

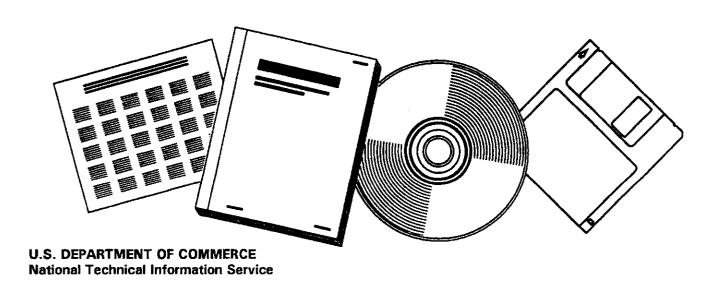
DE82006944



SOLVENT-REFINED-COAL (SRC) PROCESS: AXIAL DISPERSION IN TALL BUBBLE COLUMNS - TRACER TESTS

PITTSBURG AND MIDWAY COAL MINING CO. ENGLEWOOD, CO

JAN 1982



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SOLVENT REFINED COAL (SRC) PROCESS

Axial Dispersion in Tall Bubble Columns-Tracer Tests

By K. Parimi M. D. Pitchford

January 1982

Work Performed Under Contract No. AC05-76ET10104

The Pittsburg & Midway Coal Mining Company Englewood, Colorado



U. S. DEPARTMENT OF ENERGY



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SOLVENT REFINED COAL (SRC) PROCESS

Axial Dispersion in Tall Bubble Columns-Tracer Tests

> K. Parimi M. D. Pitchford

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GULF RESEARCH & DEVELOPMENT COMPANY Pittsburgh, Pennsylvania

AXIAL DISPERSION IN TALL BUBBLE COLUMNS TRACER TESTS

by

K. Parimi M.D. Pitchford

Technology & Materials Department Report No. 560RM160 File FT12 September, 1981

Prepared for the United States Department of Energy Under Contract No. DE-ACO1-79ET10104

AXIAL DISPERSION IN TALL BUBBLE COLUMNS TRACER TESTS

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SUMMARY

The degree of backmixing is an important consideration in the design and scale-up of SRC-II reactors. Several qualitative tests were therefore conducted on the 25 ft. plexiglass bubble column in order to visually observe the axial dispersion or backmixing characteristics of a column of this size. A concentrated solution of Methyl-Orange was injected into the column, and the dispersion of the dye throughout the column was observed and photographed. These observations indicated that the backmixing level was not as extensive as the existing correlations would predict. Since backmixing plays an important role in the design and scale-up of SRC II reactors, it was decided to follow up with additional quantitative tests for further elucidation of this aspect of bubble column performance. The required test apparatus was assembled and tracer tests using an electrolytic tracer in the form of a 10 N NaOH solution were conducted.

The test results confirmed the visual observations; that the degree of backmixing was less than what was expected using existing literature correlations. Part of the reason for the discrepancy between the data and theory may be due to the large extrapolation involved in using the theory, but more importantly, there is the question of adequacy of the model to describe the complex mixing patterns present in the column. Implicit in using any of the existing correlations to predict backmixing is the assumption that a simple dispersion model can adequately describe the complex mixing patterns in a large bubble column. This is not a valid assumption when the column operates well beyond the quiescent bubble flow regime. There is, therefore, a real need to identify models which would represent more closely the fluid dynamic

behavior of large columns and which can be used confidently for design and scale-up.

INTRODUCTION

The knowledge of the fluid dynamic behavior of a multiphase flow system required for the design of the SRC-II dissolver, included an understanding of the residence time distribution of each phase as the different phases comprising the system flow cocurrently in the vessel and the manner in which they are distributed with respect to each other. This latter property defines the rates of mass and heat transfer between the phases and is an important factor which determines the overall performance of the reactor.

The residence time distribution function for each phase defines, in simple terms, how long a given fluid element in that phase stayed in the vessel. However, it does not describe the residence time history of that element during its life in the vessel - the distribution of this time spent in the vessel between the different reaction environments or between the phases if that element happened to cross the interfacial boundary between phases. These latter effects can be ignored only when the interactions between fluid elements are unimportant as in linear or pseudo linear reaction rate processes. In conventional terminology, ignoring the residence time history of a fluid element is called a second-order approximation and this simplification does not provide a reasonable approximation for multiphase flow systems.

Residence time distribution functions for the flowing fluid are derived from tracer tests. In these tests, usually a pulse of a tracer is introduced into the system and its concentration as a function of time is

continuously measured at a convenient inlet and outlet location. When these tracer response curves are well behaved, they can be characterized by their first and second moments. The first and second moments, or the difference between the respective moments of the input and output curves, give the mean residence time and the degree of backmixing, respectively. property of the system, i.e. the degree of backmixing, is an important consideration in the design and scale-up of any single or multiphase flow In the SRC-II reactor, specifically, backmixing plays a very important role; for example, the design of the required preheat for the slurry feed to the reactor depends upon the extent of backmixing in the large scale vessel. Several qualitative tests were conducted on the 25 ft. bubble column to examine the mixing or liquid dispersion characteristics of such a A concentrated solution of Methy-Orange was injected into the column at three different locations - inlet, middle, and top of the column, and the dispersion of the dye throughout the column was observed and photographed. The dispersion patterns observed indicated that the backmixing level was not as extensive as the existing correlations to predict backmixing would As this is an important consideration in the design of SRC-II Demonstration Plant dissolver, it was decided to perform additional experiments with quantitative tracer tests to further elucidate this aspect of bubble column behavior. This report describes the test apparatus assembled for this purpose, details of the tests conducted, and the results of their analysis.

TRACER TESTS

The experimental system for the tracer studies is shown schematically in Figure 1. The bubble column used in these studies is the same column used previously for studying the solids withdrawal aspects of three-phase fluidized bed reactors. Provision was made in the design of the experimental system to inject tracer either at the bottom, middle, or top of the column. The tracer injection system itself is shown in Figure 2 (Drawing No. PC 30219-A measured quantity of concentrated sodium hydroxide solution, about 150 ml of 10 N solution, is used as the tracer material. The solution is introduced into the pressurized cell numbered 4 by opening valves designated by letter 'L' and closing valves designated by letter 'M'. The buret is then isolated, valves designated by letter 'M' opened to allow nitrogen from cylinder to exert about 100 psi pressure on the tracer solution in the vessel number 4. The tracer is then injected into the column at the desired location by quickly opening the solenoid valve, C-4, and its concentration, as a function of time, is measured continuously. The conductivity cells are the primary elements of concentration measurement. These are Uniloc Model 112-09 The continuously measured concentrations are recorded on a multicells. channel strip chart recorder. A comprehensive data acquisition system that would simultaneously collect, digitize, and present these data in a real-time environment was designed and assembled. Details of this system are described in a separate report included in Appendix A. The system has the capability to interface with a PDP-11 computer and also floppy disk storage. seen, the microprocessor included in the system continuously converts the measured conductivity to concentration units after applying appropriate corrections for baseline for each data set and also is capable of doing other required computations and resending these data in a form directly usable. The present as well as the future system capabilities are described in the report. The location for the conductivity cells is shown in Figure 2, and the modifications of the column required to perform these studies are shown in Figure 3 (Drawing No. PC 30219-10). Both the conductivity cells are flow-through instruments. In order to assure adequate flow through the cell, an in-line pump is used for the bottom cell.

