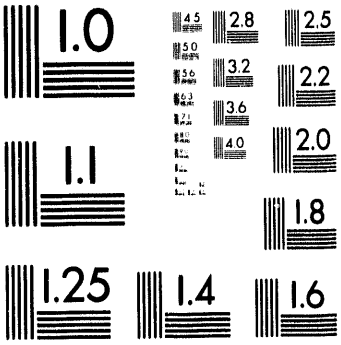
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1 of 1

WINCO-1127 UC-510

DEVELOPMENT OF A CONTINUOUS PROCESS FOR ADJUSTING NITRATE, ZIRCONIUM, AND FREE HYDROFLUORIC ACID CONCENTRATIONS IN ZIRCONIUM FUEL DISSOLVER PRODUCT

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Acknowledgement

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SUMMARY

In the Fluorinel Dissolution Process (FDP) upgrade, excess hydrofluoric acid in the dissolver product must be complexed with aluminum nitrate (ANN) to eliminate corrosion concerns, adjusted with nitrate to facilitate extraction, and diluted with water to ensure solution stability. This is currently accomplished via batch processing in large vessels. However, to accommodate increases in projected throughput and reduce waste production in a cost-effective manner, a semi-continuous system (In-line Complexing (ILC)) has been developed. The major conclusions drawn from tests demonstrating the feasibility of this concept are given below.

The ILCM consistently approached the control target of 0.045 M free HF (error range -14% to 8%.) In addition, the necessary residence time for the reaction appeared to be between 0.3 and 0.67 minutes. The titanium probe used to amperometrically and continuously the HF concentration had an adequate life for development tests. Additional work is suggested to develop a probe that will have an adequate life for plant operation.

The tests indicated that two points for ANN addition are adequate. During the last five runs of the system, complexing was essentially complete through the use of the first ANN addition point only. However, the second addition system may be required in the event of extreme upset conditions or failure of the first addition system.

The ILCM also consistently approached its nitrate (error range -7 to 13%) and zirconium (Zr) (error range -16 to 12%) targets in the absence of equipment failure. The primary problem with attaining these targets was accurate reagent addition. Another source of error in the final Zr concentration was the density equation used to estimate the initial Zr concentration. Since these errors are hardware related, and the equipment which will be used in the plant will not be identical to that used in the mock-up, it will be necessary to test the actual plant hardware in the integrated pilot plant to ensure that these problems can be resolved.

A test of the system carried out with a mixture of ANN and nitric acid demonstrated the applicability of the process to complexing Zr dissolver product with Al fuel dissolver product (coprocessing).

The control system was tuned to provide stable operation during the last run of the system. In addition, the system was able to attain the HF target within 10 minutes with a decay ratio of 0.3 and a period of oscillation of 3.5 minutes after the initial start-up disturbance. It is possible that smoothing of the incoming HF probe signal coupled with derivative control could improve the system's ability to deal with disturbances. In addition, the magnitude of the initial start-up disturbance could be reduced via closer tuning of the feedforward contribution.

The system tests were too short to obtain any information on the corrosive effects of HF/HNO3 mixing in the system.

1.0 INTRODUCTION

In the FDP upgrade, excess hydrofluoric acid in the dissolver product (ZrDP) must be complexed with aluminum nitrate to eliminate corrosion concerns, adjusted with nitrate (nitric acid) to facilitate extraction, and diluted with water to ensure solution stability of zirconium fluoride. These operations are integrated, that is, adjustment of concentration of one affects the other. The CPP currently uses a batch process for this purpose. The large vessels, instrumentation, and cell space associated with this complexing technique are expensive. The cost savings associated with the reduced requirements for cell space which would result from continuous complexing is estimated to be $60,000,000. In addition, the current batch process is not geometrically safe by design and requires the use of nuclear poisons for criticality control resulting in increased waste volumes. The batch process also involves mixing and sampling as separate steps, with significant dead time associated with sample result turnaround. In addition, the reagent additions are imprecise, resulting in a frequent need for adjustment, increasing waste volume and delaying plant operation.

In order to solve these problems, the authors developed a continuous in-line complexing system to complex the zirconium dissolver product (ZrDP) as an alternative to batch processing. By using a Ti probe to detect free HF concentrations[17], along with equations which enable the extent of complexing to be estimated, the In-line Complexing System (ILCS) is able to use a feedforward/feedback cascaded control scheme to continuously attain the free HF target concentration. The system also uses feedforward control to simultaneously attain Zr and nitrate target concentrations.

2.0 PROGRAM OBJECTIVES

- Develop a process and control system which continuously attains the HF, Zr, and nitrate targets required during batch complexing.
- Determine the residence time required for the complexing reaction.
- Determine the reliability and operating characteristics of the probes.
- Demonstrate the system's applicability to coprocessing.
- Investigate the corrosion effects of HF/Nitric acid mixing in the system.

Table 2.0-1 lists the test conditions and targets used for all of the ILCM system runs.

3.0 EQUIPMENT

The mock-up was installed in the Geometrically Favorable Dissolver (GFD) module in the Low Bay Laboratory and used the Fountain Dissolver Mockup (FDM) recycle vessel (operating capacity of 195 liters) as the uncomplexed ZrDP

TABLE 2.0-1

EXPERIMENTAL CONDITIONS AND TARGETS

Run Number (Date)	ZrDP Flowrate (liters/min)	ZrDP Temp. °C	Free HF Target* (molar)	Zr Target (molar)	TSL* Zr Analysis Error (±%)	Nitrate Target (molar)	RAL** NO_3 Analysis Error (±%)
ILCM001 (7/18/90)	2-2.25	22	0.045	0.5	9	2.0	10
ILCM002 (7/25/90)	1.5	22	0.045	0.5	9	2.3	18
ILCM003 (8/30/90)	1.5	21	0.045	0.5	10	2.3	11
ILCM004 (9/20/90)	1.0	70	0.045	0.5	9	2.4	11
ILCM005 (11/20/90)	1-1.5	75-83	0.045	0.5	15	2.4	6
ILCM006 (3/21/90)	1-1.5	77-82	0.045	0.5	3	2.4	6
ILCM007 (5/21/91)	ABORTED						
ILCM008 (5/29/91)	1-4.0	22	0.045	0.5	10	2.4	7

* Calculated at 35°C; the measured target value at operating temperature is dependant on the temperature.

** Based on two standard deviations.

supply. The recycle vessel was equipped with density, level and temperature monitoring instrumentation. These readings were used to estimate the zirconium concentration in the recycle vessel prior to transfer from the tank into the in-line complexing system. This concentration was then used in conjunction with the flow rate from the ZrDP supply pump in determining the feedforward contribution to the flow rates for the other reagents.

A sketch of the In-line Complexing Mock-up is shown in Figure 2.0-1. The system consists of a supply vessel containing the uncomplexed dissolver product, a series of six 4" I.D. Kynar tubes connected by 1" Kynar tubing, recycle loops (see Figure 2.0-2), HF probes (see Figure 2.0-3), thermocouples, metering pumps, in-line mixers, complexing reagent addition lines, and a final collection vessel.

