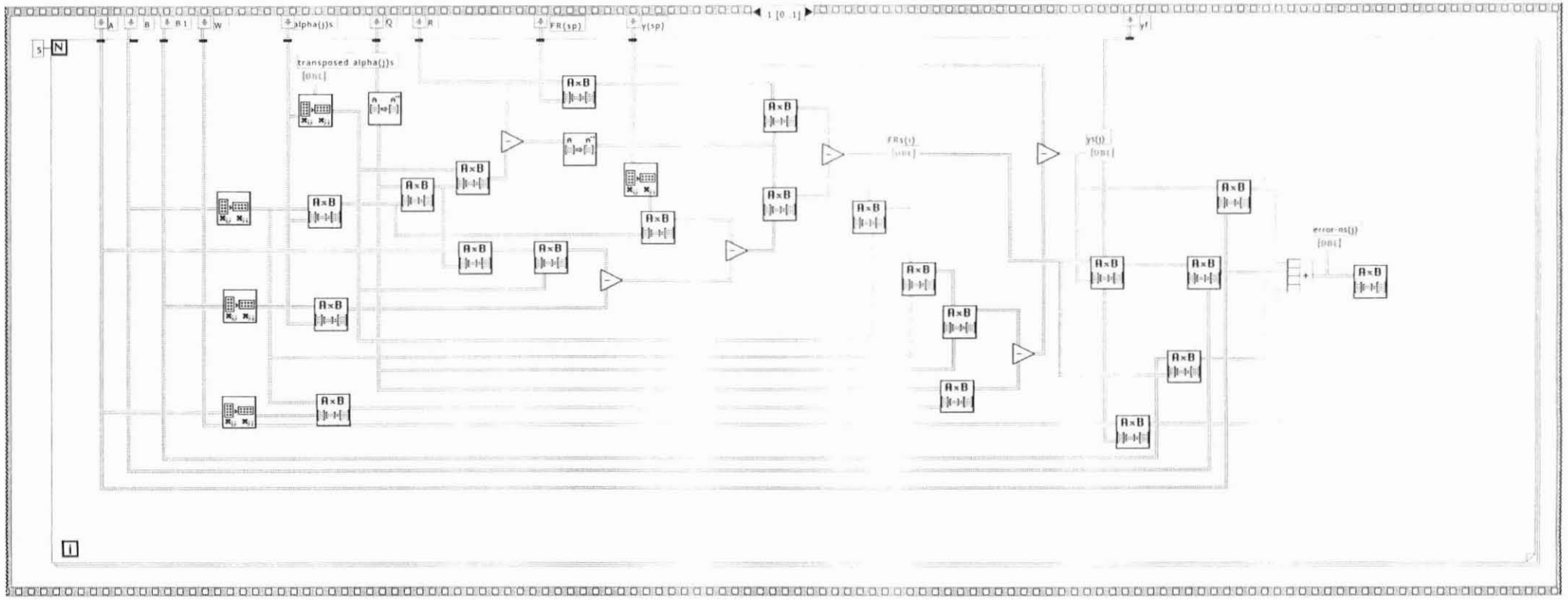
steady_state_model.vi

Front Panel




0 [0..1]
INITIALIZATION
A-MATRIX
[DBL]
B-MATRIX
[DBL]
B1-MATRIX
[DBL]
W-MATRIX
[DBL]
alpha(j)
[DBL]
Q-MATRI
[DBL]
R-MATRIX
[DBL]
FR(sp)
[DBL]
y(sp) vector
[DBL]
y!-measured
[DBL]