A number of tracer tests, investigating the effect of varying gas and liquid rates on the bubble column operation were performed. Significant time, however, had to be spent prior to obtaining any usable data from the tests in establishing the quantity, concentration, and the type of tracer used; this involved a number of trial runs, adjustments and modifications to hardware in the data acquisition system. In the final tests, gas rate was varied from 0 to 10 cm/sec while maintaining liquid rate either at 2 or 3 cm/sec. Difficulty was experienced in performing these tests at low liquid rates because of insufficient flow through the conductivity cell at the top of the column at the low liquid velocities.

A tracer test itself basically consisted of rapidly injecting a tracer in sufficient quantity at any given location in the column and continuously measuring its concentration at both the inlet and outlet of the column. Even though, there was provision to introduce tracer either at inlet, middle, and top of the column, in the tests completed thus far in this program, tracer was injected only at the inlet. Tracer injections at other locations were planned but because of the early termination of the project, this part of the work was not completed.

DATA ANALYSIS AND RESULTS

There were altogether eight tests completed. These were conducted with gas superficial velocities maintained at 0, 2, 4, and 10 cm/sec at two different liquid superficial velocities of 2 and 3 cm/sec. A sample set of input and output curves (raw data) is shown in Figure 4. The reduced data corresponding to a liquid superficial velocity of 3 cm/sec. are plotted in Figures 5 to 8. On each figure, an input and output tracer response curve is shown, corresponding to a given gas and liquid flow rate, as indicated on the figure.

In general, there are two basic steps that are involved in modeling mixing patterns in any processing equipment. These are:

- (1) Postulating a flow model which conforms to our best physical understanding of the system.
- (2) Evaluation of the model parameters which provide a quantitatively adequate description of the mixing process.

There are a number of simple and combined models commonly used to describe mixing. Once a model is postulated, the model parameters are estimated from a tracer study either by moments analysis or by a regression technique. In the moments analysis, the various moments of the mathematical model are derived in terms of the model parameters. To evaluate K model parameters, K moments are computed from the data and set equal to the corresponding equations from the model. These methods have several drawbacks, namely, the method places undue emphasis on small portions of the data, ignoring or only partially using the remainder of it and that the method tacitly assumes a priori that the model is a

perfect or nearly perfect fit to the data. The validity of parameters obtained by such a method clearly depends upon the correctness of that assumption, about which little information is provided by the method itself. The regression method, on the other hand, is relatively free of these deficiencies. In one variation of this method, first a numerical transfer function in the Fourier domain is obtained from the input and output data and then the parameters in the postulated model are so chosen as to give the best fit to the experimental transfer function. Even though this latter method of analysis is preferrable and plans were made to employ this method, because of the early termination of the project, a simpler approach was used to analyze the data. In this approach, it is assumed that a one-dimensional axial dispersion model adequately describes the mixing patterns in the column and with this assumption, model parameters are estimated using moments analysis.

In the moments method of analysis, the first and second moments for the input and tracer curves corresponding to each test are calculated. The first moments give estimates of average fluid residence time in the column. The second moment can be related $^{\{1\}}$ to the Peclet number, which characterizes the degree of backmixing in the vessel;

$$\Delta \sigma^2 = (\sigma^2_{out} - \sigma^2_{in}) / \tilde{t}^2 = \frac{2}{Pe} - (\frac{2}{Pe}) (1 - e^{-Pe})$$
 (1)

where,

Levenspiel, O., Chemical Reaction Engineering, John Wiley & Sons, Inc., Second Edition, 1972.

oin, oout = Variance of input and output curves, respectively.

 \tilde{t} = Mean residence time

Pe = UL/D

D = Dispersion coefficient, cm²/sec.

U = Liquid velocity, cm/sec.

E = Axial distance between the input and output measurement points, cm.

Table 1 shows the results of these calculations. In the table, the calculated gas holdup values and Peclet numbers were compared with the predicted values. In case of gas holdups, a comparison was also made with directly measured values and the agreement between the two, one directly measured and the other inferred from the tracer data was good, as can be seen from the data in Table 1. Also, these values agreed well with the predictions using the correlation of Akita and Yoshida. (2)

In case of dispersion levels, as measured by Peclet numbers, however, the agreement between the data and the predictions using Deckwer, et al.'s $^{(3)}$ correlation was poor. Deckwer's model substantially overestimated the degree of backmixing in the tall bubble column used in this program.

⁽²⁾ Akita, K. and Yoshida, F., Ind. Eng. Chem. Process Des. and Dev., 12, 76

⁽³⁾ Deckwer, W. D., Burckhart, R., and Zoll, G., Chem. Eng. Science, 29, 2177 (1974).

CONCLUSIONS AND RECOMMENDATIONS

Tracer tests conducted on the 30 cm x 762 cm bubble column indicated that substantially less backmixing was present in the column than was predicted by Deckwer, et al.'s correlation. This discrepancy between the data and the predictions of Deckwer, et al.'s correlation may be due to the large extrapolation involved in using their correlation. But also of importance is the question of appropriateness of the axial dispersion model to represent the complex mixing patterns in the column. In design and scaleup of bubble column reactors, what is of utmost importance is proper and adequate representation of the mixing patterns by using a suitable model and relating the parameters of the model to the system properties such as reactor geometry, volume, flow rates, and fluid properties. Further research is required in this area specifically to identify models which would represent more closely the fluid dynamic behavior of large scale bubble columns.