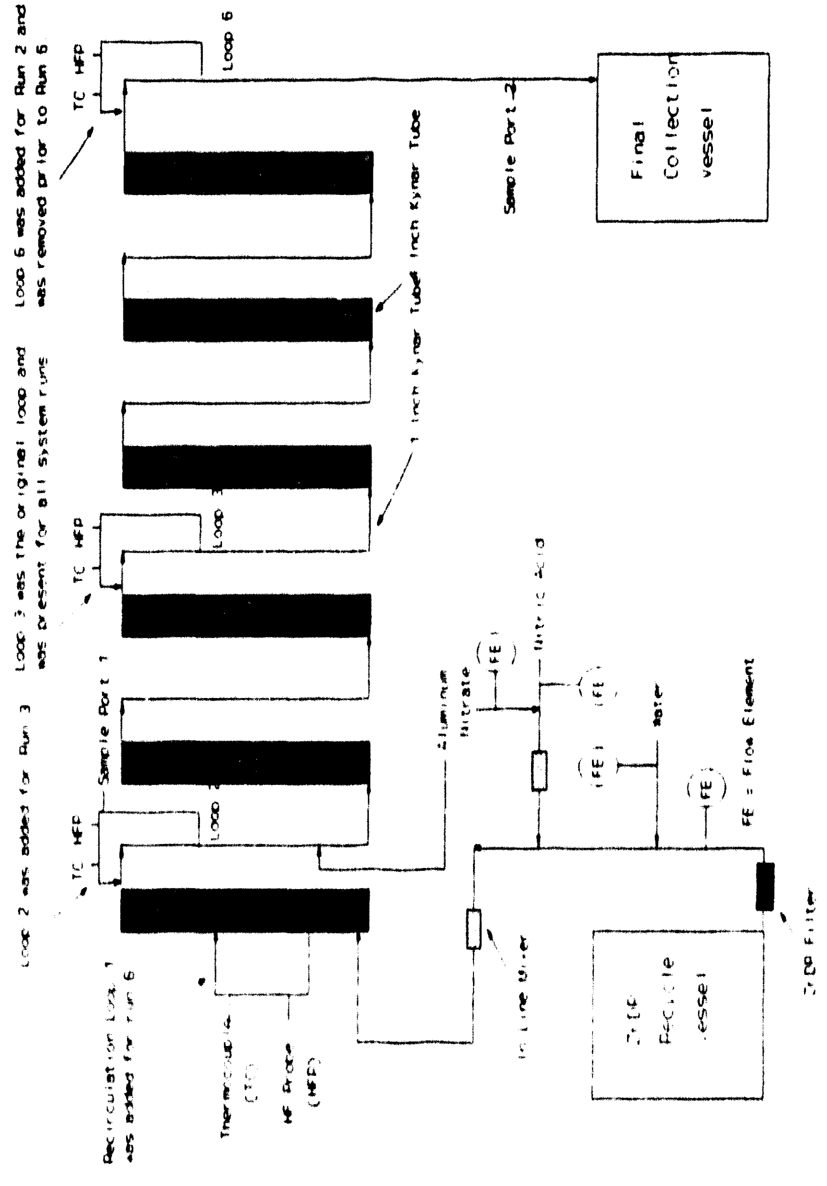


FIGURE 2.0-1 ILCM SYSTEM

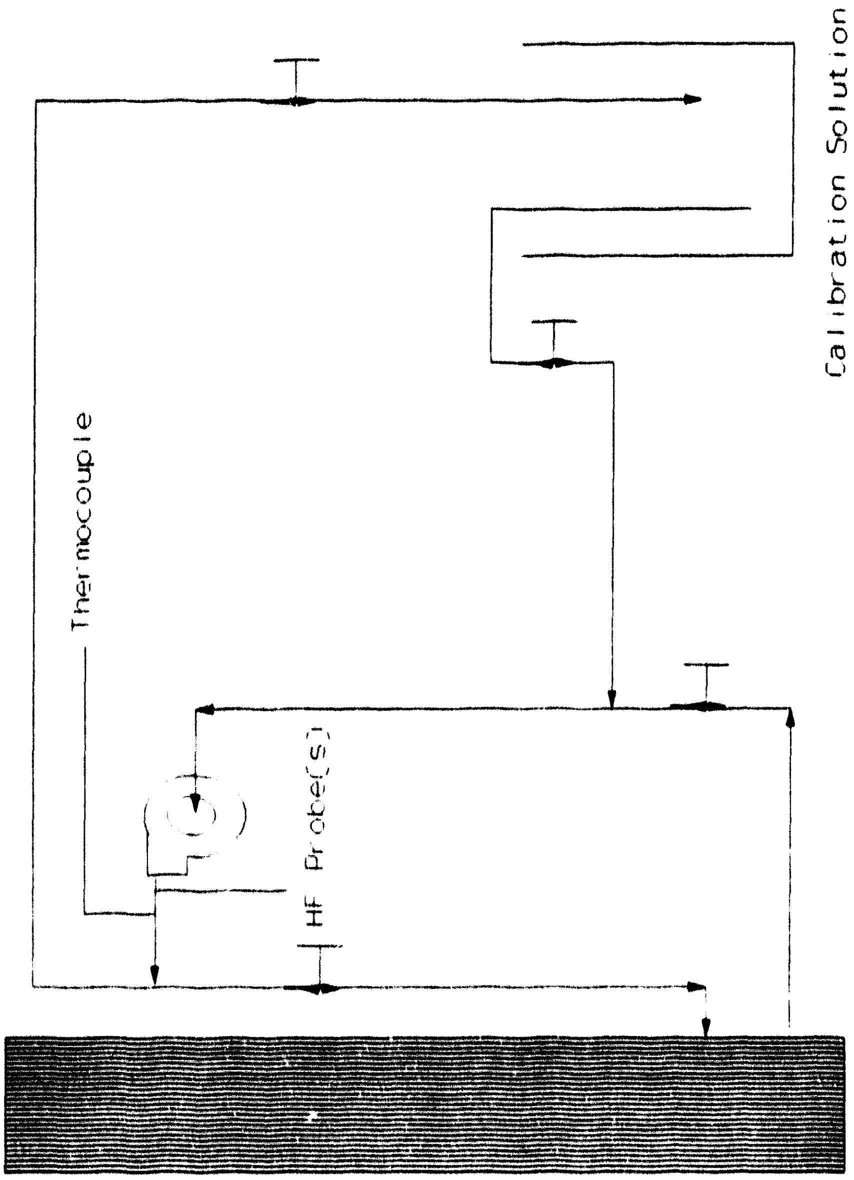


FIGURE 2.0-2 HP PROBE RECIRCULATION LOOP

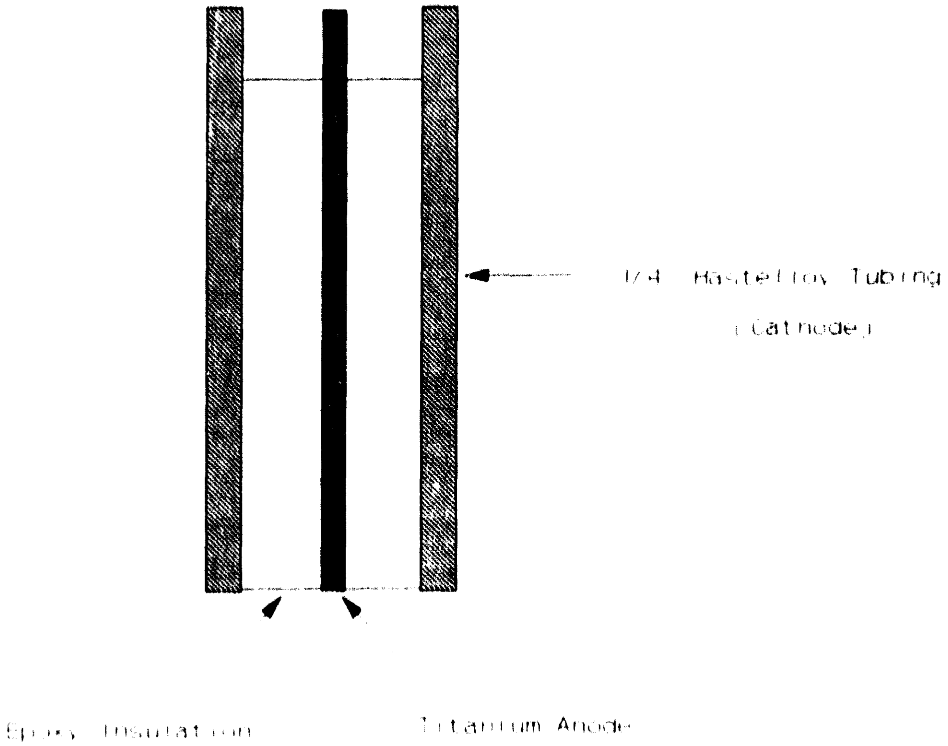


FIGURE 2.0-3 HF PROBE

The Fluorinel Pilot Plant (FPP) reagent tanks for water (FPP-VES-107), 13M nitric acid (FPP-VES-108), and 2.2M aluminum nitrate (ANN) (FPP-VES-109) were used as feed tanks for the complexing reagents used in the first addition. ANN for the second addition was obtained from a carboy. The complexing reagents were added to the system using peristaltic pumps which were feedback controlled using turbine flowmeters.

During the initial complexing step, water was added first, followed by a mixture of ANN and nitric acid. The use of a static mixer after each reagent addition ensured adequate mixing of the streams. After the initial addition of complexant, a series of free HF measuring probes, in conjunction with thermocouples to compensate for temperature effects, followed the reaction. The information provided by the probes was used to determine the amount of ANN required at the first and second addition points. After the second addition was made, one more reading was taken downstream to determine if the product was adequately complexed. The final product was collected in a 770 liter stainless steel vessel equipped with density and level monitoring instrumentation.

4.0 CONTROL SYSTEM

4.1 Current Status

The In-line complexing control scheme is substantially complete. Individual reagent flow control loops are an essential feature of the process. This measurement is currently performed by turbine flowmeters in the mockup. This type of meter has an inherent bias with high viscosity fluids, which varies with rate. Currently there is no compensation for this effect. Flowmeter characterization studies are planned which will allow the control system to correct the Figure 2.0-1 ILCM System flow signals. The plant may use another type of flowmeter. The control scheme would work with any accurate meter. The most recent test was performed at a 4 liter/min dissolver product feedrate. This required larger flowmeters and higher capacity pumps than previous runs, but the same control system gave good performance with the modified equipment. The current configuration uses 7 interacting PID control loops and an additional on-off loop for heating the feed. A setpoint track/run feature is used to provide bumpless startup of cascade loops (see Appendix A).

4.2 Development History

The MICROMAX process control system was selected to control in-line complexing. It was already installed for use with the Fluorinel Pilot Plant and GFD dissolver. It had analog I/O, PID loop control, and logic and calculational ability that made it a logical choice for in-line complexing. The original control system for in-line complexing was strictly feedforward. The first aluminum nitrate addition was based on equations used for vessel adjustment based on [F], [Zr], SpG and temperature of the feed solution. A Ti probe measured [HF] downstream of the first reagent addition point and calculated the additional ANN to be added downstream. Additions of nitric acid and water are calculated by a running mass balance to meet product targets and were added near the first ANN location. This system gave acceptable results but required constant attention and frequent adjustment by a skilled chemist.

The development of In-line Complexing required precise flow control. Metering pumps are used for all feeds to the process. It was found that these pumps did not have linear output, so a characteristic response curve was generated for each pump and programmed into the MICROMAX. This improved pump response, but was not entirely satisfactory as the back pressure on the pump affected flow. Runs 1 and 2 were performed with this configuration.

A feedback loop was added to correct the initial ANN addition based on the probe response. At the same time orifice flowmeters were added to the ANN, nitric acid, and water additions. The flowmeters provided measured signals for closed loop control of these flows. It was especially important to be able to add measured amounts of nitric acid and water since the product concentrations could not be measured directly. The feed solution was too corrosive to measure with available orifice meters so its flowrate was measured by level depletion. This gave an accurate value in theory, but was the difference of two noisy values over a short time base. Numerical damping gave some improvement, but the results were still marginal.

The ANN flow loop became the inner loop of the ANN control system. The outer loop was a combination of the feedforward calculated value and a feedback PID function operating on the probe signal. The feedforward contribution undercomplexed the feed solution and the feedback signal brought the free [HF] to the target setpoint. The piping arrangement between the addition point and the probes included a tube to allow residence time for the reaction to reach equilibrium. An adaptive tuning constant was selected to deal with the variable time lag in the outer, probe-controlled loop. The inner loop was the orifice meter-pump flow controller which received a rate setpoint from the outer loop.

The process performed well during run 3 with the controller correcting upsets. The time lag due to the solution holdup prior to the probe location resulted in slow oscillations which took about 30 minutes to dampen out. The final ANN addition system was operated in the same feedforward mode that was used before. This system did not add ANN except during upsets to the process because the first loop controlled to setpoint. The final ANN addition system could be considered a safety backup for this reason. The probe readings used to control the system had a bias that caused high readings so the final product was slightly overcomplexed (free [HF] = 0.040 M vs a 0.045 M target) during the first run with this control scheme, but still within acceptable limits. The control system is expected to work as well with unbiased probe readings. The primary weaknesses of the system at this stage of development were rate control by level depletion and zero bias on the orifice meters due to difficulty bleeding the meters.

In-line run ILCM004 demonstrated the feasibility of this system for co-processing with Aluminum dissolver product. A mixture of ANN and nitric acid was used as a complexing reagent. This required modification of the nitrate and ANN mass balance equations, and rewiring and relocation of other equipment. Run 4 succeeded in demonstrating the flexibility and adaptability of this process.

Later in the development work, turbine meters were added to measure all flows except final ANN to improve accuracy. A suitable material turbine meter was found for the feed solution, allowing closed loop feed control which was a great improvement over the level depletion method. The MICROMAX control logic was modified to accept the pulse frequency signals from the turbine meters. An additional control loop was added for heating the feed tank. Run 5 was performed with this configuration.

A new probe location was added to reduce the time lag in the probe loop, thereby improving the response of the controller. The new location gives about 30 seconds residence time at typical flows. This probe provides the signal for a third controller in the ANN cascade added between the other two. The new loop controls the flow loop and in turn is controlled by the original probe loop, making this a triple-effect cascade controller. If the reaction is incomplete at the new probe location, the outer loop trims the setpoint to compensate for it. This improved response time by a factor of 4 during run 6. A turbine flowmeter was also added to the final ANN flow during this run. It was configured as the cascaded inner loop of the final ANN demand. The primary control system worked so well that the demand for final ANN was infrequent and the rate was low. The flow loop was determined to be unnecessary and the configuration was returned to direct control of final ANN.

The most recent in-line complexing runs have been performed at the plant design feed flowrate of 4 liters/min. Higher capacity flowmeters and extra pump heads for greater capacity were required for some reagents. Run 7 was performed with this configuration until terminated as the result of flowmeter plugging. After this, a filter was installed on the feed stream to prevent flowmeter plugging and run 8 was performed successfully.

The in-line complexing control system combines complex feedforward/feedback loop control with chemical equilibrium calculations, a feedforward correction calculation, mass balance equations, and a special probe reading converted to a free [HF] reading all in a real-time continuous process.

5.0 RESULTS AND DISCUSSION

5.1 HF Target Attainment

As discussed in the controls section, the method of HF target attainment gradually evolved with the development of the control system. While initial attempts entailed only feedforward control, the final system involved the use of feedforward/feedback cascaded control loops.

Figure 5.1-1 is a plot of the percent deviation of the final free HF concentration (which represents all of the solution gathered during an experiment) from its targeted value of 0.045 for all of the system runs. The impressive system performance during run 1 is deceptive in that the system was not self-regulating. During this run it was noted several times that the free HF reading at tube 3 was too high (refer to Figure 2.0-1). As a result it was necessary to change the parameters used to calculate the ANN flow five times.

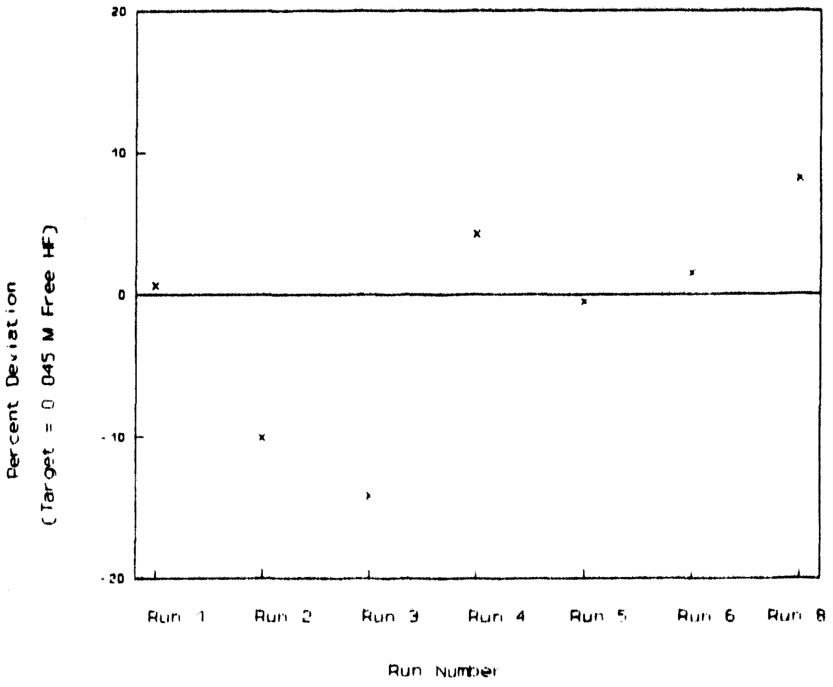


FIGURE 5.1-1 PERCENT DEVIATION OF COMPOSITE FREE HF CONCENTRATION FOR ALL ILCM RUNS

Thus, the system required constant operator intervention and it was merely coincidental that the deviations observed during the run averaged out to the proper overall concentration. The same control scheme was used during the second run with the goal that a better initial estimate of the HF concentration would allow the system to be self-regulating. The data point for run 2 in Figure 5.1-1 shows that the system's performance was worse than the first run.

At this point, it was apparent that a feedforward/feedback control scheme would be better suited to attaining the target without requiring operator intervention. In preparation for this run another recirculation loop was added at the top of the first column as is shown in Figure 2.0-1. The intended goal was to decrease the lag time between the ANN addition and the subsequent probe response. While the control scheme seemed to work as can be seen from Figure 5.1-2, the poor performance of the system with respect to

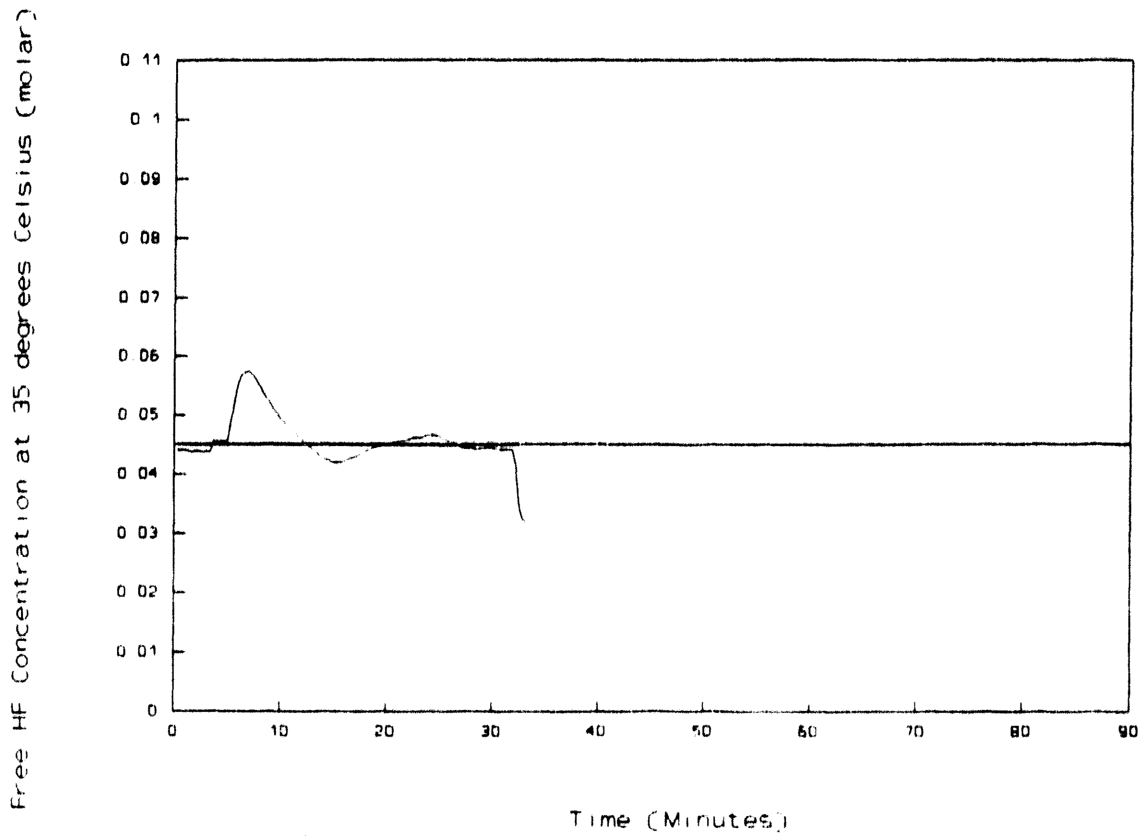


Figure 5.1-2 FREE HF READING AT TUBE 1 DURING RUN 3 VERSUS TIME

free HF concentration (shown in Figure 5.1-1) is attributed to improper calibration of the probe at the start of the run.