Block Diagram



```
%
%
%MATLAB TRIAL PROGRAM FOR STEADY STATE MODEL
%
%
%Equilibrium equation of resin/solid phase relationship.
a = [1.1;1.2;1.5;1.55;1.61;1.66;1.68;1.8];
b = [0.08;0.09;0.1;0.11;0.12;0.13;0.14;0.15];
%
%Flow-rate, Liquid and Resin holdups.
FL = 2000;
H = 28.96;
h = 37.62;
%
%Model matrices entries.
for i = 1:1:8
    l(i) = FL/(H+a(i)*h);
    m(i) = a(i)/(H+a(i)*h);
end
for i = 1:1:7
    k(i) = b(i+1)-b(i);
    mj(i) = a(i+1)/(H+a(i)*h);
end
k(8) = -b(8);
%
%A MATRIX
A(1,1) = -l(1);
A(2,1) = l(2);
A(2,2) = -l(2);
A(3,2) = l(3);
A(3,3) = -l(3);
A(4,3) = l(4);
A(4,4) = -l(4);
A(5,4) = l(5);
A(5,5) = -l(5);
A(6,5) = l(6);
A(6,6) = -l(6);
A(7,6) = l(7);
A(7,7) = -l(7);
A(8,7) = l(8);
A(8,8) = -l(8);
```

```
%
%B MATRIX
B(1,1) = -m(1);
B(1,2) = mj(1);
B(2,2) = -m(2);
B(2,3) = mj(2);
B(3,3) = -m(3);
B(3,4) = mj(3);
B(4,4) = -m(4);
B(4,5) = mj(4);
B(5,5) = -m(5);
B(5,6) = mj(5);
B(6,6) = -m(6);
B(6,7) = mj(6);
B(7,7) = -m(7);
B(7,8) = mj(7);
B(8,8) = -m(8);
%
%B1 MATRIX
B1 = k';
%
%W MATRIX
W = [l(1);0;0;0;0;0;0;0];
%
%C MATRIX
C=[0;0;0;0;0;0;0;1];
%
%Disturbance,Coordnating variable,Control and State
FRsp = [2.5];
ysp = [0.15;0.1;0.85;0.8;0.7;0.05;0.02;0.01];
lamda = [0.1;0.2;0.05;0.3;0.4;0.75;0.6;0.22];
yf = 1.12;
%
%Weighting matrices Q and R.
Q' = [100;100;100;100;100;100;100;100];
Q = diag(Q');
R = [0.12];
%
%Stopping procedure values
alpha = 0.01;
e_sml = 0.001;
M = 200;
```

```
for j = 1:1:M
    Q1 = inv(Q);
    F = (lamda)'*Q1*B'*lamda;
    F1 = inv(R-F);
    FRs(j) = [F1*R*FRsp-F1*((ysp)'*B'*lamda-lamda'*A*Q1*B'*lamda-B1'*lamda)];
    ys = ysp-Q1*A'*lamda-Q1*B'*lamda*FRs(j);
    e_lamda = A*ys+B*ys+B1*FRs(j)+W*yf;
    lamda = lamda+alpha*e_lamda;
    if (e_lamda(j)'*e_lamda(j)<=e_sml);
      else j = j+1
    end
end
```

```
%
%
%MATLAB PROGRAM FOR DYNAMIC MODEL
%
%
%Equilibrium equation of resin/solid phase relationship.
a = [1.1;1.2;1.5;1.55;1.61;1.66;1.68;1.8];
b = [0.08;0.09;0.1;0.11;0.12;0.13;0.14;0.15];
%
%Flow-rate, Liquid and Resin holdups.
FL = 2000;
H = 28.96;
h = 37.62;
%
%Model matrices entries.
for i = 1:1:8
    l(i) = FL/(H+a(i)*h);
    m(i) = a(i)/(H+a(i)*h);
end
for i = 1:1:7
    k(i) = b(i+1)-b(i);
    mj(i) = a(i+1)/(H+a(i)*h);
end
k(8) = -b(8);
%
%A MATRIX
A(1,1) = -l(1);
A(2,1) = l(2);
A(2,2) = -l(2);
A(3,2) = l(3);
A(3,3) = -l(3);
A(4,3) = l(4);
A(4,4) = -l(4);
A(5,4) = l(5);
A(5,5) = -l(5);
A(6,5) = l(6);
A(6,6) = -l(6);
A(7,6) = l(7);
A(7,7) = -l(7);
A(8,7) = l(8);
A(8,8) = -l(8);
```

```
%
%B MATRIX
B(1,1) = -m(1);
B(1,2) = mj(1);
B(2,2) = -m(2);
B(2,3) = mj(2);
B(3,3) = -m(3);
B(3,4) = mj(3);
B(4,4) = -m(4);
B(4,5) = mj(4);
B(5,5) = -m(5);
B(5,6) = mj(5);
B(6,6) = -m(6);
B(6,7) = mj(6);
B(7,7) = -m(7);
B(7,8) = mj(7);
B(8,8) = -m(8);
%
%B1 MATRIX
B1 = k';
%
%W MATRIX
W = [l(1);0;0;0;0;0;0;0];
%
%C MATRIX
C=[0;0;0;0;0;0;0;1];
%
%Weighting matrices Q and R.
Q' = [100;100;100;100;100;100;100;100];
Q = diag(Q');
R = [0.12];
%
%Introducing the model parameters
K = 200;
N = 8;
ys = [0,1,2,3,3,2,1,0];
FRs = [0.1];
L = [-1;1.5;-2;-3;-4.5;-5;-6;7];
Ldiag = diag(L);
S = [10;-10;10;-10;10;10;10;10];
Sdiag = diag(S);
GK1 = Sdiag;
Q = [1;1;1;1;1;1;1;1];
Qd = diag(Q);
R = 0.1;
%
%Solution for Riccatti equation G(k)=Q+L^TG(k+1)[I+RG(k+1)^-2]L
G1 = zeros(N,N);
G2 = zeros(N,N);
G3 = zeros(N,N);
RI = inv(R);
```

```
for k = K:-1:0
    G1 = (RI*Sdiag)+eye(size(A));
    G2 = inv(G1)*Ldiag
    G3 = Sdiag*G2;
    GK = Qd+Ldiag'*G3
    GK1 = GK
end
%
%Calculation of the controller matrix H=-R^-1(L^T)^-1(G-Q)
G = GK
H = -RI*inv(Ldiag')*(G-Qd);
```