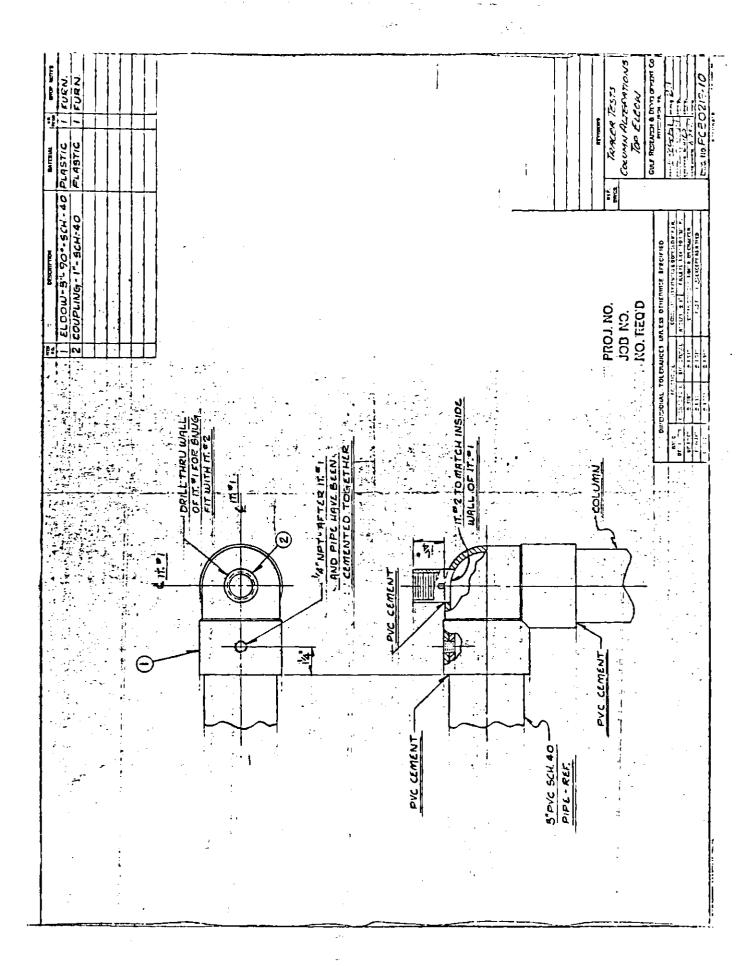
Table 1
Tracer Test Results

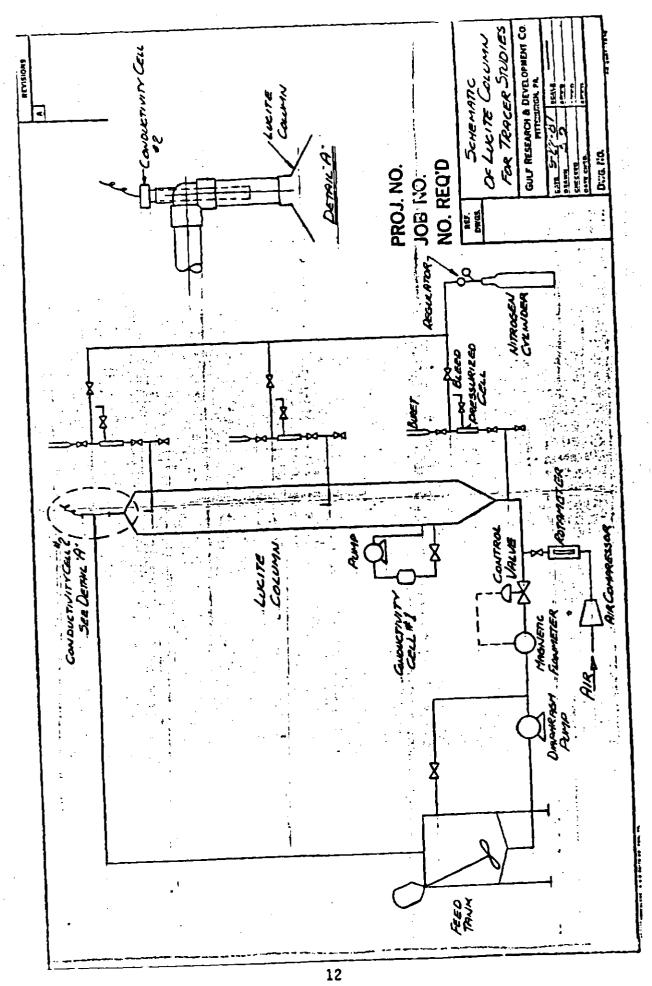
Test No.	Flui Velocit cm/se Liquid	ies	Mean Residence Time,Min	Gas I	loldup Predicted	Δσ ²	Peclet Observed	Number Predicted
1630 401	3	0	3.84			0.035	54.1	44.6 ^b
1	. 1i	-	3.60	0.063	0.061ª	0.266	6.5	3.5 ^c
2		2		•	0.105 ^a	0.311	5.4	2.9 ^C
3	11	4	3.45	0.102		-		2.2 ^c
4	tt	10	3.04	0.208	0.170ª	0.381	4.0	2.2

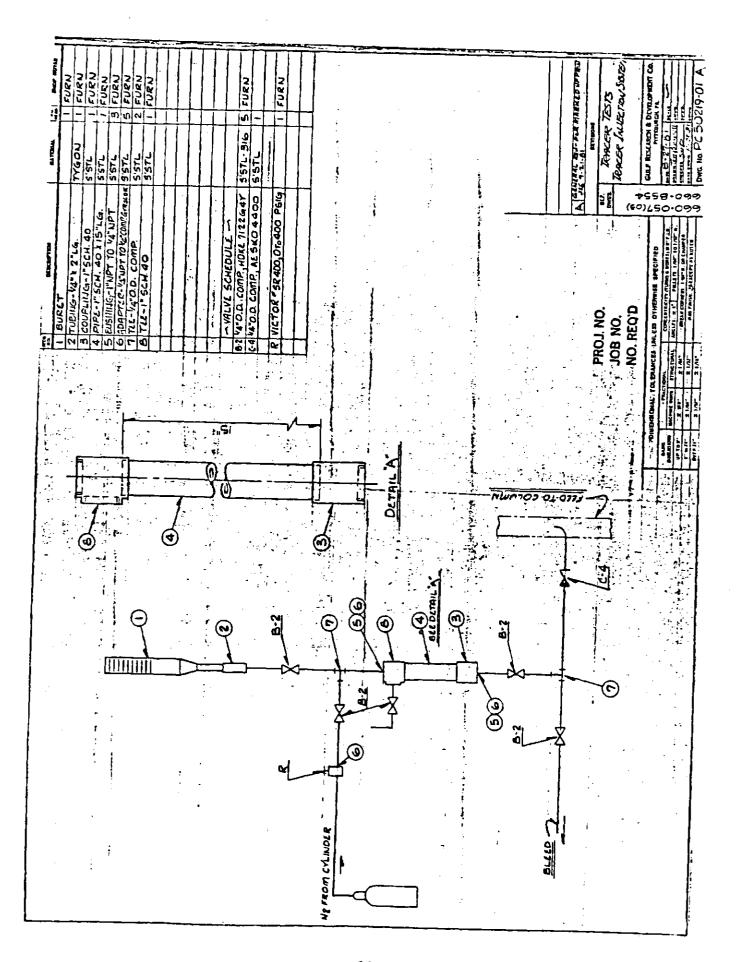
⁽a) Direct gas holdup measurements which agreed well with Akita & Yoshida correlation, Ind. Eng. Chem., Process Des. Develop., 13, 84 (1974).

⁽b) Data on dispersion of fluids flowing in pipes, O. Levenspiel, Ind. Eng. Chem., 50, 343 (1958).

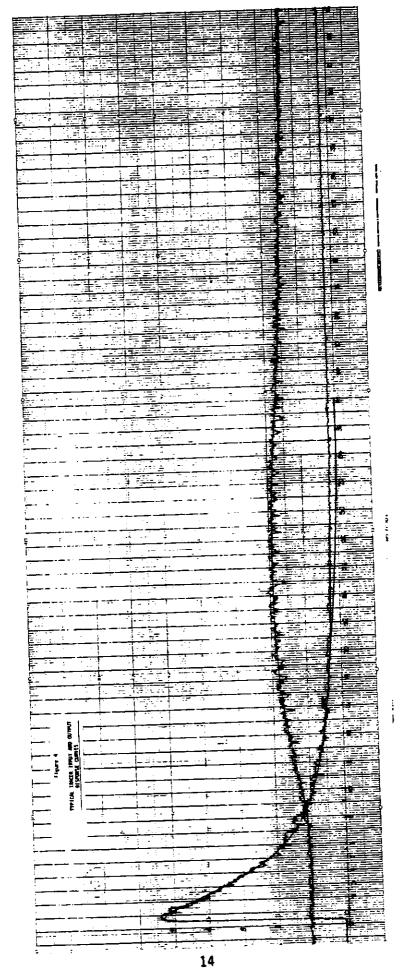
⁽c) Predicted using the correlation of Deckwer, et al., Chem. Eng. Sci., 29, 2177 (1974).