The same control scheme was used during run 4, with the exception that a mixture of HNO3 and ANN was used in place of ANN to demonstrate the ability of the system to use the product from the aluminum dissolvers during co-processing. This required changing the nitrate mass balance equations to account for the difference in nitrate concentration between ANN and the mixture used during this test. Although the test was a success from this standpoint, the initial estimate of the HF/Zr ratio was too high, and resulted in the feedforward contribution providing more of the ANN/HNO3 mixture than was needed to complex the product solution to 0.045 M free HF. At this time the control system had not been set up for two-sided control, and although the feedback portion of the control scheme could compensate for too little complexant, it could not compensate for an excess of complexant. Thus, it was necessary to change the HF/Zr ratio several times in order to achieve the 0.045 setpoint.

Prior to Run 5, the control system was changed to allow for two-sided control (see discussion in APPENDIX A). Figure 5.1-3 shows the damped approach to set point which resulted from the control scheme after the initial start-up disturbance. In addition, this Figure shows that the peaks and valleys resulting from this control scheme roughly compensate for each other, resulting in an average product composition which was close to the desired value as is shown in Figure 5.1-1.

In preparation for run 6, the system was modified by placing a recycle loop closer to the addition point for ANN as shown in Figure 2.0-1. It can be seen from Figure 5.1-4 that the system modifications resulted in a significant decrease in both the period of oscillation and the decay ratio. In addition, the final product was close to the desired value as is shown in Figure 5.1-1.

During run 7 an attempt was made to operate the system at the design basis feed flow rate of 3.5 lpm. Problems were encountered during run 7 with solids plugging the ZrDP meters and the run had to be aborted[1] . However, the addition of a filter enabled the system to be successfully operated at the design basis flowrate during run 8. The fact that the final HF concentration shown in Figure 5.1-1 is above the targeted value is attributed to the system upsets during run 7 resulting in undercomplexed ZrDP going to the final collection vessel (the solutions from runs 7 and 8 were collected in the same batch.)

It can be seen from Figure 5.1-5 that further improvement in system control was realized through the decrease in lag time which resulted from operating at

Figure 5.1-3 Free HF Reading at Tube 1 During Run 5 versus Time.

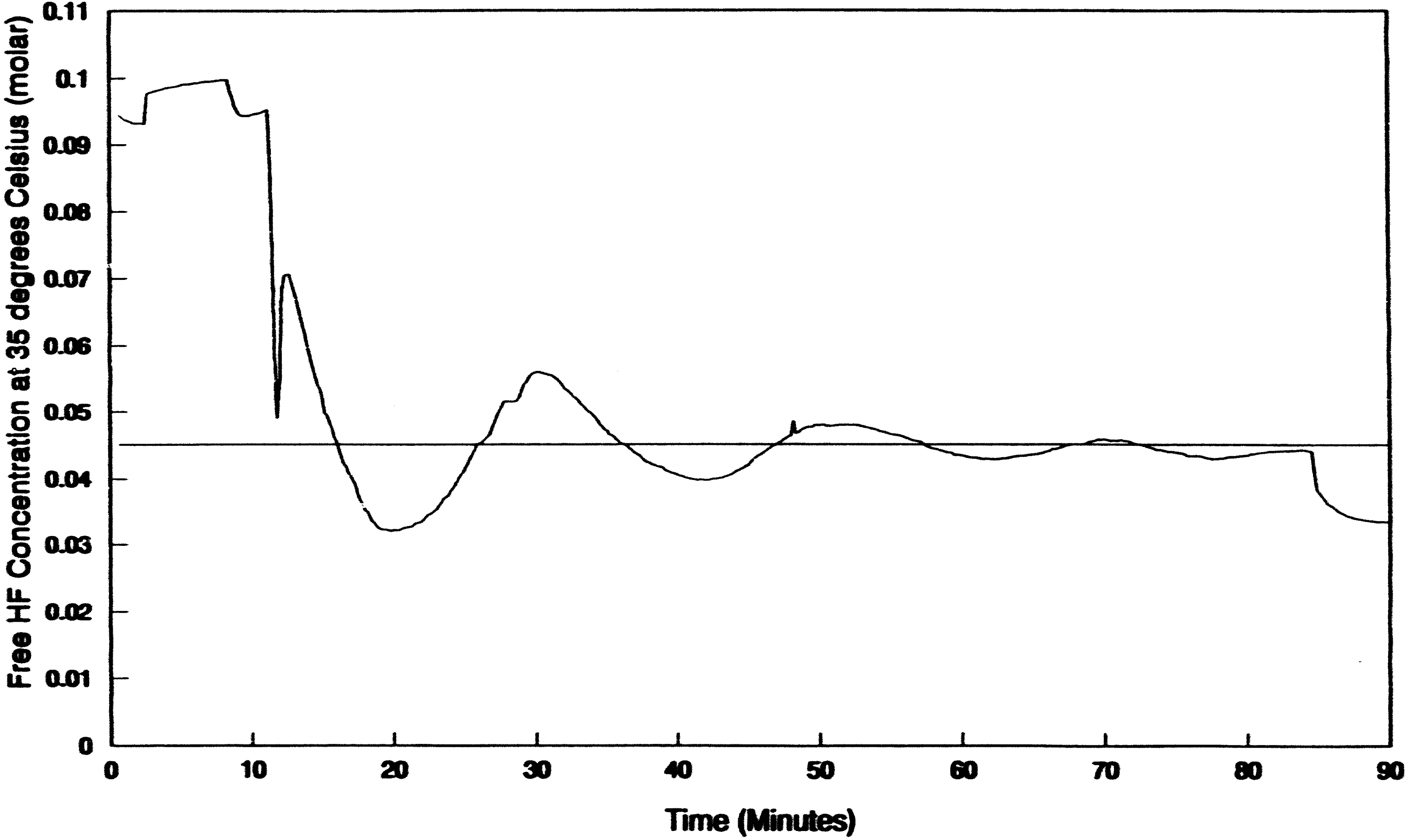
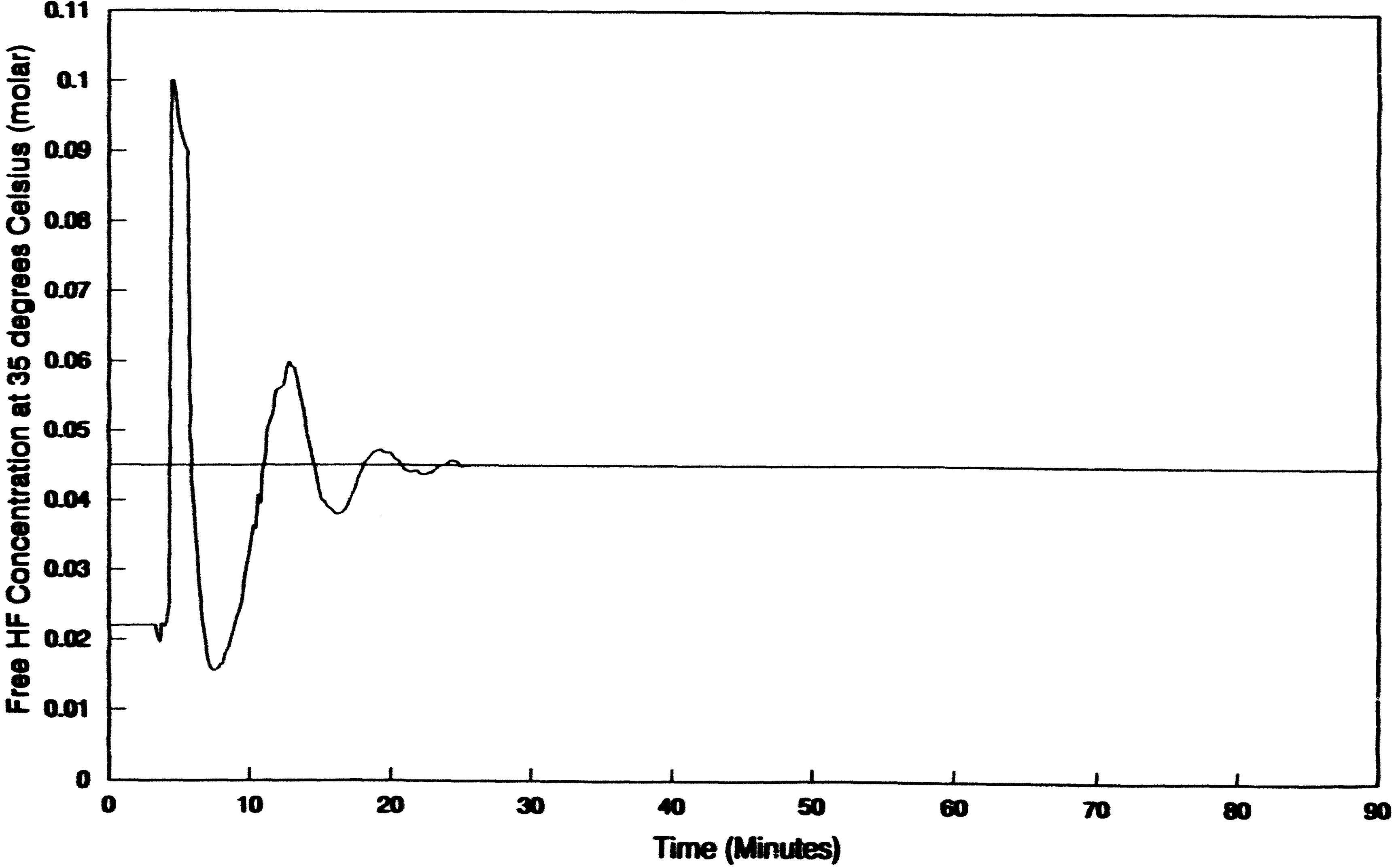


Figure 5.1-4 Free HF Reading at
Tube 1 During Run 6 versus Time.



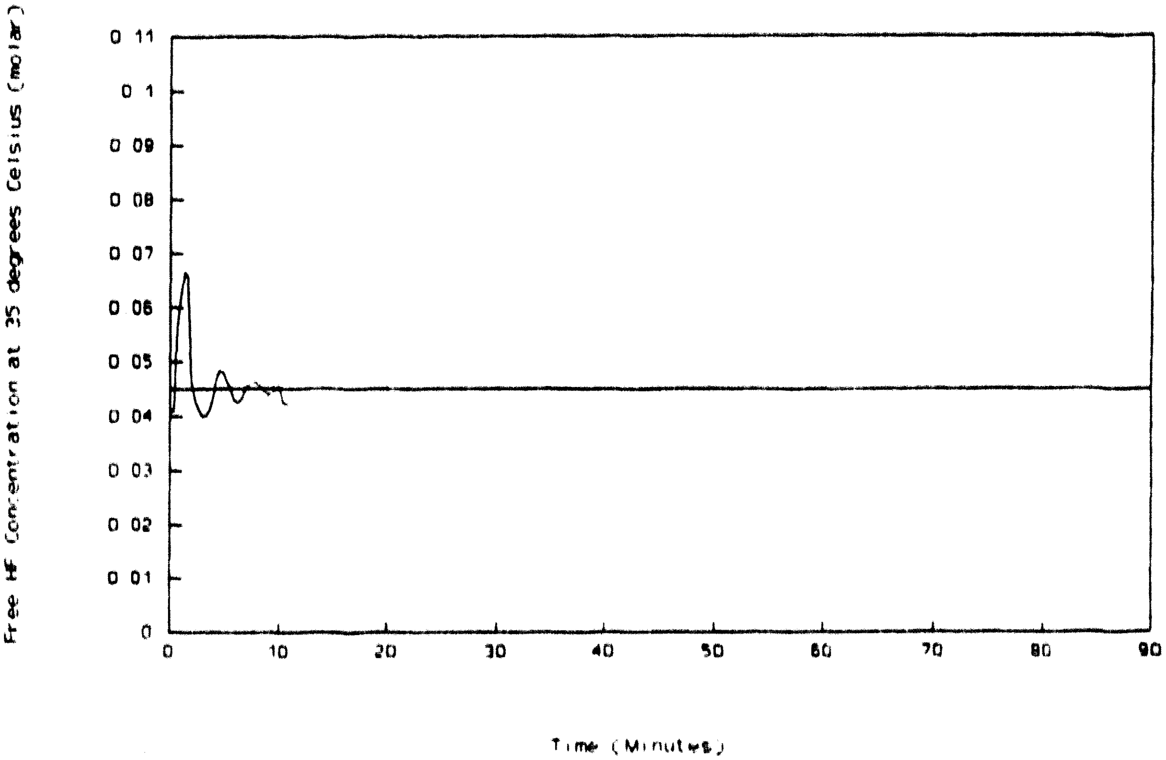


FIGURE 5.1-5 FREE HF READING AT TUBE 1 DURING RUN 8 VERSUS TIME

the higher flow rate. This result indicates that system control at lower flow rates could be improved by moving the first recirculation loop further upstream. A summary of the periods of oscillation and decay ratios for runs 5, 6, and 8 is contained in Table 5.1-1.