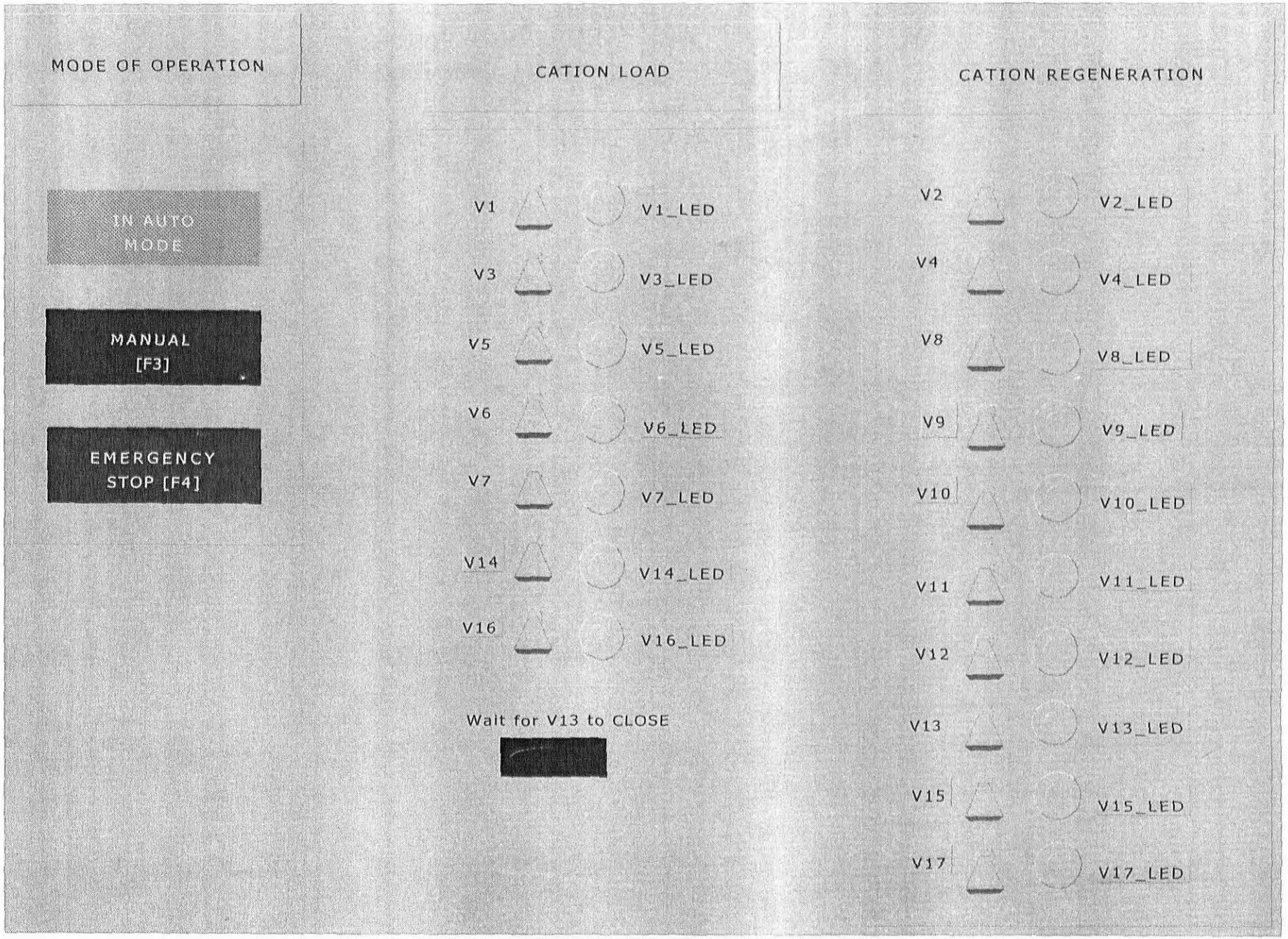
APPENDIX B

---

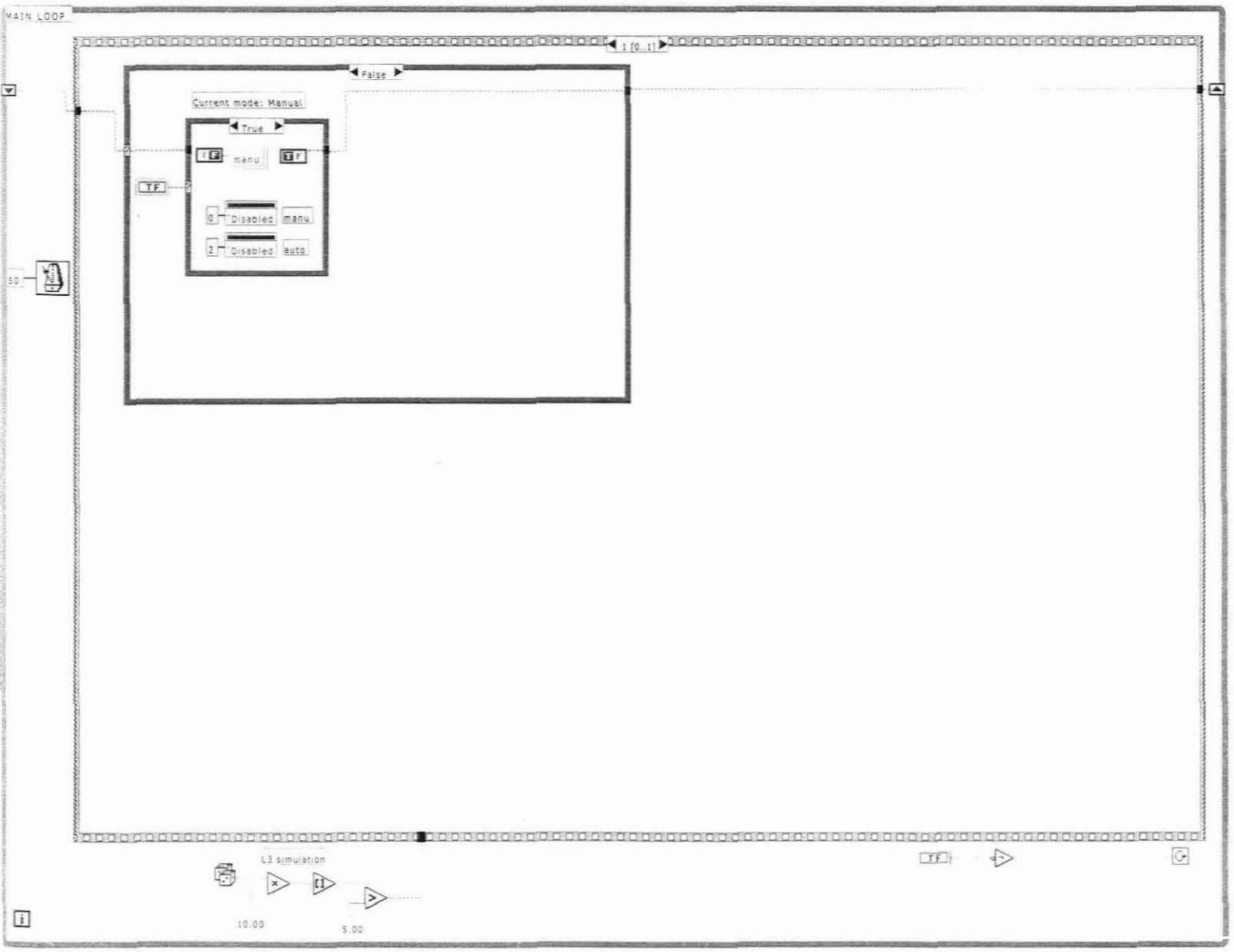
7. Sequence Control Program – LabVIEW Program.