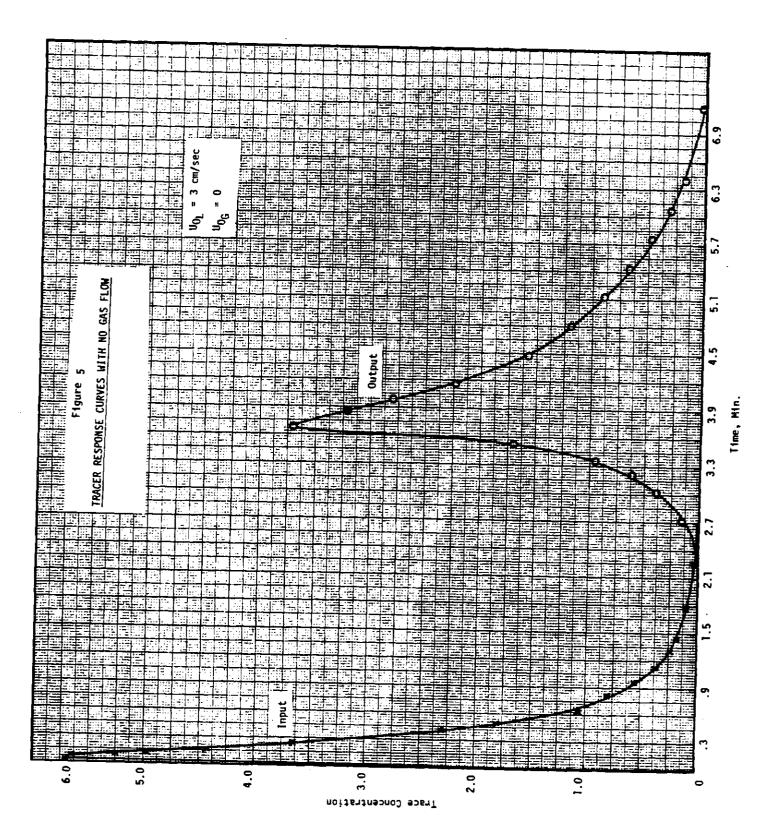


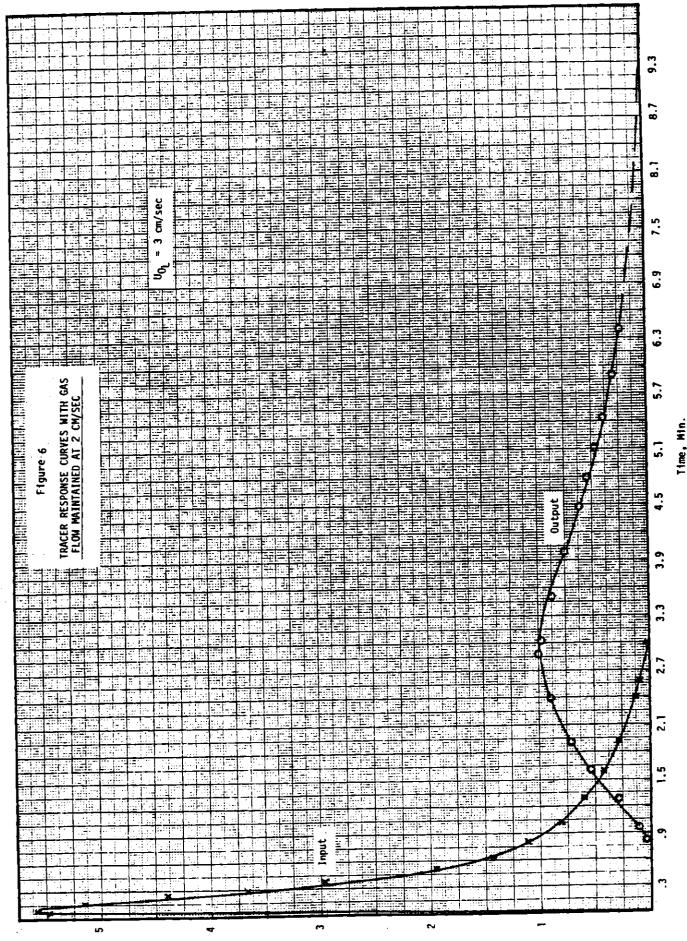


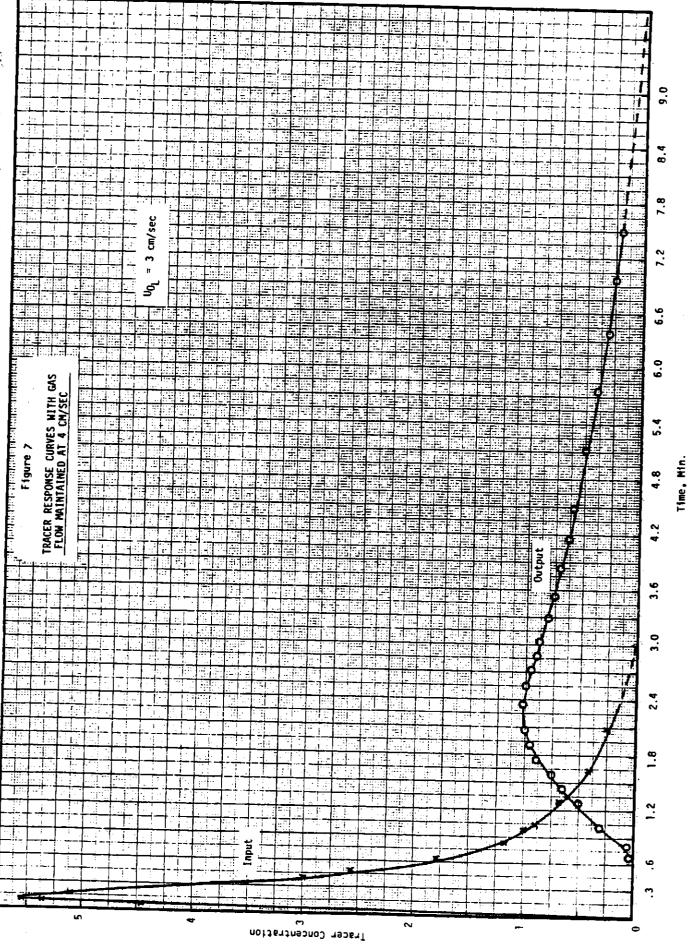


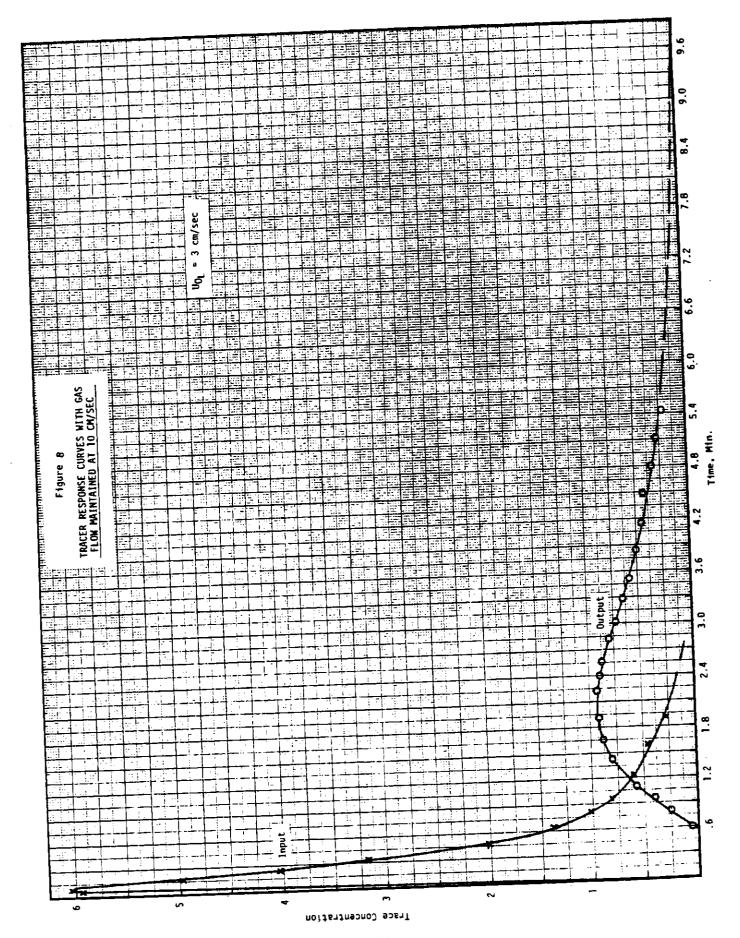
e Tagligae (1921)