In addition to obtaining information on the performance of the control system during runs 6 and 8, the apparent residence time for the complexing reaction was also bracketed. This was accomplished by comparing the probe reading at the first recirculation loop (at the bottom of column 1) to the reading at the second recirculation loop (at the top of column 1). A lower reading at the top of the column would imply that the solution did not have enough time to react before it reached the first loop and was consequently being over-complexed. A comparison of the data for these two probe locations indicated that they were in close agreement during run 6[2], however there was a consistently low bias in the reading at loop 2 relative to loop 1 during run 8[3]. (Loop 3 was in close agreement with loop 2 indicating that this was not

TABLE 5.1-1

Summary of Decay Ratios and Periods of Oscillation for
Runs 5, 6, and 8.

Run Number	Period of Oscillation (minutes)	Decay Ratio
5	18	0.8
6	7	0.6
8	3.5	0.3

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simply an erroneous probe reading.) The estimated reaction residence time at the flow rates observed during run 6 was approximately 40 seconds (This value differs from that given in DSH-03-91 due to the failure to account for the 10 seconds residence time in the 1" line leading up to the first 4" tube.) The estimated residence time at the flow rates observed during run 8 was approximately 17 seconds. Thus, it appears that between 17 and 40 seconds were required for the complexing reaction to attain equilibrium under the observed experimental conditions. However, it is important to note that the temperature at column 1 was 41 degrees Celsius during run 6, whereas it was closer to 28 degrees Celsius during run 8. While it is probable that the reaction will be slower at the lower temperature, this effect has not been quantified.

5.2 Probe Operation and Reliability

While Figures 5.1-3 through 5.1-5 indicate that the control system will control to set point, there is still uncertainty associated with the probe's ability to provide an accurate reading. Probe signals have been observed to drift apart by as much as 11% during system runs and complete probe failures have also been noted[4]. Subsequent observation of the probes indicated that the epoxy used to insure one-dimensional attack on the Ti anode was failing from exposure to the process solution. While this is one of the more probable modes of probe failure, a fundamental understanding of the reaction mechanism and mass transfer phenomena associated with probe operation is still lacking. This information should be obtained prior to building the process around the probe. (A collaborative research project with Montana State University for this purpose had been initiated at the time this report was being written.) Figure 5.2-1 compares the computer program[5] calculated free HF concentrations based on the RAL's analysis for 30 samples obtained during runs of the system to the probe readings. Although the significant scatter has been attributed to error in the RAL's analysis[6], this plot indicates that the program may not be reliable for independently confirming the probe readings.

An unexpected and interesting result was observed during the fifth run of the ILCM. It was noted that a probe was not responding to the calibration solution during the beginning of the run so it was disconnected. Later inspection of the probe indicated that the probe had corroded back into the epoxy approximately 1/8th of an inch. This was estimated to be between one and two orders of magnitude greater than the corrosion observed for the other probes which had remained connected to the bias box during the experiment. This was surprising in that it implied that making the Ti anodic decreased rather than increased its corrosion rate. It was suspected that this was the result of a change in the dissolution mechanism upon applying the bias voltage to the Ti. A literature search revealed that Ti forms a passive oxide film upon the application of an anodic bias voltage in HF/HNO3 solutions[7]. This is attributed to a passivation reaction during which a dilute phase of adsorbed isolated metal hydroxide ions is transformed into a continuous oxide layer upon the application of sufficient potential[8]. The TiO2 film is destroyed when the oxygen atoms (double bonding) are replaced by fluorine atoms (single bonding) destroying the 2-dimensional network protecting the metal[9]. The reaction by which this is thought to occur is given below[10]:

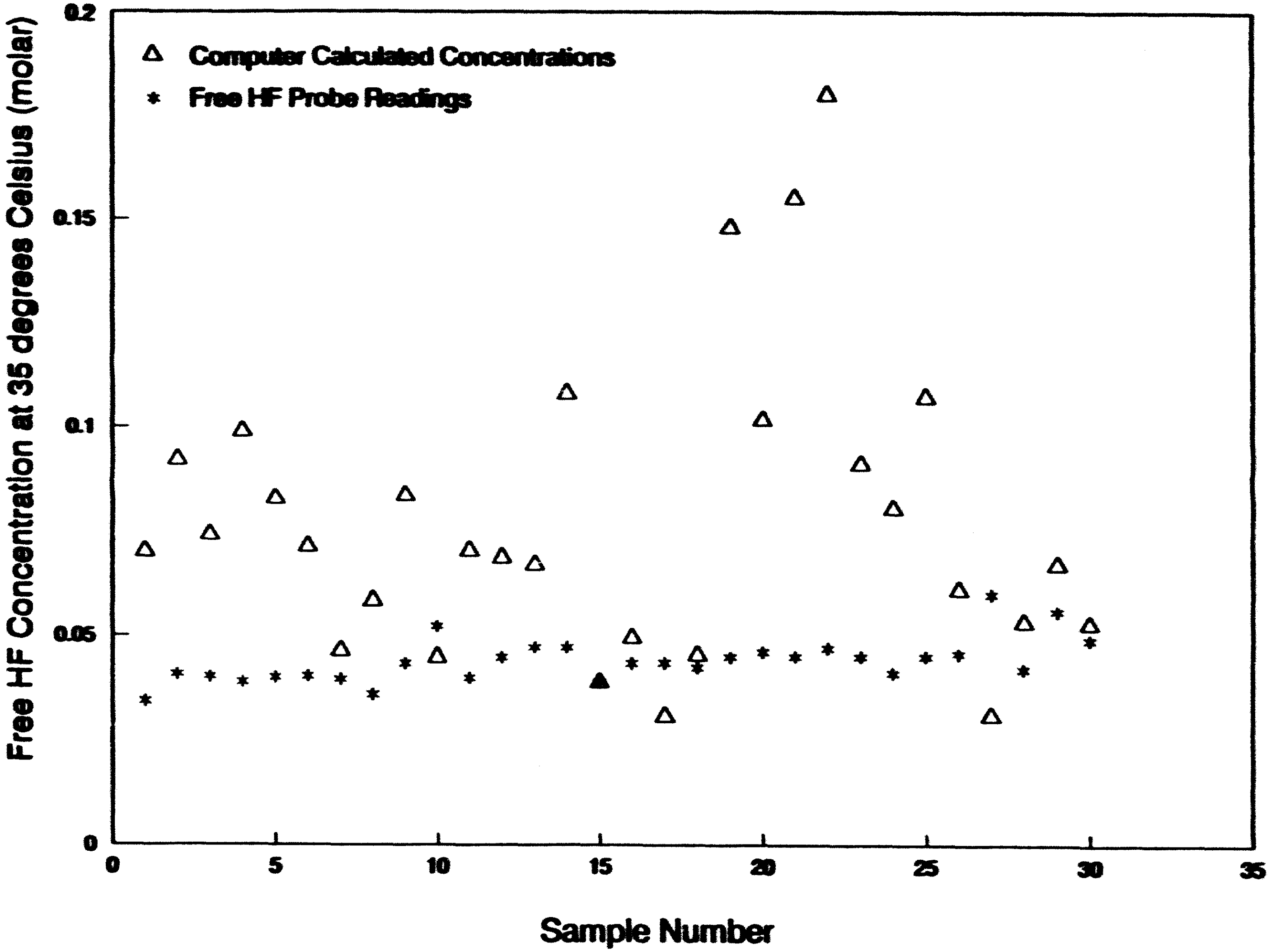
$$TiO_2 + nHF \dashrightarrow (TiF_n)^{(n-4)-} + (n-4)H^+ + 2H_2O$$

Observation of the probes after exposure to process solution under an optical microscope indicated that the metal was pitted. Thus, it is thought that the current produced by the HF probe results from HF attack on the TiO2 film and its subsequent regeneration. It is also possible that direct attack on the Ti metal occurs if the TiO2 film is completely removed. In addition, it is likely that the formation of this oxide film accounts for part of the current spike of current observed during probe calibration[11].

Unexpected probe behavior was also observed during a recirculation test intended to determine the long term reliability of the probes. Approximately 34 hours into the test the velocity of the solution past the probes was changed from 17 cm/s (Reynolds number $\simeq$ 940) to 11 cm/s (Reynolds number $\simeq$ 590). Surprisingly, this 38% decrease in fluid velocity resulted in no decrease in the probe readings. The fact that there was no perceptible change in probe reading despite a change in the rate at which reactant was being supplied indicates that another process could have been limiting the response of the probe. It is possible that this could be either the reaction of HF at the probe's surface or the formation of a film of corrosion products generating another layer through which the HF must diffuse prior to reacting with the surface. If the system is limited by the reaction there are no implications related to probe operation. However, if the system becomes limited by HF diffusion due to accumulation of corrosion products or other material on the surface of the probe, this would increase the response time and cause a steady downward drift in the probe reading. In fact, steady downward drifts in probe reading have been observed during recirculation tests (although this may be attributable to epoxy swelling.) One method which could be used to determine if the accumulation of material on the probe is influencing the probe response would be to expose the probe to spikes or step changes in concentration at different times during recirculation tests. If the probe is limited by the reaction the response time will not be a function of the time of exposure to the process solution, whereas if a film of material is forming as time passes which limits the reaction, the response time will be a function of exposure time.

Due to the advantages of using metal Tees instead of plastic Tees for probe placement in the plant, a comparison between the signal produced by probes

Figure 5.2-1 Comparison of Computer Calculated Free HF Concentrations to the Corresponding HF Probe Readings.



placed in adjacent stainless and Kynar Tees was made during run 8. Prior to this run there had been an unsubstantiated opinion that the probes would not work if placed in a metal Tee. Placing a probe in a metal Tee grounds it, and the original probes had always been placed in plastic Tees to avoid grounding. However, due to the insignificant resistance to current flow in metals and the small amounts of current being generated, placing a probe in a metal Tee should merely serve to increase its effective cathode area. If changing cathode area influenced the operation of the probes then the rate determining step in HF determination would not be HF consumption at the anode, but hydrogen production at the cathode. This mode of probe operation would be unacceptable for a probe used to determine HF concentrations. Fortunately, the test results indicated that there was no difference in the readings produced by the two probes[12]. Thus, it appears that metal Tees may be used for probe placement in the plant. However, additional tests are recommended to confirm this result and a fundamental understanding of grounded versus ungrounded probe operation should be developed.

5.3 Zr Target Attainment

As discussed in the controls section, the Zr target was attained with feedforward control using a simple mass balance. The system's deviation from this target was primarily the result of error in the determination of the reagent addition rates, and error in the equation used to estimate the amount of Zr in the ZrDP feed. This equation is a function of density, and although the error in the density determination is only ±1.4%[13], the equation increases the error to ±8%. This error combined with the error in the RAL's analysis (shown in Table 2.0-1) implies that a difference as large as ±17% between the mass balance values and those provided by the RAL would be within experimental error (Although the 95% confidence level is ±13%).

Figure 5.3-1 is a plot of the percent deviation of the final Zr concentration from its target value based on the RAL's analysis of the final product along with the corresponding error observed for the nitric, water, ZrDP, and total ANN flows. Figure 5.3-2 compares the error based on the RAL's analysis to the error in Zr concentration which would be expected based on tank depletion and the Zr concentration in the recycle vessel from the density equation. It can be seen from Figure 5.3-2 that the mass balance values consistently agree with those obtained from the RAL's analysis within the error associated with the density equation and the analysis (i.e. ±13%). This supports the assumption that the tank depletion readings were accurate.

Referring to Figure 5.3-1, the relatively large deviations in the flow rates observed for runs 1 and 2 are attributable to operating the pumps without feedback control. The problem with attempting to control the system's peristaltic pumps without flowmeters was that they were subject to pressure effects (both inlet and outlet), viscosity effects, tubing hysteresis, and nonlinear response to control output. This nonlinearity produced the increase in error observed for the ZrDP pump during run 2 since it was run at a lower flowrate than in run 1. As can be seen from the Figure for these two runs, the resulting combination of higher than expected flows for water, ANN, and HNO3, along with lower than expected flow for the ZrDP resulted in Zr concentrations which were lower than the targeted values.

As a solution to these problems during run 3, orifice flowmeters were placed on all of the pumps with the exception of the ZrDP pump (due to materials problems) and the ANNII pump (due to its relatively low flow rate.) In addition, the ZrDP rate was determined by observing the tank depletion rate and setting the control output to the pump at a value which produced the desired flow. As shown in Figure 5.3-1, this resulted in a considerable improvement in the system's overall performance.

One of the purposes of run 4 was to demonstrate the viability of coprocessing with the ILCM. In order to accomplish this, a mixture of nitric acid and ANN which was nitrate deficient was used in the place of ANN. Although from this standpoint the test was a success, the failure of the orifice meter which was used to meter this mixture resulted in a considerable amount of dilution and a consequent low concentration of Zr as can be seen from Figure 5.3-1.