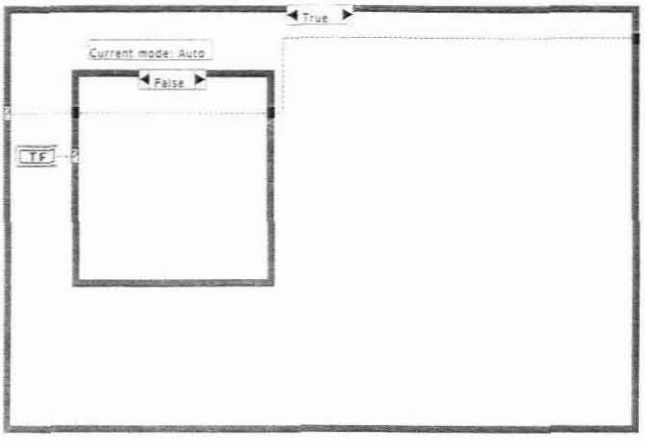
Front Panel
Connector Pane

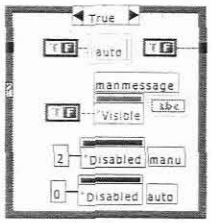
sequence_control_001.vi

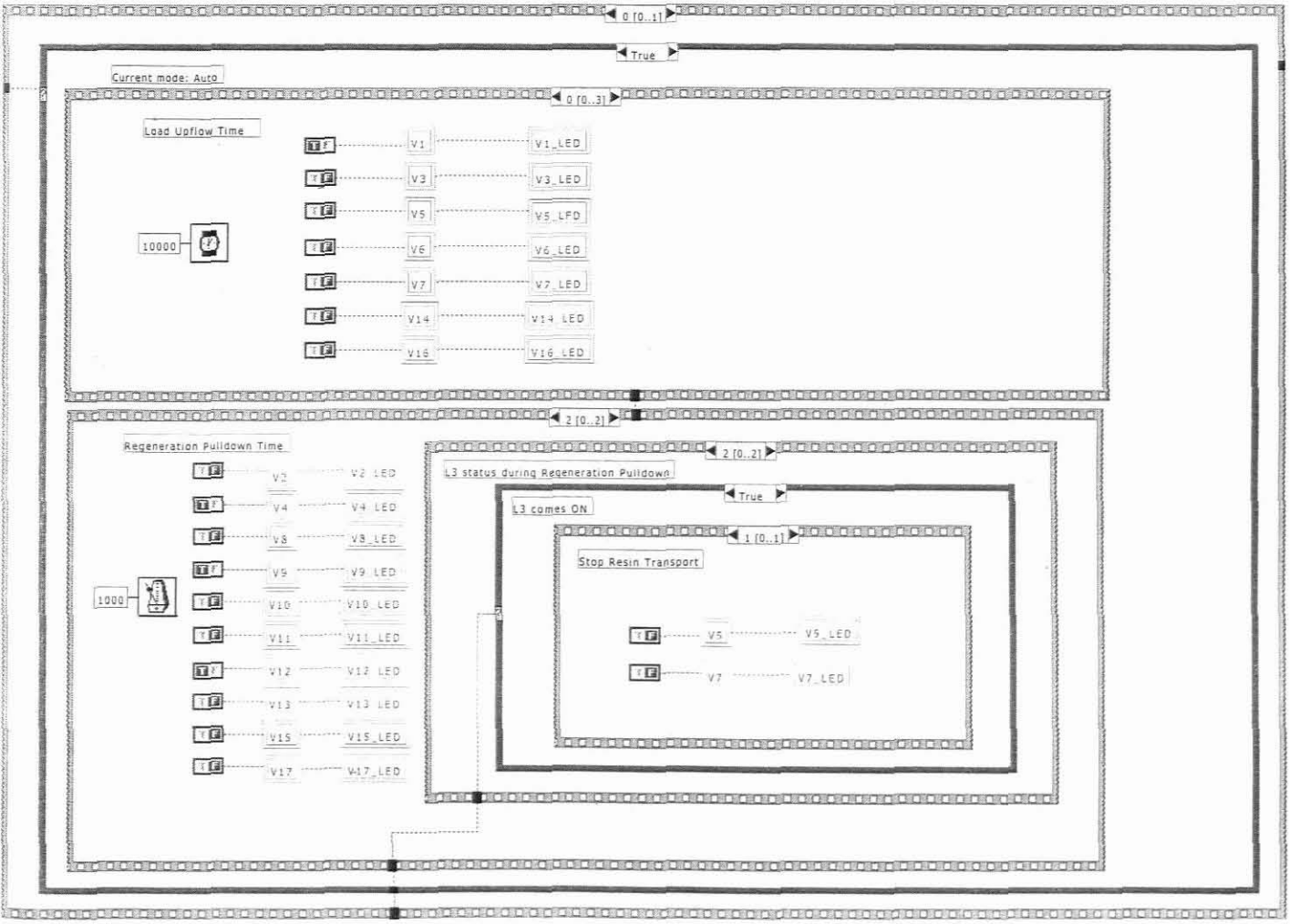

MODE OF OPERATION
IN AUTO MODE
MANUAL [F3]
EMERGENCY STOP [F4]
CATION LOAD
V1
V1_LED
V3
V3_LED
V5
V5_LED
V6
V6_LED
V7
V7_LED
V14
V14_LED
V16
V16_LED
Wait for V13 to CLOSE
CATION REGENERATION
V2
V2_LED
V4
V4_LED
V8
V8_LED
V9
V9_LED
V10
V10_LED
V11
V11_LED
V12
V12_LED
V13
V13_LED
V15
V15_LED
V17
V17_LED