APPENDIX

GULF SCIENCE & TECHNOLOGY COMPANY Pittsburgh, Pennsylvania

PRELIMINARY MANUAL ON
GMAC DATA SYSTEM
FOR
THREE-PHASE FLOW HYDRODYNAMICS

bу

Kent L. Miller

Systems & Controls Department Report No. 532TM032 File No. CA00

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1) INTRODUCTION

1.1) Description of the Problem

The three-phase flow hydrodynamics project has a pilot plant for studying the nature of mixing when gas is rising in a vertical column filled with water. This study is vital because many future energy processes involve multiphase flows and because the nature of multiphase flows is presently not well understood. Air and water are pumped into the bottom of a 30 cm diameter x 10 m high column and vented out the top. Salt is used as a tracer to study the mixing. Conductivity cells are used to sense the passage of a slug of salt solution. Then the known relation between conductivity and normality is used to compute the concentration.

The problem was to design a data acquisition system to collect and digitize the data in a real-time environment as well as provide a visual representation such as with stripcharts.

1.2) <u>Description of Solution</u>

A microprocessor based system was recommended and implemented. Four conductivity cells with transmitters were selected with a range of $0\text{--}50000~\mu$ mhos. Range selector switches provided increased sensitivity to low conductivity. These transmitters were input to GMAC (Gulf Microcomputer Acquisition and Control) System. GMAC performs the necessary calculations, prints out the normality of the salt solution, and drives the stripcharts. (See Figure 1.1).

GMAC is an in-house system that uses our own task-oriented Acquisition and Control language to facilitate software development. GMAC is based on the Motorola M6800 hardware that has become industry standard.

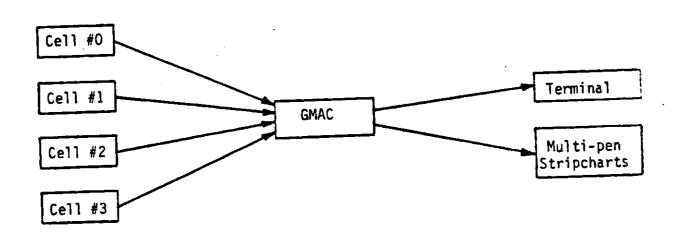


Figure 1.1 Schematic of GMAC System as Configured for Three Phase Flow Hydrodynamics Pilot Plant

1.3) Future Possibilities

Our GMAC system is interfacable with the PDP-11 and also to floppy disk storage. Presently the rate of data acquisition is limited by the speed of the terminal that must print out the data. A floppy disk or PDP-11 interface could vastly speed up the data acquisition.

2) DESIGN

2.1) <u>Hardware Configuration</u>

The components are:

- 4 Conductivity Cells (Uniloc Model 112-09, Cell Constant =5, 500-50000 µ mho)
- 4 Conductivity Transmitters (Uniloc Model 750-12, Isolated Output 4-20 m amps, Instrument Multipliers 100,1000,10000 Temp. Compensated to 25°C Reference)
- 1 M68MMLC1, Chassis w/Card Cage and Power Supply
- 1 M68MM15A1, HLA/D
- 1 M68MM15CI4, 4 Channel D/A
- 1 M68MM01A2-1, Micro Computer MCM21L14P2O, Ram (1024 x 4) MCM2532L, E Prom
- 1 M68MM14, 2 MHz Arith. Proc. Unit
- 1 E-2006 Cabinet 79-1/2"
- 4 RC-7758 Casters
- 5 PS-1255GY, Panels 31F1381 Hamilin, Solid State Relay
- 1 Terminal
- 1 Multipen Stripchart

The 4 cards were plugged into the chassis. The D/A card drove the stripcharts. The HLAD card read the conductivity transmitters. The APU card provided floating point arithmetic. The MC card contained: 1) PIA (Peripheral Interface Adapter) to read the Instrument Multipliers, read the run switch and ring the bell, 2) ACIA (Asynchronous Communications Interface Adapter) to write out to the terminal.

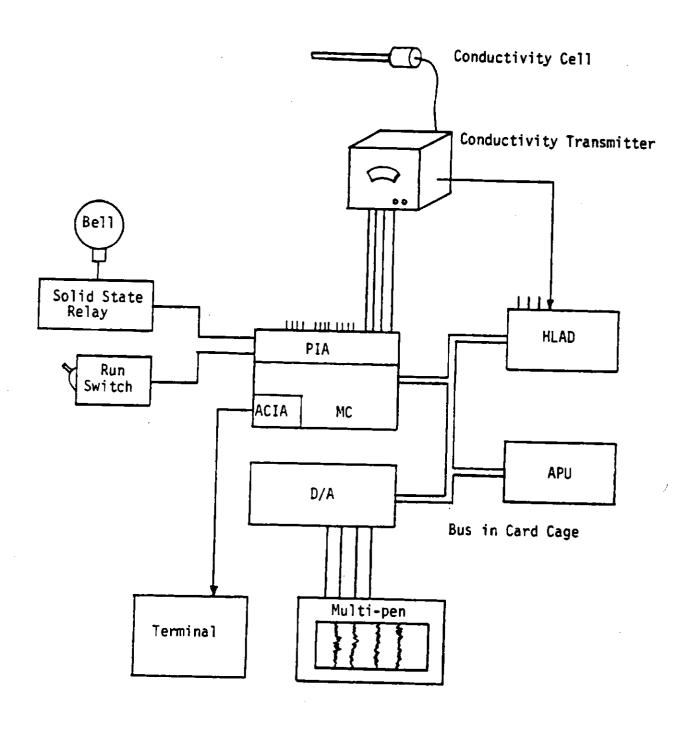
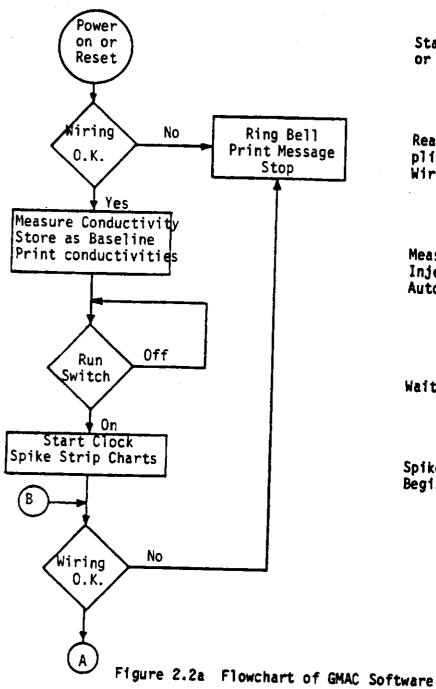


Figure 2.1 Schematic of Hardware Configuration

2.2) Software Configuration

While a detailed flowchart of the GMAC software is shown in the Appendix, I shall still present a macroscopic overview of what the software is trying to accomplish (see Figures 2.2a and 2.2b).