Figure 5.3-1 Zirconium Target Attainment

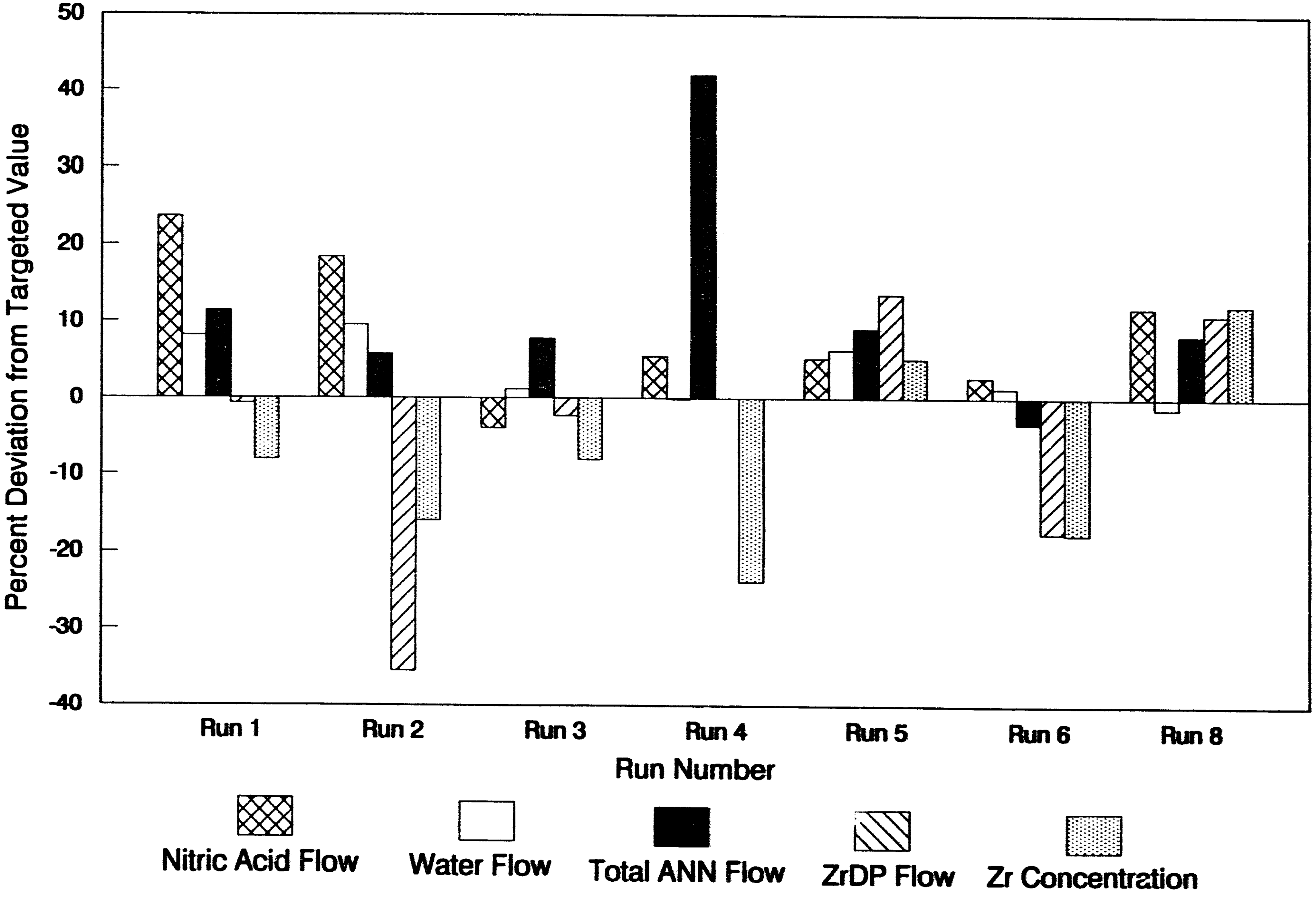
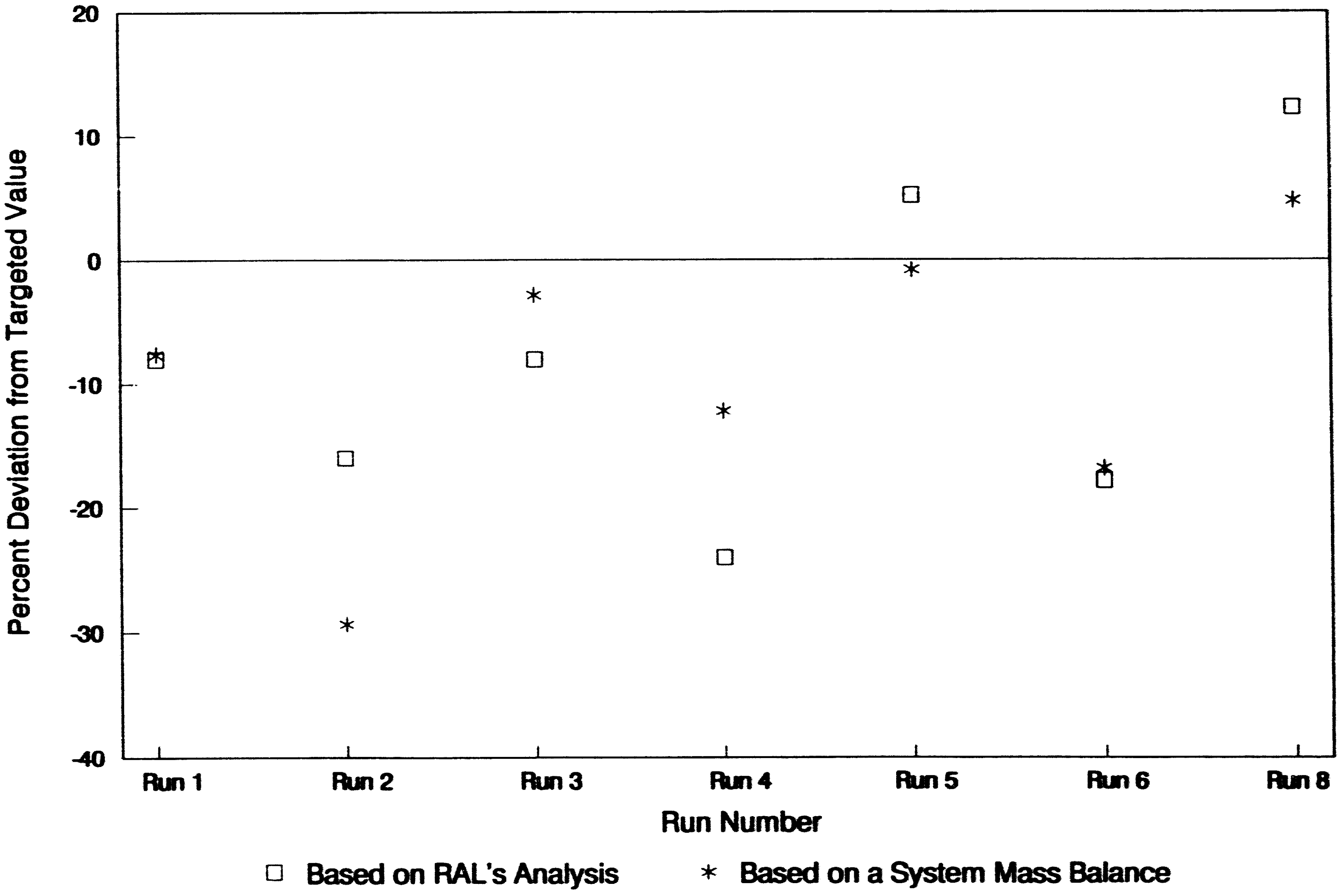


Figure 5.3-2 Comparison of RAL's Zirconium Analysis to the System Mass Balance Results.



After this test all of the pumps (with the exception of the ANNII pump) were converted to feedback control using turbine flow meters for run 5. Due to the hazards associated with calibrating the meters with reagents such as ZrDP, and HNO3, it was assumed that the flowmeters would exhibit a linear relationship between the pulses produced by the turbines and flowrate. Although this is not always the case[14], it proved to be an adequate first approximation as can be seen from the results for run 5 in Figure 5.3-1

The relatively poor performance with respect to the Zr target which was observed during run 6 is attributable to the 20 percent bias in the ZrDP pump. It is suspected that this bias resulted from solids plugging the screen in front of the flowmeter producing cavitation[15]. This in turn caused the meter to read a higher flow rate than was actually occurring resulting in less Zr in the final product.

In preparation for run 7, two flowmeters were placed into the ZrDP feed line to prevent an abort of the run due to a single flow meter being jammed by solids. Despite this precaution, the filters on both meters plugged and the run was aborted. The filters were subsequently disassembled and were found to contain a rubbery substance. It is suspected that this material came from thread sealant which was used on the recycle vessel.

Prior to run 8 a new filter was placed in front of the ZrDP flowmeter and this solved the plugging problem. In addition, larger flowmeters were placed on the ANN and HNO3 pumps to enable the system to operate at the design basis flowrate. The error in the Zr concentration during this run was primarily the result of a higher than expected flow of ZrDP. This probably resulted from calibrating the meter with water prior to this run and the higher viscosity of the ZrDP resulting in a bias in the flowmeter reading. It is suspected that the same phenomena is responsible for the high bias in the flow from the ANN and HNO3 pumps.

5.4 Nitrate Target Attainment

As with the Zr target, the nitrate target was attained with feedforward control using a simple mass balance. The error observed in the system's attainment of this target was primarily the result of errors in the determination of the amount of nitric acid and ANN being added.

Figure 5.4-1 is a plot of the percent deviation of the final nitrate concentration from its target value based on the RAL's analysis of the final product along with the corresponding errors for the nitric, water, ZrDP, and total ANN flows. Figure 5.4-2 compares the deviation based on the RAL's analysis to the nitrate concentration which would be expected based on tank depletion and the nitrate concentration in the ANN and HNO3. Figure 5.4-2 shows that the mass balance values agree (with the exception of run 4 which will be discussed later) with those obtained from the RAL's analysis within the associated analysis error. This supports the assumption that the tank depletion readings were accurate.

It can be seen from Figure 5.4-1 that higher than expected flows of HNO3 and ANN during run 1 resulted in the nitrate concentration exceed the target value. The same can be said for run 2, although the lower than expected flow of ZrDP resulted in an increase in the relative error. During run 3, a slightly lower than expected flow of HNO3 resulted in the nitrate being lower than its target.

Although the results for runs 1-3 are easily explained, the results for run 4 are not. Referring to Figure 5.4-2, it can be seen that the nitrate from the RAL's analysis does not agree with the value resulting from a mass balance. Evidence indicates that the error can be attributed to the RAL's analysis of the final product. The first reason for this assessment is that the low concentration of Zr in the final product is consistent with failure of the ANN/HNO3 mixture's flowmeter and consequent dilution of the final product. Secondly, a mass balance was performed on the Al in the system and this indicated that the analysis of the HNO3/ANN mixture used during this run was reasonably accurate. Finally, a comparison of the nitrate predicted to be in the final product by the following expression:

$$NO3 = HNO3 + 3Al$$

Where:

$HNO3 = Acid - (F-4Zr)$[16]

to that given by the RAL's analysis for this run indicated that the analysis values were lower then would be expected based on this equation (refer to the data point shown for run 4 in Figure 5.4-2)

The ZrDP feed for runs 5, 6, and 8 was all produced with minor quantities of HNO3 as an oxidant during the dissolution process. The slightly high bias in the mass balance values shown in Figure 5.4-2 for runs 5 and 8 is attributable to the erroneous assumption that all of this nitrate was consumed. The same can be said for run 6, although the low ZrDP flow was the primary contributor to the error.

5.5 Corrosion

Due to the short duration of coupon exposure, the data obtained on the corrosive effects of HNO3/HF mixing in the system is inconclusive. The apparent coupon weight loss was within the error of the measurement.

6.0 RECOMMENDATIONS FOR FUTURE WORK

Based on the results and problems encountered during operation of the ILCM, future work to ensure the successful operation of the integrated pilot plant and the full scale facility is recommended.

First, a robust probe should be developed which will operate reliably under the process conditions expected in the plant. While the performance of the current probe was found to be acceptable during relatively short mockup runs, occasional failures and unexplained drifts indicate that it is inadequate for