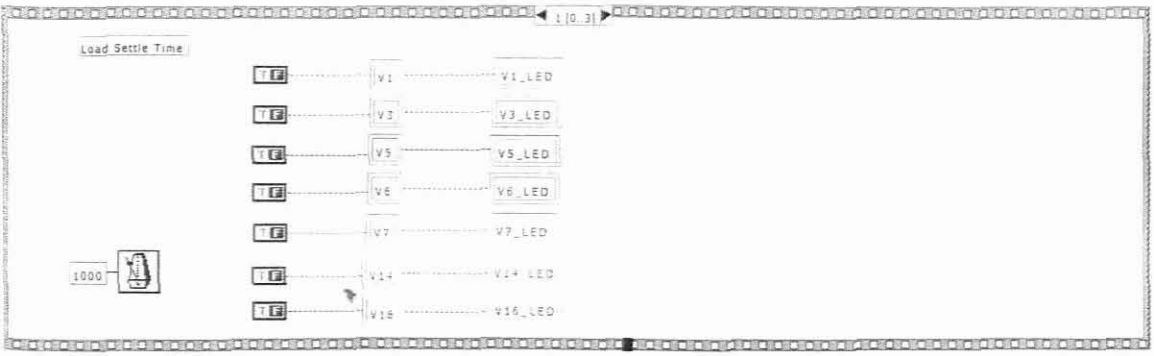
Block Diagram

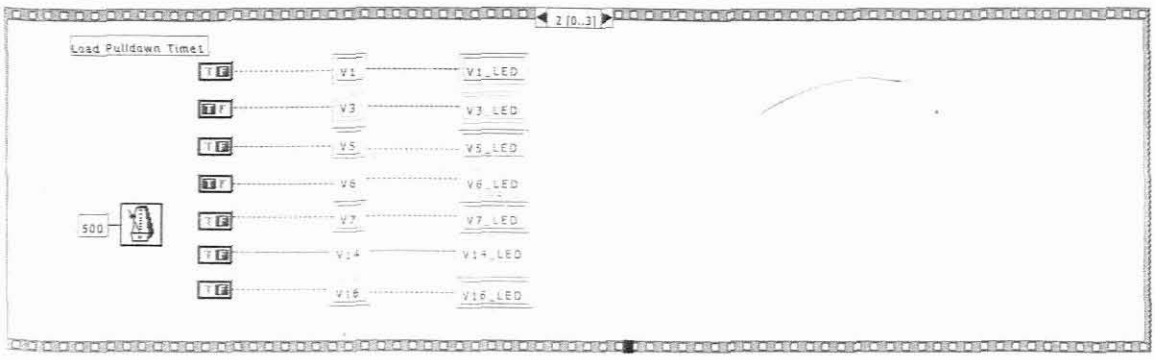


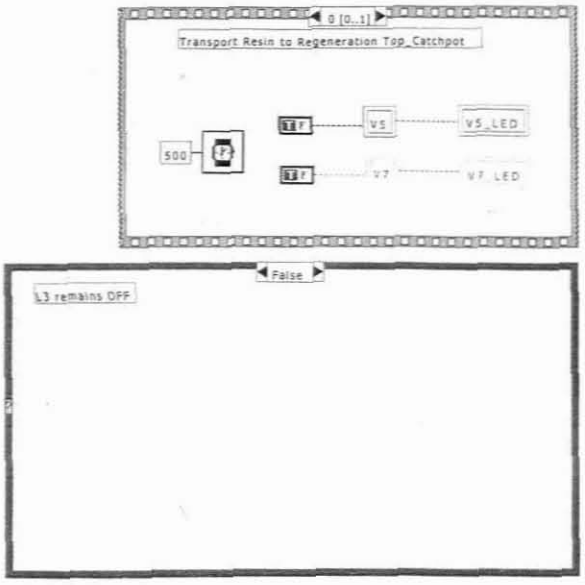


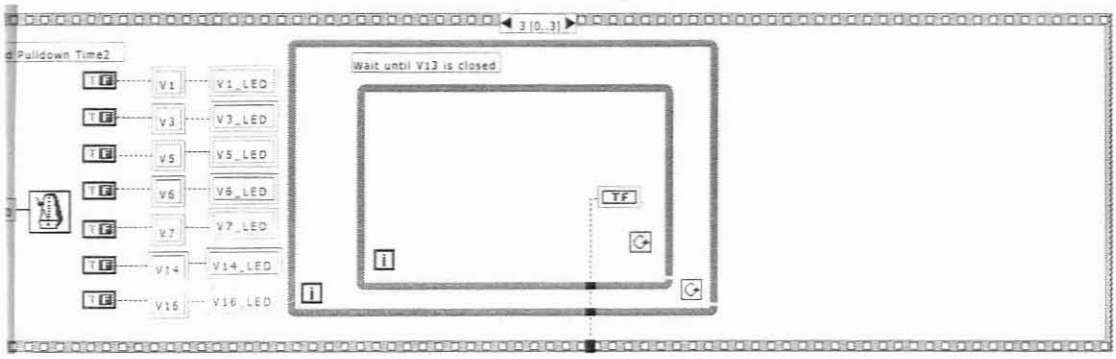

True
auto
manmessage
Visible
abc
2
Disabled
manu
0
Disabled
auto

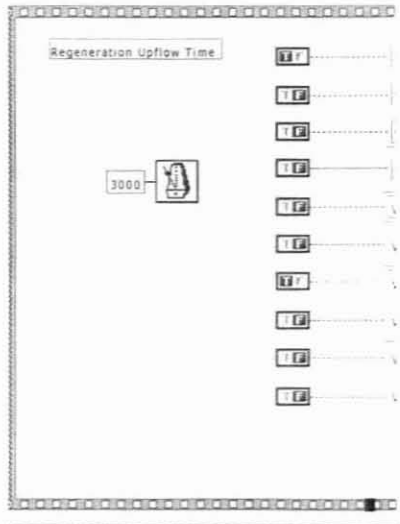

0 [0..1]
True
Current mode: Auto
0 [0..3]
Load Upflow Time
10000
V1
V1_LED
V3
V3_LED
V5
V5_LED
V6
V6_LED
V7
V7_LED
V14
V14_LED
V16
V16_LED
2 [0..2]
Regeneration Pulldown Time
1000
V2
V2_LED
V4
V4_LED
V8
V8_LED
V9
V9_LED
V10
V10_LED
V11
V11_LED
V12
V12_LED
V13
V13_LED
V15
V15_LED
V17
V17_LED
2 [0..2]
L3 status during Regeneration Pulldown
True
L3 comes ON
1 [0..1]
Stop Resin Transport
V5
V5_LED
V7
V7_LED