Start Here on Power-up or Reset.

Read Instrument Multipliers and Ring Bell if Wired Wrong.

Measure Before Salt is Injected. This is an Auto-Zero Feature.

Wait for Run Switch.

Spike on Charts Marks Beginning of Run.

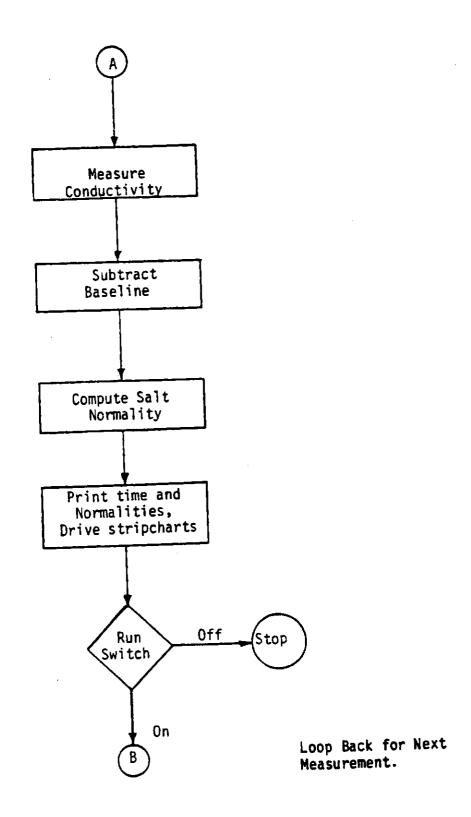


Figure 2.2b Flowchart of GMAC Software (cont.)

3) OPERATING INSTRUCTIONS

Installation:

- Insert conductivity cells into solutions.
- Connect cells to transmitters.
- Select range for transmitter (inside transmitter box).
- Connect stripcharts to DTOA terminals. All other connections inside cabinet should already be made, but are listed here for completeness.
- Connect transmitter to Microprocessor (Instrument
 Multiplier --> PIA) (Signal --> HLAD)
- Connect terminal to Microprocessor ACIA Port.Operation:
- Set run switch to OFF.
- Power on (terminal, stripchart, and microprocessor in that order). Immediately, the terminal should print the conductivity (μ mho) ready by the four cells. Then it should wait for run switch.
- Adjust Instrument Multipliers if desired and press RESET. The system will repeat the previous step.
- To take data, flip run switch ON. This starts the clock. The time and salt concentrations (equiv/l) should appear on the terminal at regular intervals.
- To stop data acquisition, flip run switch OFF.
- To get a new base-line, press RESET.
- Kill power when finished for the day.
- Maintain the conductivity cells as instructed by the manufacturers manual.

4) STATUS

Status as of mid-September:

Components	Arrived	Tested
4-Conductivity Cells 4-Conductivity Transmitters	One One	One One
Cabinet Castors Panels	X	X
Solid State Relays Chassis w/Card Cage & Power Supply HLAD	X X	X X
4 Channel D/A Micro Computer Ram (1024 x 4)	X X	X
E Prom APU	X	X

Software

Software is completed and awaits testing in final installation. One final iteration in software development is expected.

System

The components of the GMAC have been assembled and tested with simulated sensor input. Software passed these tests with flying colors. The remaining hardware has not arrived and thus the final system must yet be assembled, tested, and installed.

A) APPENDIX

A.1) Flowchart of GMAC Software

Presently, up to 255 tasks can be programmed into the GMAC EPROM. I have segmented the program into subroutines. The tasks allocated to these subroutines are shown in Figure A.1. The call hierarchy is shown in Figure A.2. Each of these subroutines is flow charted in the following pages.

Subroutine	Task #'s
Main	0-39
Cond	40-59
Auto-zero	60-71
Spike	72-79
Salt	80-119
Write	120-149
Common	150-179
Mult	180-229
Bell	230-239
Time	240-255

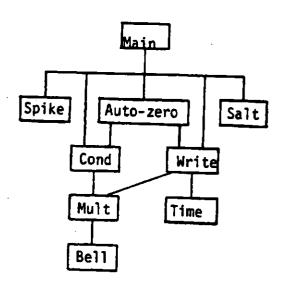
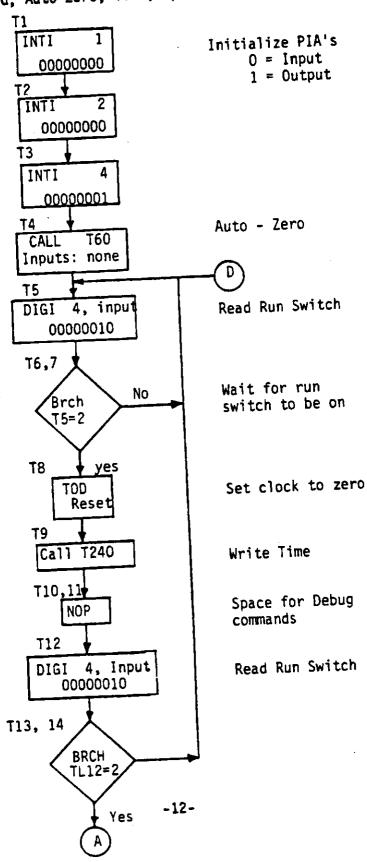


Figure A.1 Task Allocation For Subroutines

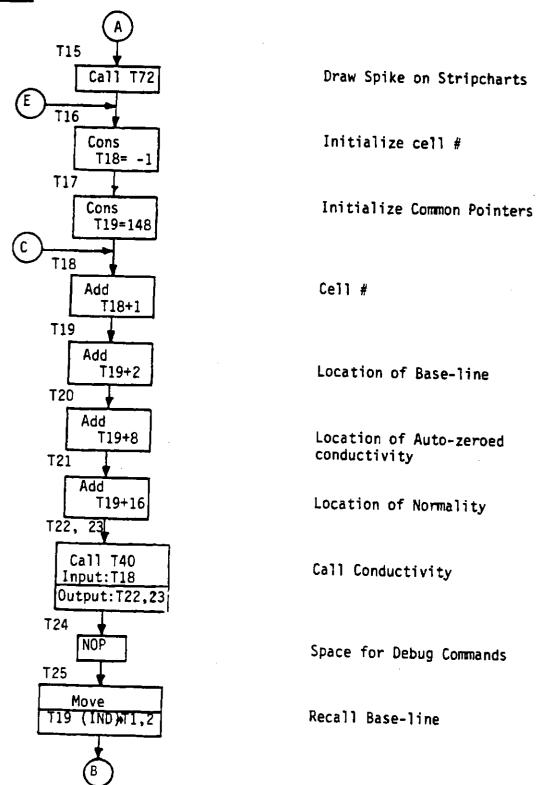
Figure A.2 Basic Call Hierarchy

MAIN

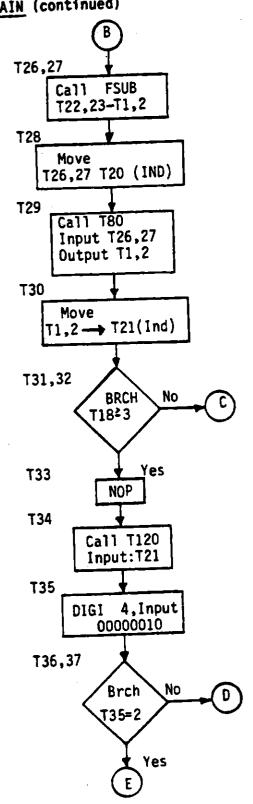
Calls: Cond, Auto-Zero, Time, Spike, Write, Salt