Figure 5.4-1 Nitrate Target Attainment

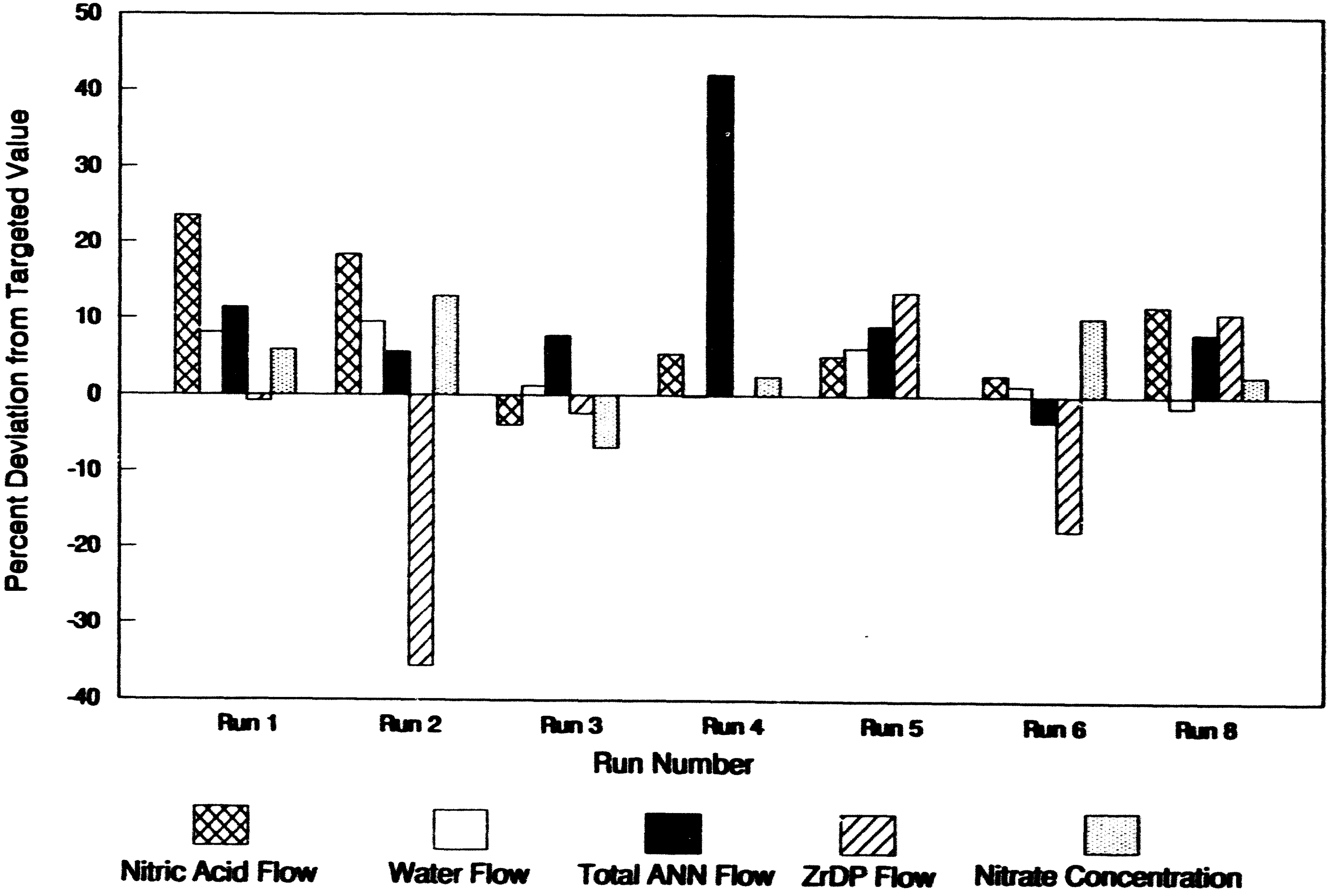
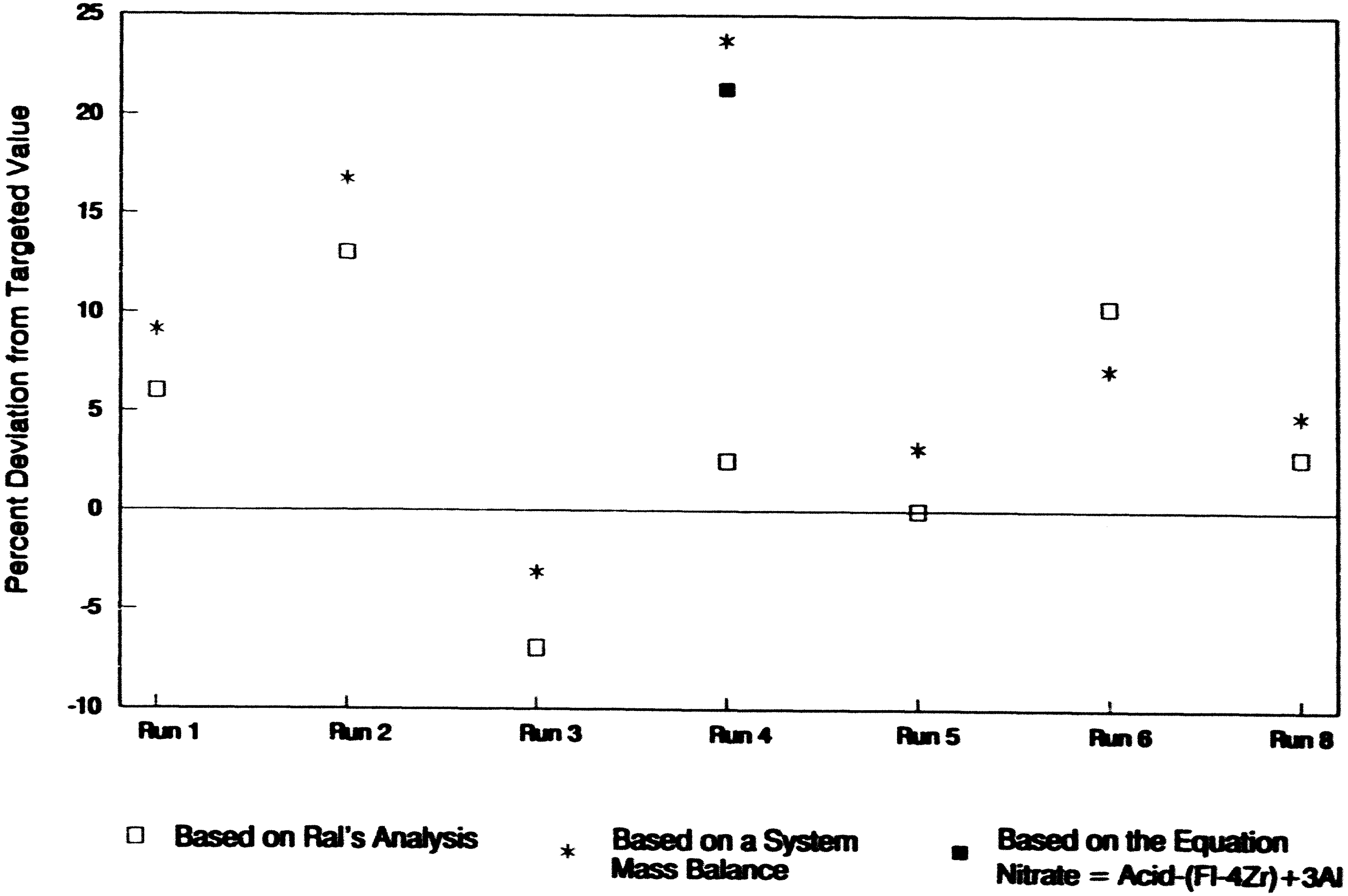


Figure 5.4-2 Comparison of the RAL's Nitrate Analysis to the System Mass Balance Results.



long term plant service. In consideration of the important role the HF probe would play in the full scale facility, a fundamental understanding of the reactions and transport phenomena associated with its operation should be developed.

Additional work will also be required to determine the types of equipment needed to provide the necessary control of reagent flows in the plant. Although pumps and flowmeters would be acceptable for the cold reagents, they would be unacceptable for radioactive service such as ZrDP flow determination. One possibility for measuring the ZrDP flow and its specific gravity would be a corriolis mass flowmeter. Although vendor data indicate this meter is capable of providing the required accuracy with respect to flow rate and specific gravity, it has a significant pressure drop associated with it. Another possibility would be a metering head pot, however, actual tests with this equipment should be performed to ensure it can provide the required accuracy.

Experiments investigating the corrosion effects of HNO3/HF mixing should be set up. Initially, this should involve the use of limiting conditions, i.e. HNO3/HF without ANN present in the solution. If this test indicates that further investigation is warranted, then a continuous flow experiment should be setup on a reduced scale to avoid generating excessive amounts of product solution.

Additional pilot plant runs should be carried out to confirm the required residence time for reaction under all anticipated operating conditions. During these runs the possibility that smoothing the incoming HF probe signal coupled with derivative control could improve the system's ability to deal with disturbances should be investigated. In addition, the magnitude of the start-up disturbance could be reduced via closer tuning of the feedforward contribution.

7.0 CONCLUSIONS

In-line Complexing is a viable concept with the system consistently approaching the HF (error range -14% to 8%), Zr (-16 to 12%), and nitrate targets (-7 to 13%).

The necessary residence time for the reaction under the conditions observed during these experiments appeared to be between 0.3 and 0.67 minutes.

The tests indicated that two points for ANN addition are adequate. During the last five runs of the system complexing was essentially complete through the use of the first ANN addition point only. However, the second addition system may be required in the event of extreme upset conditions or failure of the first system.

A test of the system carried out with a mixture of ANN and nitric acid demonstrated the applicability of the process to co-processing.

Metal fittings appear to be acceptable for placing probes in the plant, although further testing and theoretical studies are recommended.

Concerns about the reliability of the HF probe need to be addressed. In addition, a fundamental understanding of its operation should be developed.

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Appendix A MICROMAX Description and Configuration

The MICROMAX is a Leeds and Northrup process control system used for Inline Complexing development. It is similar to a DCS but does not have certain DCS features such as peer-to-peer communication. It consists of a management station and local processing stations (LPUs). The short distances in the ILCM made it feasible to wire all analog signals to one LPU and all discrete signals to an expansion unit wired to the LPU for this purpose. Having all the signals in one unit made the highly interactive control system possible. All control logic is performed in the LPU. The system as a whole reads signals, performs calculations, displays and stores data, and processes alarms and control logic. The MICROMAX provides high resolution analog inputs (15 bits) and direct reading of thermocouples (within 0.25 C). This high accuracy assisted in process analysis and development. The scan rate for all signals was fixed at 0.75 sec. This provided plenty of speed for this process. A listing of pertinent analog inputs follows this text. Each point has a designator U 1 AI XX, standing for unit 1, analog input xx. Inputs 61 through 65 are pulse counters for the turbine meters. Each input has a designated range, which provides a linear conversion to engineering units. The range listing is included in the configuraton. The control configuration was entered in a graphical format which the machine compiled. An example of graphic programming is included in the configuration listing.

The overall structure of the control system is shown in the following diagram. The first ANN addition is a calculated feedforward amount based on an estimated Zr concentration. It is adjusted by a feedback signal from an HF probe. This same probe feeds forward to a second ANN addition point which is also feedback adjusted by another HF probe downstream. The logic flow of the two feedforward calculations is shown after the control diagram. Water and Nitric acid flowrate setpoints are calculated by an overall mass balance. Many of the equations were provided by James A. Murphy for HF probe signals and feedforward additions. These are described in the attachment prior to the configuration.

Equations

Pseudo points are variables that can be set by equations or other numerical functions (integration, derivative, filter, etc). The MICROMAX does not use the standard hierarchy of arithmetic operations but evaluates expressions in sequence so A+B*C = (A+B)*C not A+(B*C) so parenthesis are used extensively in equations to force the desired calculations. In the following listing, the labels for each PP are given at the top of the page, along with a brief description. The equation is given with the corresponding number at the bottom of the page. Most PP's are numerical expressions. Some are MICROMAX functions such as totalizer, rate of change, lead/lag, and select. Each loop requires a dedicated PP for it's execution. Equation abbreviations are as follows:

AI	analog input
AO	analog output
CO	control output
CN	constant
LS	local setpoint (of loop number)
PP	another pseudo point

The 6 Titanium probes that measure HF are central to the system. A custom made electronics box provides a bias voltage and the current through each probe is converted to a voltage by a current amplifier. A schematic of the box circuit is included after the configuration listing. Each probe signal is read as a voltage by the MICROMAX and has an associated thermocouple. Each has an equation that converts the voltage to HF, and another one that corrects this value to a standard temperature. The probes are numbered 1, 2, 3, 5, 6, and 7. The position that would be number 4 is used to read the bias voltage applied to the probes. The probe output is strongly dependent on this value so it is monitored.

Constants

Constants are numerical values that can only be set by the operator. Often these are used in equations instead of numerical values because of the way that the system uses memory. There is no significance to the order of constants and pseudo points. The feedforward calculation for ANN depends on the feed SpG. The SpG is monitored but the signal is noisy and contributes to noise in the control system. An assigned constant SpG is used instead and the value is set at the beginning of the run. The HF value is provided by the Zr dissolution data. The probe equations use all the same constants except for a calibration factor which is unique for each probe.

Control Loops

There are seven interacting PID loops in the control system. The feed stream and each reagent except final ANN has its own flow controller, and probe signals control outer cascade loops. The following diagrams show the structure and flow of logic in the control loops, and the control labels are shown in the attached configuration. The MICROMAX allows loops to be tuned with integral action only if desired. This mode was selected for flow loops after preliminary testing. It has the advantages of providing filtering action to the noisy flow signals, and eliminates offset to achieve control at setpoint. The same tuning mode was implemented for the probe controlled outer loops with acceptable overall system response times.

Several of the pseudo points and constants are selector switches which can be assigned values of zero or one. These are used as factors in equations and control whether part of the equation is implemented, for example whether feedback is combined with a feedforward signal. There are several forceback equations. These are used in cascade loops to set the initial values of the outer loop controller output to allow bumpless startup. This is accomplished by solving for the controller output with other known signals.

Originally the equations converting the outer cascade controller output to a setpoint for the ANN flow loop only allowed for single-sided control, that is, the amount of ANN could only be increased over the feedforward contribution by feedback. A "normal" controller output would be 0 in this case. The configuration was later changed to allow double-sided control in which feedback could add or subtract from the feedforward ANN. This was accomplished by subtracting 50 from the controller output of 0 to 100, resulting in a "normal" controller output of 50. This allowed a much closer approach by the feedforward system. Previously, the feedforward contribution had to provide a low estimate of the ANN required to allow the single-sided control to work. Now a best estimate can be used, and the closer the system approaches setpoint with feedforward, the less feedback correction is required. This is implemented in PP53. PP5 scales and transfers the output of the outer loop (4) to the setpoint of the middle loop (2) in similar double-sided fashion. The target HF is multiplied by CO/50 giving a "normal" CO of 50 and a range of 0 to twice the free HF. PP53 also contains the factor LS5 in the feedback portion. This is the setpoint for the feedrate. Including this factor allows the system to respond immediately to setpoint changes, without having an error propagate through the loop before correction.

Discrete I/O

There are several discrete I/O points in the control configuration which are primarily used as logic flags to direct control of the loops. A box was wired with switches to discrete inputs which provided override of automatic shutdown to allow startup and provide for a forced shutdown. The MICROMAX provided a ladder system for logic programming. The shutdown logic was programmed on a ladder diagram which is included in the configuration listing. The discrete I/O also provided control of the outer loops of the cascades for forceback (track) or run status to allow bumpless startup, and control feedforward only or feedforward plus feedback for ANN startup. This allowed these important functions to be performed without switching display screens during periods of intense operator demand. A discrete output was also used to control the feed temperature via an on/off loop and a steam solenoid. The system also has control relays which are internal status flags. Most of these were used for alarms.

Alarms

Process alarms and setpoints are given in the configuration listing. These were advisory only and did not perform any automatic functions. Vessel levels and temperatures were alarmed, as were high HF levels in the process. Plugged lines or jammed turbine meters were detected by calculating an expected ratio of control output signal to measured flowrate, and alarming if the ratio fell outside of an expected range.