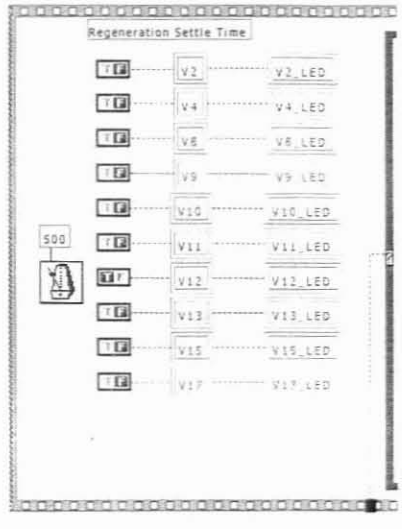

1 [0..3]
Load Settle Time
1000
V1
V1_LED
V3
V3_LED
V5
V5_LED
V6
V6_LED
V7
V7_LED
V14
V14_LED
V16
V16_LED

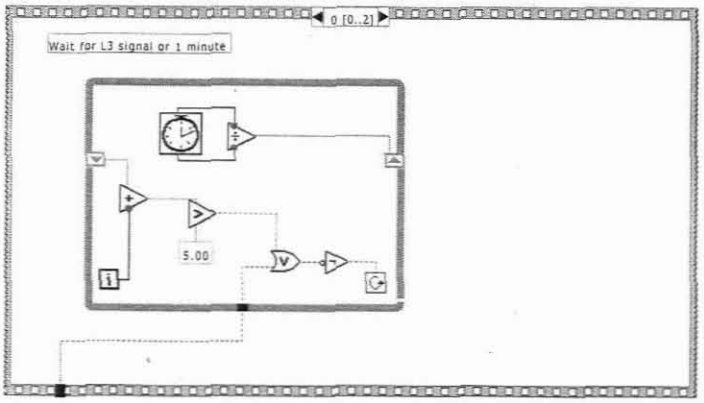

2 [0..3]
Load Pulldown Time1
500
V1
V1_LED
V3
V3_LED
V5
V5_LED
V6
V6_LED
V7
V7_LED
V14
V14_LED
V16
V16_LED

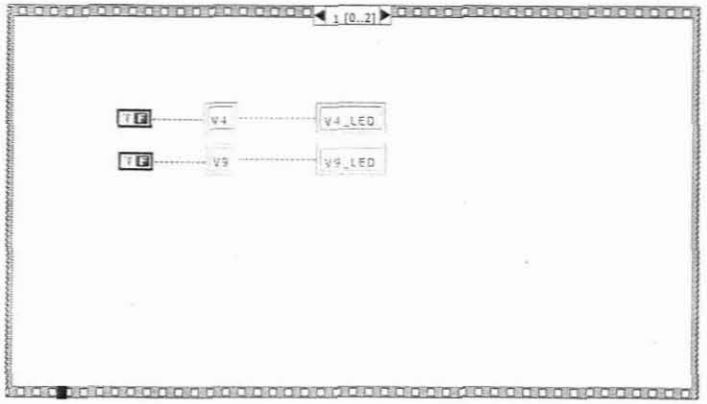

0 [0..1]
Transport Resin to Regeneration Top_Catchpot
500
V5
V5_LED
V7
V7_LED
False
L3 remains OFF

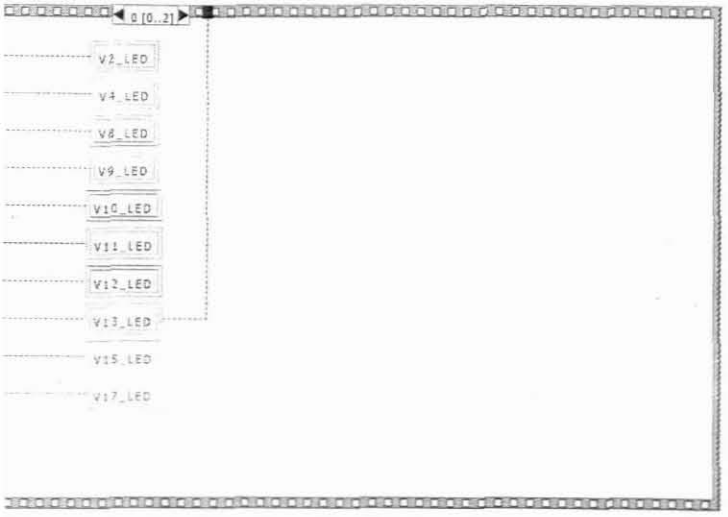

Pulldown Time2
V1
V1_LED
V3
V3_LED
V5
V5_LED
V6
V6_LED
V7
V7_LED
V14
V14_LED
V16
V16_LED
3 (0..3)
Wait until V13 is closed
TF