MAIN (continued)



MAIN (continued)



Subtract baseline (auto-zero)

Store Auto-zeroed conductivity

Call Salt

Store Normality of Salt

Loop over Cell #

Space for Debug Commands

Write Out Salt Normality and Drive Stripcharts

Read Run Switch

CONDUCTIVITY

Purpose:

Read HLAD and Convert to Conductivity

Inputs:

Cell #

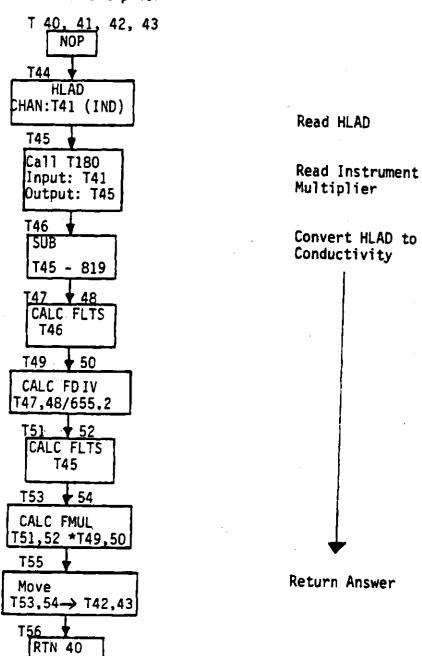
Outputs:

Conductivity

Called by: Main, Auto-Zero

Calls:

Multiplier



AUTO-ZERO

Purpose:

To store conductivity of water in order to subtract

its contribution from the salt solution.

Inputs:

None

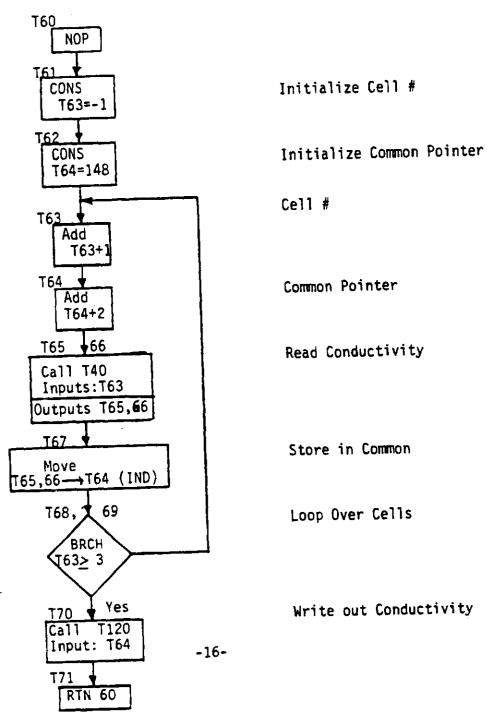
Outputs:

None

Called By: Main

Calls:

Write, Cond



SPIKE

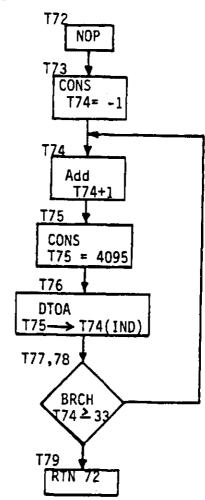
Purpose: To mark stripcharts at beginning of run.

Inputs: None

Outputs: None

Called By: Main

Calls: None



SALT

Purpose: Computes normality of salt solution given

conductivity.

Input:

Conductivity

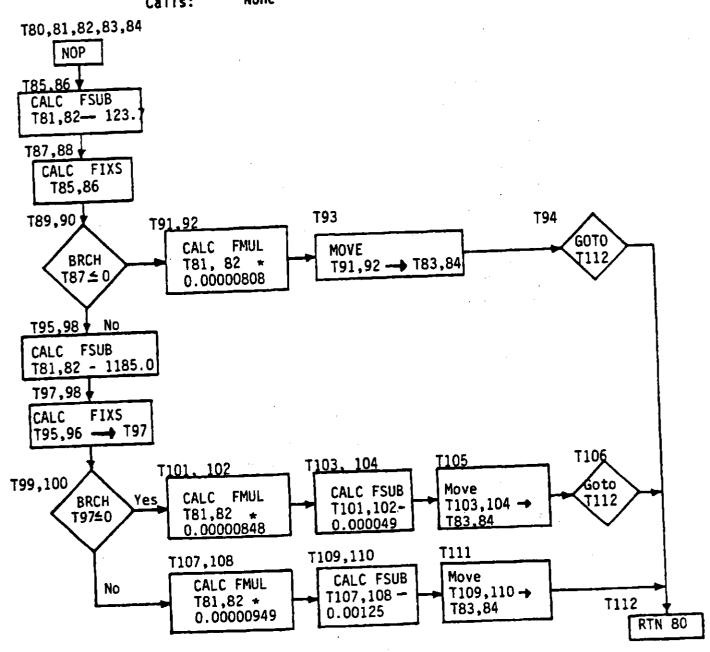
Output:

Normality of Salt Solution

Called By: Main

Calls:

None



WRITE

Write out time and data. Drive DTOA convertors for Purpose:

stripcharts.

Input:

Location + 6 of common data to write out.

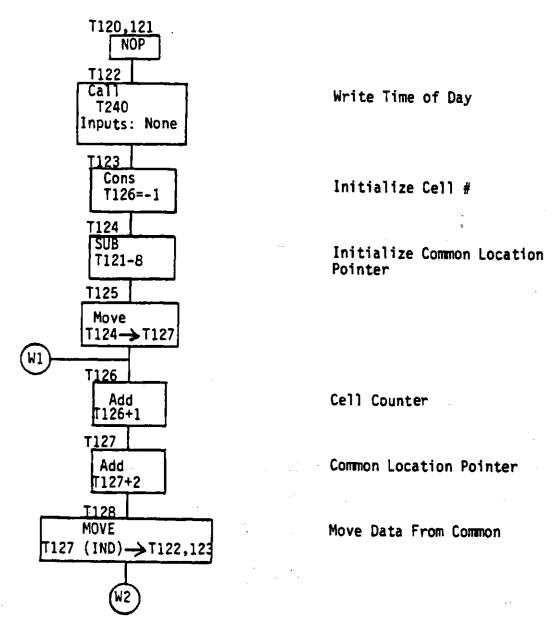
Output:

None

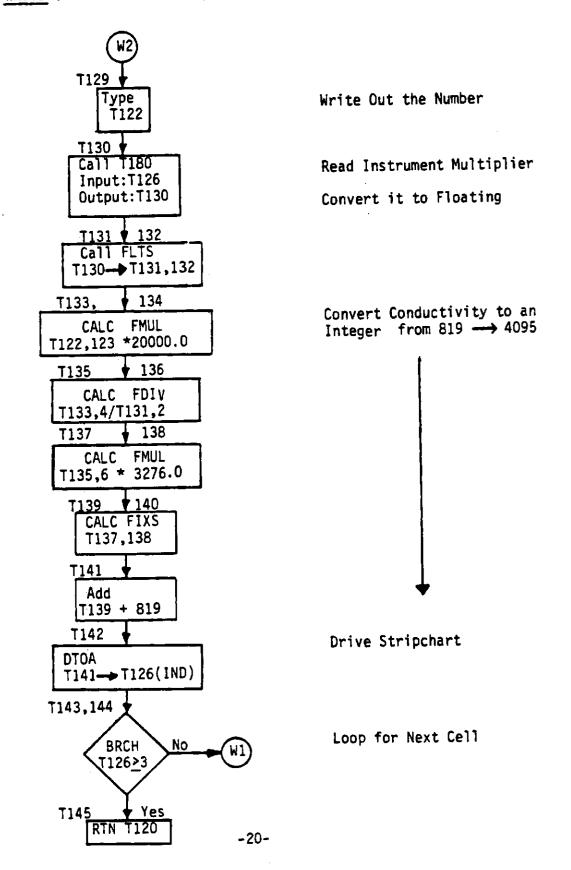
Called By: Main, Auto-Zero

Calls:

Time, Mult



WRITE (continued)



COMMON

Purpose:

Stores data in a manner accessible to all subroutines.

<u>T150</u> → T179	Locations	Four Floating Point Numbers (2 Tasks Each)
NOP	T150 - 157	Conductivity of Water (for Auto-Zero)
	T158 - 165	Auto-Zeroed Conductivity
	T166 - 173	Normality of Salt Solution

MULTIPLIER

Purpose:

Reads range of conductivity monitors.

Input:

Cell #

Output:

Instrument Multiplier (100,1000,10000,0)

Called By:

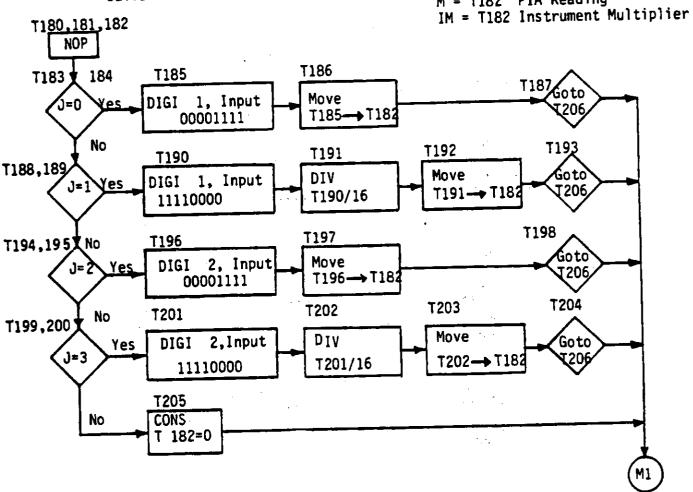
Cond, Write

Calls:

Bell

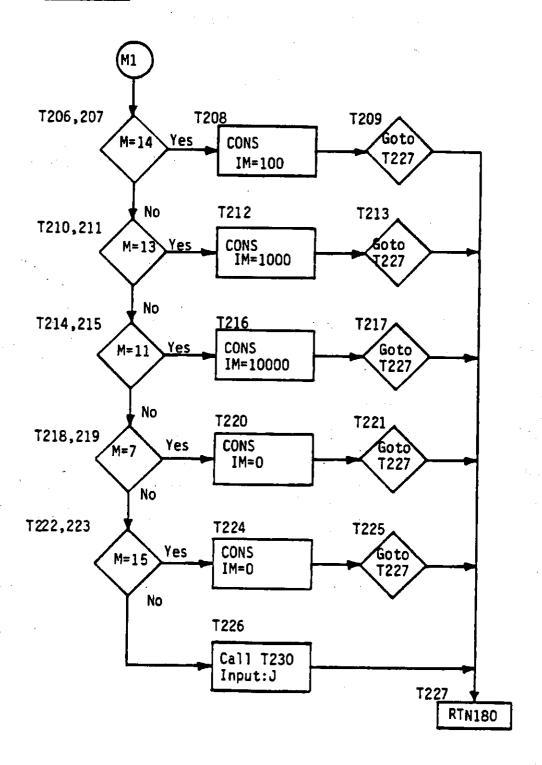
J = T181 Cell #

M = T182 PIA Reading



T183 - T205 Determine the instrument setting for Cell # J.

MULTIPLIER (continued)



T206 - T227 Assigns the appropriate Instrument Multiplier or rings bell if error in wiring.

BELL

Ring bell and print error message if Instrument Multipliers are wired wrong. Purpose:

Inputs:

The Cell # in Error

Outputs:

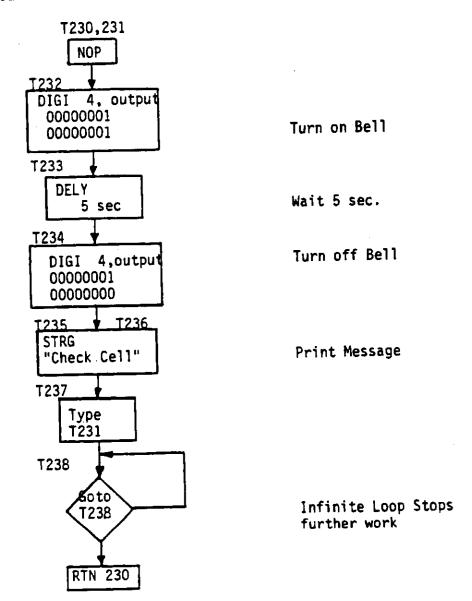
None

Called By:

Mult

Calls:

None



TIME

Purpose:

Print out the time on the system clock.

Inputs:

None

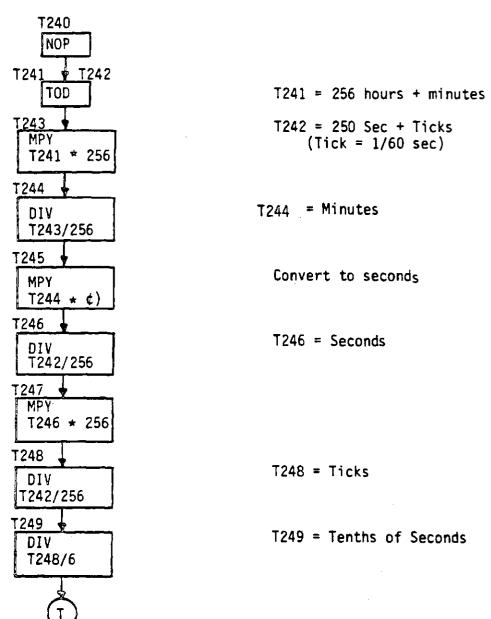
Outputs:

None

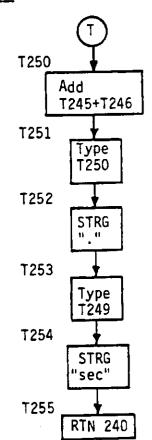
Called By: Main, Write

Calls:

None



TIME (continued)



Total Seconds