Displays

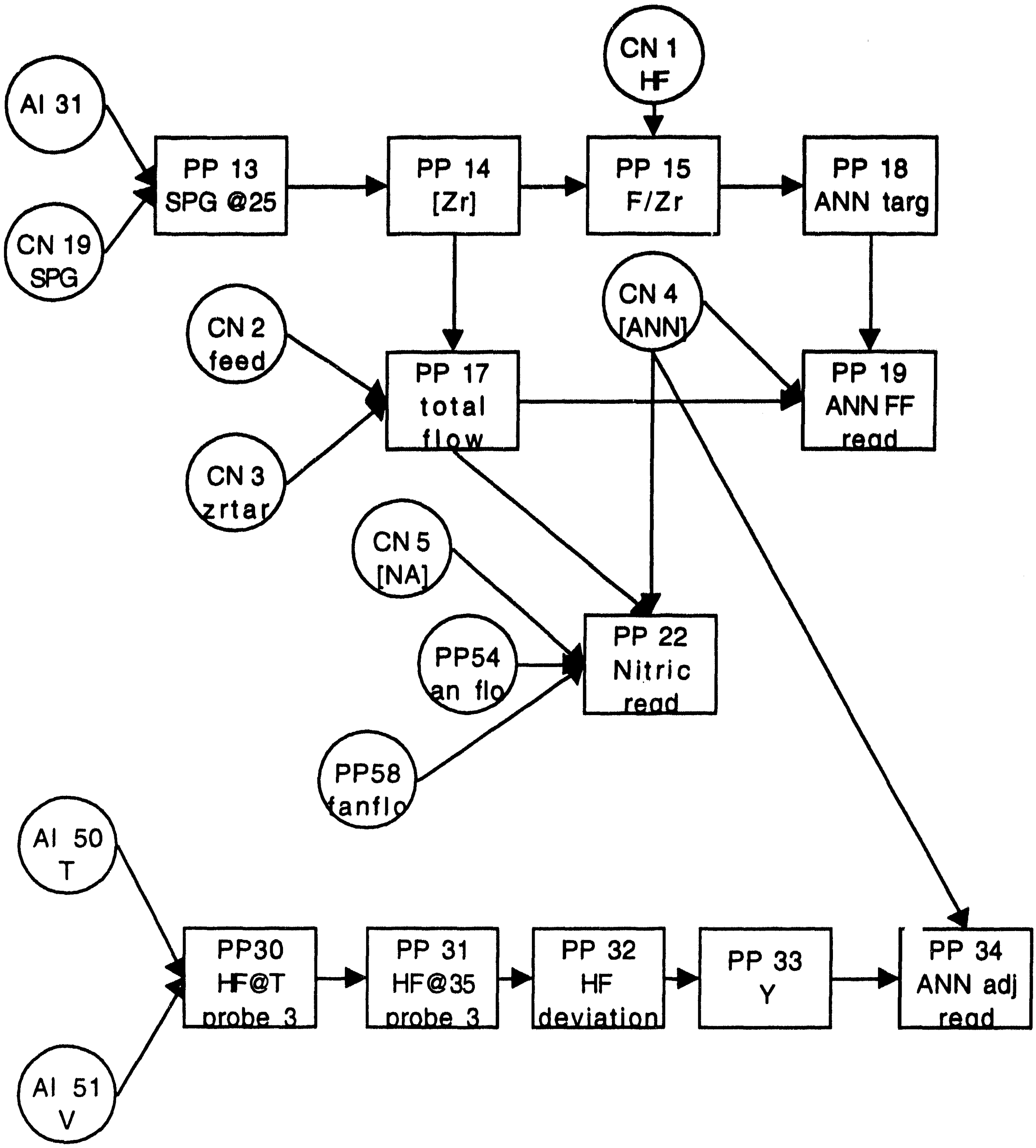
All process data were available on the MICROMAX screen. The signals were logically arranged into display groups which are given in the configuration listing. These groups could be trended on the screen. The bar/status screen gave an overview of the whole process and direct control over the loop settings and was the primary screen used for operation.

Storage

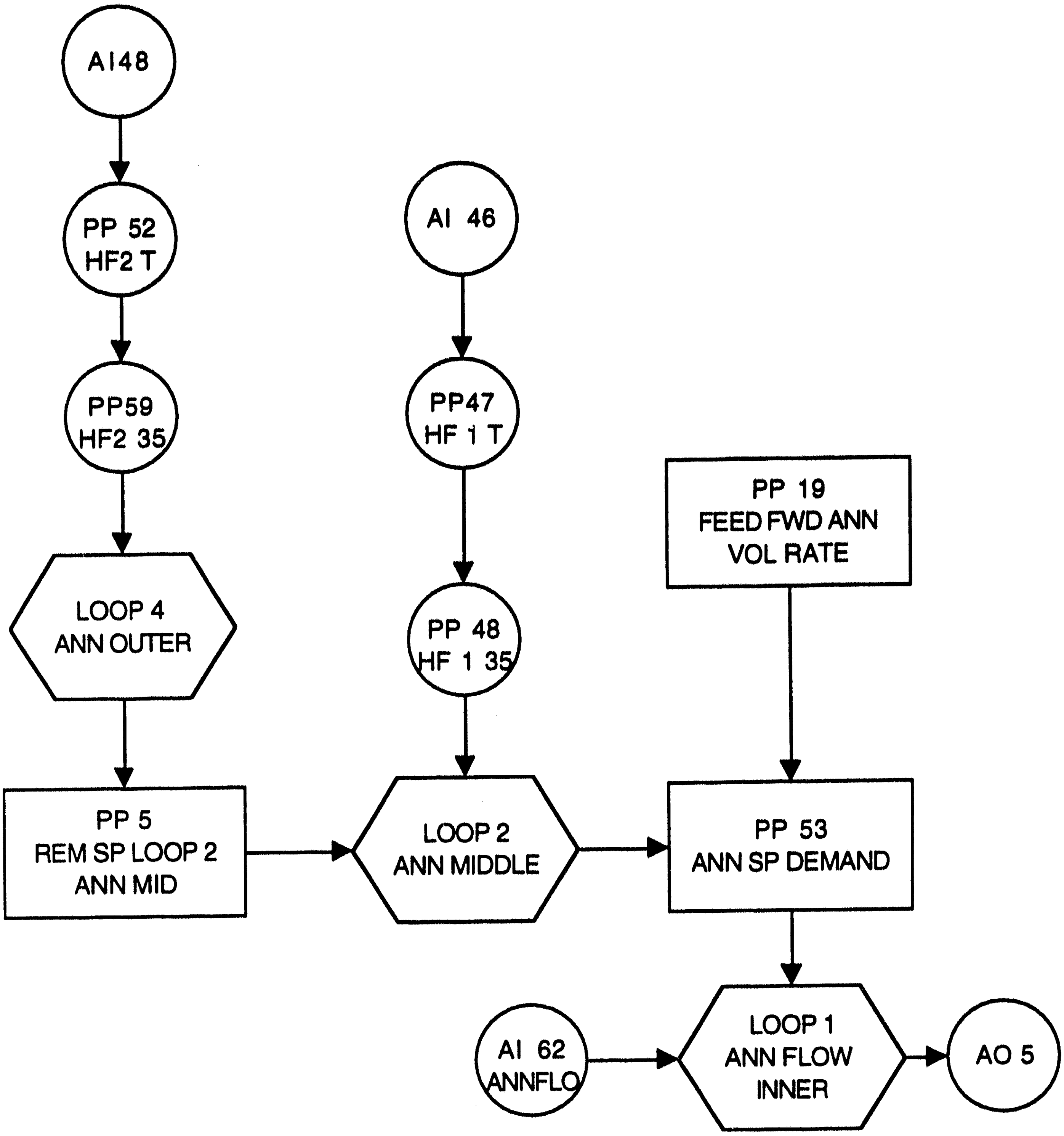
All important signals and pseudo points were recorded on streaming magnetic tape for storage and transfer to Lotus files on a PC for later analysis.

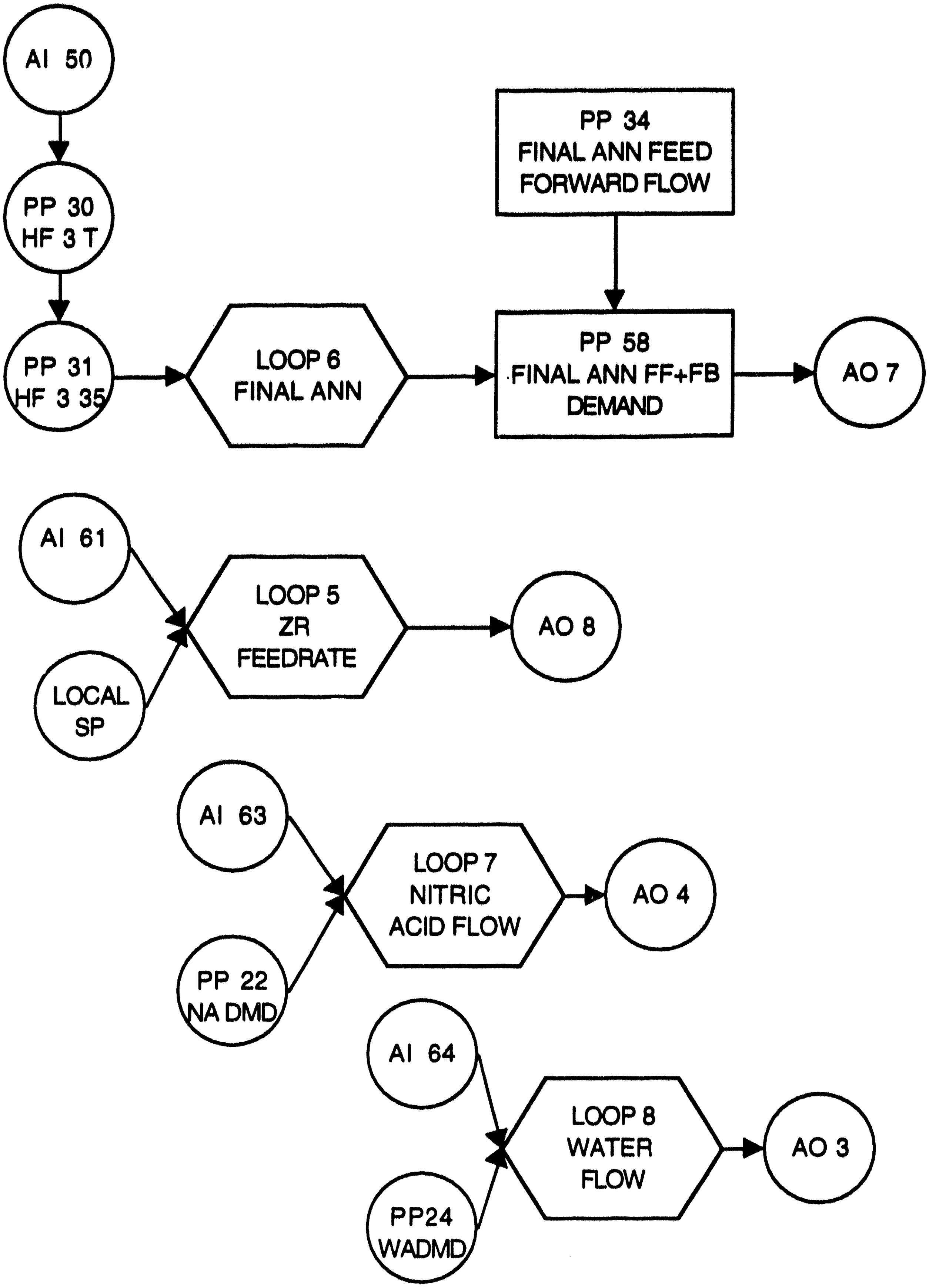
Operations

Each run was performed on a single shift. The actual operation took only 1 to hours, but the setup and calibration of the instruments took several hours. The following sheets describe the pre-run checkout, startup, and operation instructions. This system was highly dependent on instruments and the startup of the cascade loops required a complex series of steps.



AI 31
CN 19 SPG
PP 13 SPG @25
PP 14 [Zr]
CN 1 HF
PP 15 F/Zr
PP 18 ANN targ
CN 2 feed
CN 3 zrtar
PP 17 total flow
CN 4 [ANN]
PP 19 ANN FF read
CN 5 [NA]
PP54 an flo
PP58 fanflo
PP 22 Nitric read
AI 50 T
AI 51 V
PP30 HF@T probe 3
PP 31 HF@35 probe 3
PP 32 HF deviation
PP 33 Y
PP 34 ANN adj read


A I48
PP 52
HF2 T
PP59
HF2 35
LOOP 4
ANN OUTER
PP 5
REM SP LOOP 2
ANN MID
AI 46
PP47
HF 1 T
PP 48
HF 1 35
LOOP 2
ANN MIDDLE
PP 19
FEED FWD ANN
VOL RATE
PP 53
ANN SP DEMAND
AI 62
ANNFLO
LOOP 1
ANN FLOW
INNER
AO 5


AI 50
PP 30
HF 3 T
PP 31
HF 3 35
LOOP 6
FINAL ANN
PP 34
FINAL ANN FEED
FORWARD FLOW
PP 58
FINAL ANN FF+FB
DEMAND
AO 7
AI 61
LOCAL
SP
LOOP 5
ZR
FEEDRATE
AO 8
AI 63
PP 22
NA DMD
LOOP 7
NITRIC
ACID FLOW
AO 4
AI 64
PP24
WADMD
LOOP 8
WATER
FLOW
AO 3

J. A. Murphy
6-4453/CPP-637/5219
June 4, 1990
Transmittal of Equations for Control of In-Line Complexing

Attached are the equations and associated supporting information to control the complexing chemistry for the In-Line Complexing Pilot Plant. These equations were generated so to avoid the lengthy iterative calculations which must take place to calculate the free HF in equilibrium with a complex matrix of metals and anions. These equations were created for the GFD type matrix and should not be used to complex other matrixes until I can verify their validity.

J. A. Murphy
Sr. Scientist, Process Development

Attachment Text

In-Line complexing is identified clearly as the choice method of complexing zirconium dissolver product in the FDP upgrade primarily because of cell space savings.[1] This process is downstream of the dissolution equipment and continuously adds the proper additions of aluminum nitrate, nitric acid, and water to bring the free HF of the complexed product to a quantity suitable for stainless steel vessels downstream of the dissolution facility. Other aspects controlled are the nitrate concentration (for uranium extractability) and final zirconium concentration (for solution stability).

Once the solution has left the dissolution portion of the system, it enters a geometrically favorable vessel where the specific gravity and temperature are measured. These measurements are used in conjunction with the initial HF concentration of the feed to calculate the zirconium and fluoride concentration of the solution. These are fed with product flowrate into the computer and flowrates of the three complexing solutions, aluminum nitrate, nitric acid, and water, are generated. The solution is initially slightly undercomplexed to a degree adequate for the anticipated error of the conversion of specific gravity to fluoride and zirconium concentrations to ensure overcomplexing does not occur. The undercomplexed solution is mixed, allowed some time to reach equilibrium and measured by a titanium electrode for free HF. The free HF concentration is fed into the computer and a flowrate of aluminum nitrate is calculated to complete the complexing to the goal free HF of 0.045 Molar at 35 degrees C. This goal will be optimized following some corrosion and stability laboratory tests.