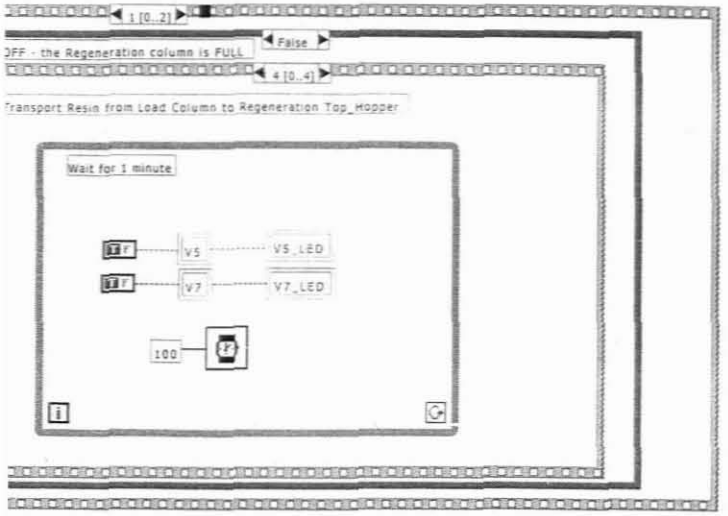

Regeneration Upflow Time
3000

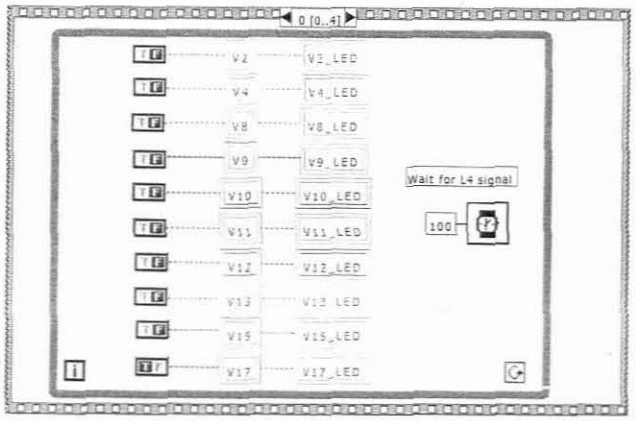

Regeneration Settle Time
500
V2
V2_LED
V4
V4_LED
V8
V8_LED
V9
V9_LED
V10
V10_LED
V11
V11_LED
V12
V12_LED
V13
V13_LED
V15
V15_LED
V17
V17_LED

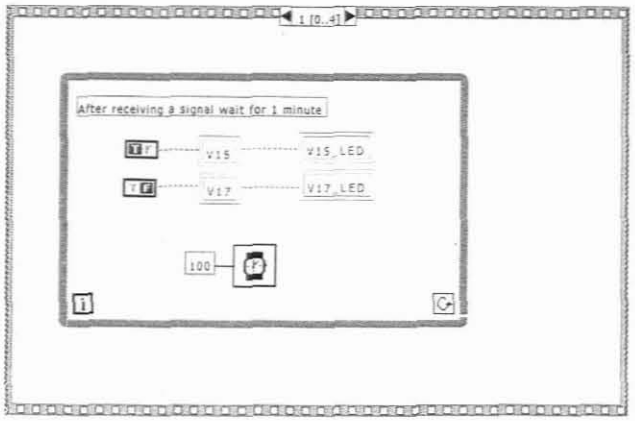

0 [0..2]
Wait for L3 signal or 1 minute
5.00

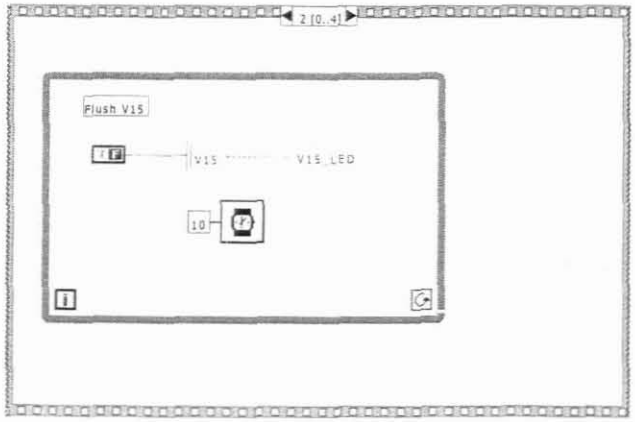

1 [0..2]
V4
V4_LED
V9
V9_LED


0 [0..2]
V2_LED
V4_LED
V8_LED
V9_LED
V10_LED
V11_LED
V12_LED
V13_LED
V15_LED
V17_LED

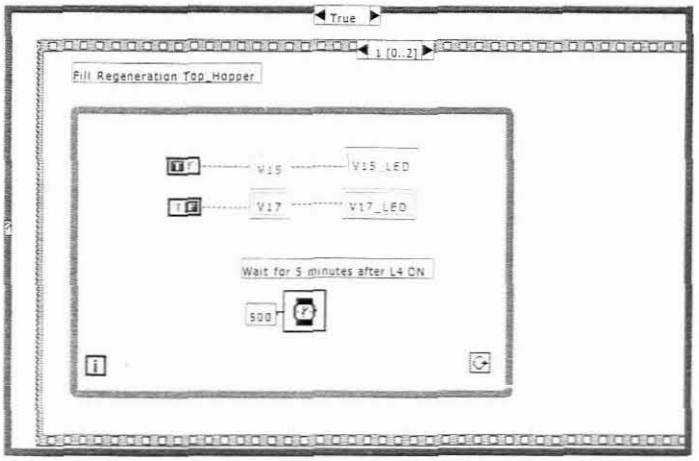

1 [0..2]
False
OFF - the Regeneration column is FULL
4 [0..4]
Transport Resin from Load Column to Regeneration Top_Hopper
Wait for 1 minute
V5
V5_LED
V7
V7_LED
100

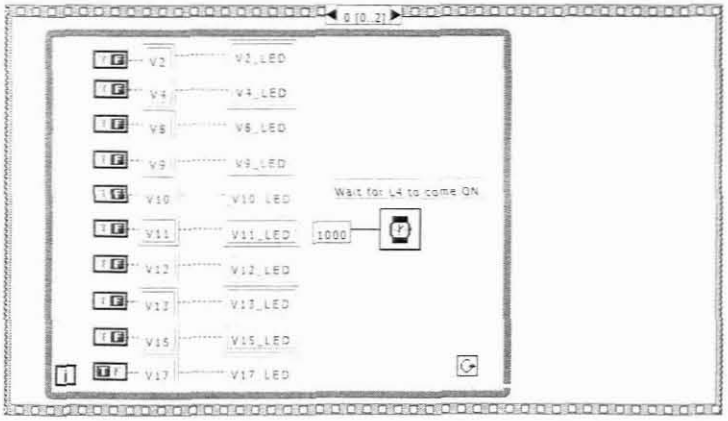

0 [0..4]
V2
V2_LED
V4
V4_LED
V8
V8_LED
V9
V9_LED
V10
V10_LED
V11
V11_LED
V12
V12_LED
V13
V13_LED
V15
V15_LED
V17
V17_LED
Wait for L4 signal
100

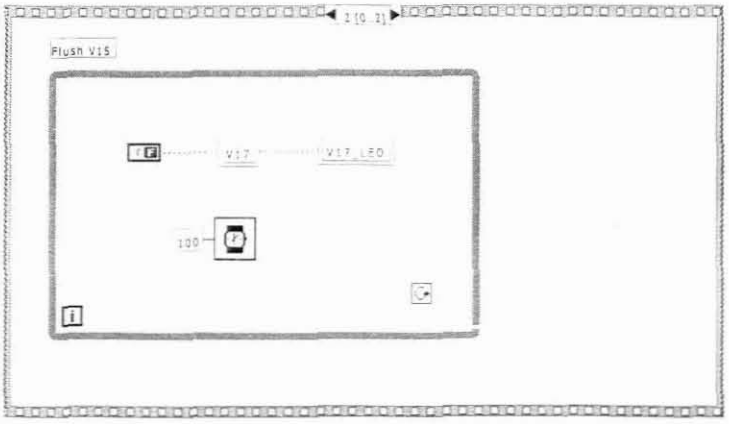

1 [0..4]
After receiving a signal wait for 1 minute
V15
V15_LED
V17
V17_LED
100

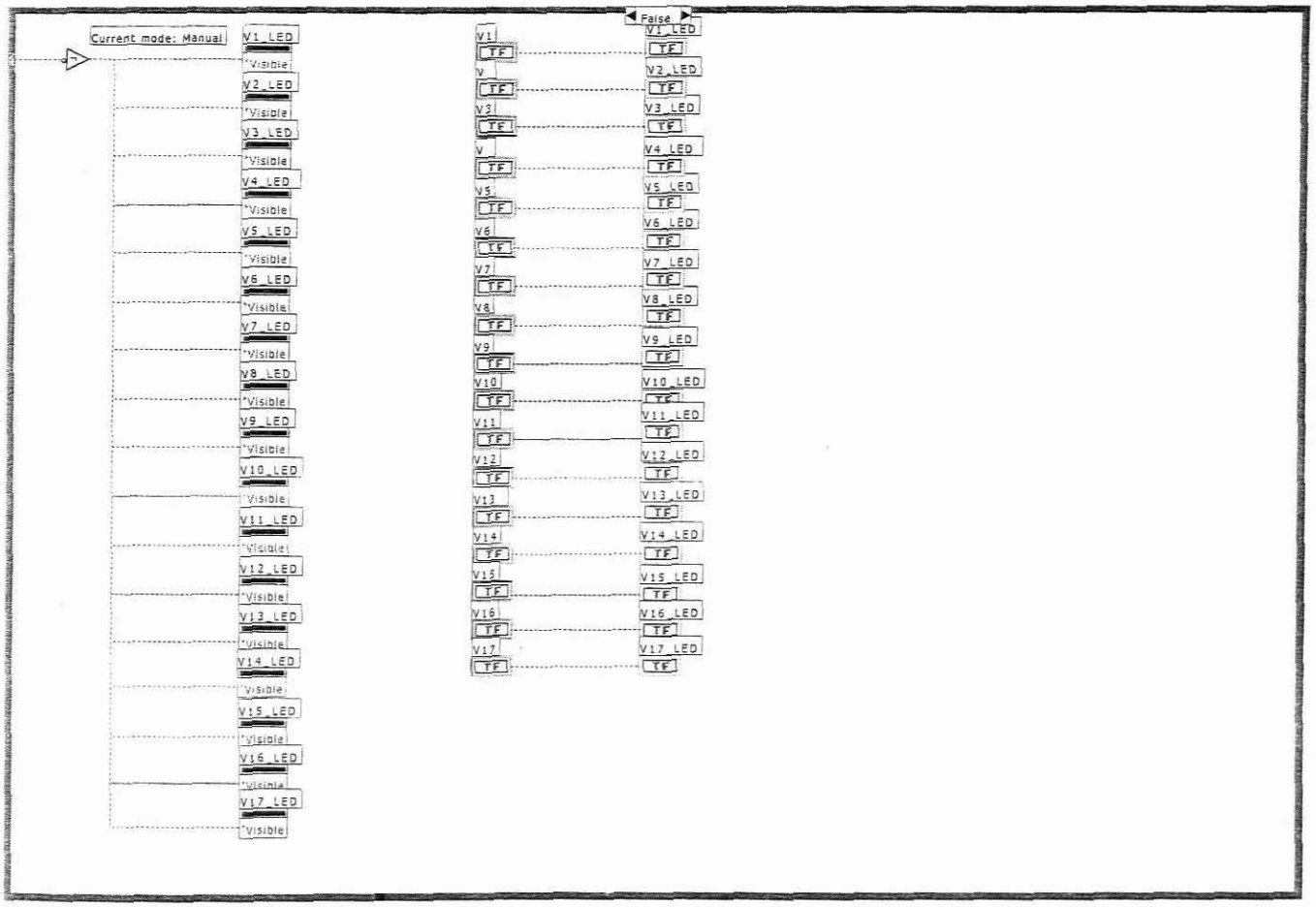

2 [0..4]
Flush V15
V15
V15_LED
10


3 [0..4]
V15
V15_LED
V17
V17_LED


True
1 [0..2]
Fill Regeneration Top_Hopper
V15
V15_LED
V17
V17_LED
Wait for 5 minutes after L4 ON
500


0 [0..2]
V2
V2_LED
V4
V4_LED
V8
V8_LED
V9
V9_LED
V10
V10_LED
V11
V11_LED
V12
V12_LED
V13
V13_LED
V15
V15_LED
V17
V17_LED
Wait for L4 to come ON
1000


1 [0..1]
Flush V15
V17
V17_LED
100


Current mode: Manual
V1_LED
V2_LED
V3_LED
V4_LED
V5_LED
V6_LED
V7_LED
V8_LED
V9_LED
V10_LED
V11_LED
V12_LED
V13_LED
V14_LED
V15_LED
V16_LED
V17_LED
Visible
False
TF