The solution will run past another titanium electrode where a second adjustment will attempt to finalize the complexing within an operating envelope. However, it will be attempted to finish all adjustments with only one adjustment. The final solution will be measured in a holding tank and adjustment lines into these vessels will allow correction of the free HF if necessary to prepare the product for pilot plant calcine feed.

In the past, in order to complex a zirconium dissolver product, a computer program used an iterative calculation method to converge all the equilibria and, a solution with a number of ions, takes several thousands of calculations. This is an adequate way to determine the complexing needs for a bulk tank, but inadequate for a continuous process which changes from time to time. The equations generated in the following sections will allow the Micromax computer, which has simple mathematical capabilities, to calculate the needed additions in a real-time fashion.

The equations will be broken up into two sections. First, the initial complexing which will intentionally undercomplex the dissolver product. Second, the adjustment equations are given to finalize the free HF to the proper concentration.

Many of these equations are derived using laboratory data because the free HF equilibrium equations appear to calculate about 10-15 percent low as compared to the titanium electrode. Studies of the titanium electrode are continuing, to give assurance that the reading is proportional to only free HF.

Initial Complexing

Initially, the computer will need three inputs to properly generate the initial flowrates. These inputs are the specific gravity, temperature, and the initial HF concentration. It is important that these measurements be as accurate as possible to reduce the associated error in the flowrate calculations. The first equation converts the specific gravity and associated temperature to a normalized specific gravity at 25 degrees C. This equation was generated by L. L. Burger, et al,[2] and is rewritten to solve for specific gravity at 25 degrees C as follows:

Equation 1 $\text{Sp-Gr}_{25} = (D_t + 0.0036 - .000145 * t) / (1.0125 - 0.0005 * t)$

where D_t is the density at the measured temperature, t in degrees C.

The next step is to calculate the zirconium concentration as described in equation 2.

Equation 2 $[Zr] = (\text{Sp-Gr}_{25} - 0.0066 * [F] - 0.99707) / 0.1367$

where [F] is the initial HF concentration in the dissolvent feed and [Zr] is the molarity of zirconium in solution (ref 3,4). These equations were generated prior to the development of highly analytical specific gravity instrumentation as available in the Remote Analytical Laboratory and were specific for a different flowsheet than GFD. This is one area where improvement is possible if these density equations would be reevaluated using current technology and current flowsheet compositions.

These calculations are necessary to properly calculate the fluoride (same as initial HF) to zirconium molar ratio. This ratio affects the quantity of aluminum that is needed to complex the free HF. This simple equation is:

Equation 3 $F/Zr = [F] / [Zr]$

The zirconium target concentration must be stipulated to continue. The product coming out of FDP is currently 0.49 molar zirconium[5]. This concentration is necessary to ensure stability in the tank farm. (There is some hope this concentration will be increased because of anticipated increased stability after running through FPR's first cycle and an increase in the minimum temperature in the tank farm to 25 degrees C. These two changes may take the zirconium concentration in the product to 0.68 molar in the final GFD product[6].) Because there is an anticipated 7 % volume addition expected for adjustments, the initial target will be set at 0.524 molar zirconium. This target calculates the final volume the product must be diluted to as follows:

Equation 4 $\text{final flowrate} = ([Zr] / 0.524) * \text{ZrDP}_{\text{flowrate}}$

For a fixed final zirconium concentration, the targeted aluminum concentration is proportional to the [F] to [Zr] molar ratio from equation 3. The slope and intercept of this linear equation are cubic functions of the targeted free HF concentration and should be calculated prior to insertion into the

Micromax calculational control loop. This equation will allow the free HF to be changed without redoing all the equations. Equation 5 shows the function to calculate m, the slope. Equation 6 shows the function to calculate b, the intercept. Equation 7 shows the function to calculate the final aluminum concentration. The targeted free HF based on equilibrium equations used will probably be 0.045 molar but, because of the differences between the equilibrium equations and the titanium electrode, the actual electrode target is 0.053 Molar free HF.

Equation 5 $m_{slope} = 1.4374 * [HF]^2 - 0.4997 * [HF] + 0.2672$

Equation 6 $b_{intercept} = -899.6 * [HF]^3 + 168.8 * [HF]^2 - 11.7952 * [HF] - 0.8281$

Equation 7 $[Al] = m * [F] / [Zr] + b$

where [HF] is the targeted free HF at 35 degrees C, [Al] is the final aluminum concentration needed, [F] is the initial feed HF concentration, and [Zr] is the zirconium concentration calculated from the density measurements. The amount of aluminum needed to complex the excess free HF is, as defined above, is a function of the desired final free HF concentration and a function of the fluoride to zirconium ratio. Several fits were attempted, but the best statistically significant fit was the above relationship. The equations for the slope and intercept were calculated using several subsets of data with different final free HF's and F/Zr ratios. These set up a family of curves which individually were linearly fitted with respect to the F/Zr molar ratio and the final needed aluminum concentration. Each subset's coefficients were fitted to many functions of final HF concentration until the cubic relationship was found to be the best for both the slope and intercept in relationship to the targeted free HF molarity. It is important to note that the first two cubic equations 5 & 6 should be calculated prior to placing equation 7 into the Micromax computer. Equations 5 & 6 are only needed when changing the initial free HF target.

These equations were generated using data calculated by a program which used the HF equilibrium constants and varied the $[HF]_{initial}$ concentration and the density measurements. Because the data calculated were generated by a computer program, a good R^2 factor was achieved as follows. R^2 on the equation for the slope was 0.9998, the R^2 on the equation for the intercept was 0.9995. The R^2 on the linear equations for each [HF] concentration was 1.0. The combined fit was not calculated simply because the slope and intercept fits were so good and the overall fit of the three equations as a whole is better than 0.999.

Once the [Al] (aluminum molarity) is calculated, the amount of aluminum to add is calculated by multiplying the [Al] by the final flowrate and dividing by the concentration of the aluminum feed concentration, [ANN], as follows in equation 8:

Equation 8 Aluminum flowrate = [Al] * final flowrate / [ANN]

The nitric acid concentration is next to calculate and uses a target of approximately 2.0 molar. Realize the final targeted nitrate concentration is set for 2.4 molar to allow for good uranium extractability. However, because there are still additional adjustments with aluminum nitrate, the nitrate is adjusted initially low. The nitric acid concentration is naturally related to the nitrate concentration desired with the amount already added by the aluminum in equation 8 subtracted out as follows:

Equation 9 $HNO_{3flowrate} = (2.0 - 3 * [Al]) * \text{final flowrate} / [HNO_3]$.

This and other equations assume that an aluminum basket will not be used in the pilot plant. Some adjustments to these equations may be needed to allow for the aluminum from the basket material used in the actual plant.

The last step of the initial complexing equations is the water flowrate. This is a simple subtraction of the total added volume from the needed volume for zirconium stability. Equation 10 explains.

Equation 10 $H_2O_{flowrate} = \text{final flowrate} - ZrDP_{flowrate} - ANN_{flowrate} - HNO_{3(flowrate)}$.

Although these equations are simple, they nevertheless removed many of the iterative calculations necessary to initially complex a solution. Some of the targets as stated in earlier parts, may need to be adjusted when actual plant data are available to optimize the solution's chemistry.

Adjustment Routines

These routines are based on having a calibrated titanium electrode reading (shown how to do later) and a calibrated temperature reading. These equations were not trivial to get and are somewhat limited in scope. They should however, work adequately well with the In-Line complexing of GFD product. The equations will need revision if they are to be used on poisoned product or solutions out of the operating ranges set for the GFD.

The first equation deals with the proper calibration of the Titanium electrode. Currently, the majority of the electrodes in the In-Line complexing pilot plant module contain the Ti-0.2%Pd electrode material. The equation given below is good for these types only. A similar equation will be generated by D. B. Illum soon for the commercially pure titanium electrode. The calibration of the electrode involves accurately measuring the electrode voltage in a $HF\text{-}HNO_3$ calibration solution. The temperature must also be measured. A calibration factor, f, is calculated as follows. For this equation, an example will be used where an HF solution [HF] is measured at temperature t and with a voltage volts.

Equation 11 $f = \frac{(0.0851 * t^2 * [HF] - 2.033 * t * [HF] + 32.54 * [HF] + 0.12 - 0.00871 * t)}{\text{volts}}$

This factor, f, can be used to directly calculate the free HF in solution at any temperature by using equation 12.

Equation 12 $[HF] = ((v * f1) + 0.00871 * t - 0.12) / (0.0851 * t^2 - 2.033 * t + 32.54)$

where t is measured degrees C, v is measured volts, and [HF] is free HF in solution at the measured temperature, t. Both of these equations are the same except solved for different factors. When analyzing this equation, a relationship was found by observing the subsets of temperature data which created a family of curves. Each individual curve at a given temperature exhibited a linear relationship between current and free HF in solution. The coefficients were then examined and the slope was found to be a cubic function of the temperature and the intercept was a linear function of temperature. Once the entire equation was evaluated, the residual standard deviation published[7] for this equation was 0.00088 molar free HF ($R^2 = 0.9992$).

The next calculation is to normalize the free HF measurement to a standard temperature to allow correct adjustments. 10 solutions were accurately prepared and measured by the titanium electrode at varying temperatures (from 25 to 45 degrees C) to get the next equation which normalizes the measured free HF at process temperature to the free HF at 35 degrees C. The expected temperature at the first adjustment point is 40 degrees C. The linear equation's slope is a function of the measured temperature.

Equation 13 $[HF]_{35} = [-0.01434 * t + 1.4934] * [HF]_t + 0.00044$

Each subset of data had a linear fit with free HF at the given temperature and the free HF at 35 degrees C. The coefficients from the family of curves were fitted to temperature. The slope was found to be linear with respect to temperature. However, the intercept remained constant (virtually zero). This equation has a residual standard deviation on the estimated [HF] at 35 degrees C of 0.00053 molar.

The next equation is a bit more tricky to use and will be done in a couple of steps to make it easier to understand. About 15 slightly undercomplexed solutions were titrated with 2.22 Molar $Al(NO_3)_3$ to a measured free HF of approximately .035 molar at 35 degrees C. They were actually titrated to a normalized voltage which happened to be about .035 molar HF normalized to 35 degrees C. The relationship found for the adjustment part of the In-Line complexing is a proportionality of the free HF which needs to be complexed with the molar change in aluminum necessary to complex it. The equation defined has several parts to it. First, the "x" parameter must be defined. This factor is the measured free HF normalized to 35 degrees C minus the targeted 0.045 Molar free HF at 35 degrees C.

Equation 14 $x = [HF]_{35} - 0.045$ <----(HF target)

Next is to calculate the function for the volume of aluminum to add, but to simplify the calculation, y is calculated first.

Equation 15 $y = 612.51 * x^3 - 79.437 * x^2 + 6.062 * x - 0.00037$

Equation 16 ANN flowrate = y * product flowrate / ([ANN] - y)

where the product flowrate is the flowrate at the adjustment point (prior to adjustment $Al(NO_3)_3$ addition) and [ANN] is the stock aluminum nitrate reagent concentration.

These relationships above were found by analyzing the effects of a different starting free HF and different volumes on aluminum needed to complete complexing based on a starting relationship of

$$\frac{nAl}{V_{final}} = f(x) = y$$

where nAl is the number of moles of aluminum needed and Vfinal is the volume being adjusted. The relationship each had on the complexing calculation was not known. Some information was known about the relatively weak-complexing ability of the aluminum ion in comparison to the strong-complexing zirconium ion in the system. Because zirconium was such a stronger complexant, the free HF above the goal free HF (the difference as stated in Equation 14) was primarily the HF titrated with the aluminum. In other words, the aluminum did not strip off much of the fluoride from the zirconium but enough the linear function of aluminum needed and free HF measured was not adequate. However, the cubic function was. It was also known that the aluminum needed was a function of the volume to be complexed. This function was calculated after the insertion of the cubic function of free HF to complex was added. The last part of the equation was the conversion from aluminum volume to moles by using the aluminum nitrate concentration.

This equation is good for an initial free HF of up to 0.11 molar and for adjusting the solution to 0.045 molar free HF at 35 degrees C. The same equation can be used for calculating both adjustment points since they are similar. The standard error on this equation is the highest of all the equations and amounts to about 5 %, which results in a complexing error of 0.0022 molar free HF. The size of this error was not unexpected since these equations were generated using actual laboratory results where some measurement errors were probable. Using different concentrations of aluminum nitrate from 2.0 to 2.3 does not severely affect this equation's effectiveness on proper complexing.

Most of these equations were generated in the laboratory where conditions were controlled (such as stirring rate, stabilization time, etc.). There is a possibility that some biases in the free HF complexing will occur in the continuous system. This will be handled by adjusting these equations with actual pilot plant data to achieve proper complexing and/or modifying the equipment configuration as needed.

This letter completes, for now, the action needed to statistically generate equations capable of properly complexing zirconium dissolver product in the GFD In-Line complexing pilot plant without iterative convergence routines. In the future, an improved equation correlating zirconium concentration with the measured specific gravity of the zirconium dissolver product will be determined for specific GFD compositions. This is expected to improve the accuracy of the initial complexing.

References:

1. R. J. Kirkham, RJK-5-89 to M. D. Staiger, "Preliminary In-Cell Equipment Flowsheets for FDP Upgrade Options", dated October 31, 1989, still to be issued.

2. L. L. Burger, I. M. Rehn, H. R. Schmidt and C. M. Slansky, Properties of the System: Uranyl Nitrate-Aluminum Nitrate-Nitric Acid (or Sodium Hydroxide-Sodium Nitrate-Hexone) Part II Density, Apparent Molar Volume, and Viscosity, HW-11276, March 1949.

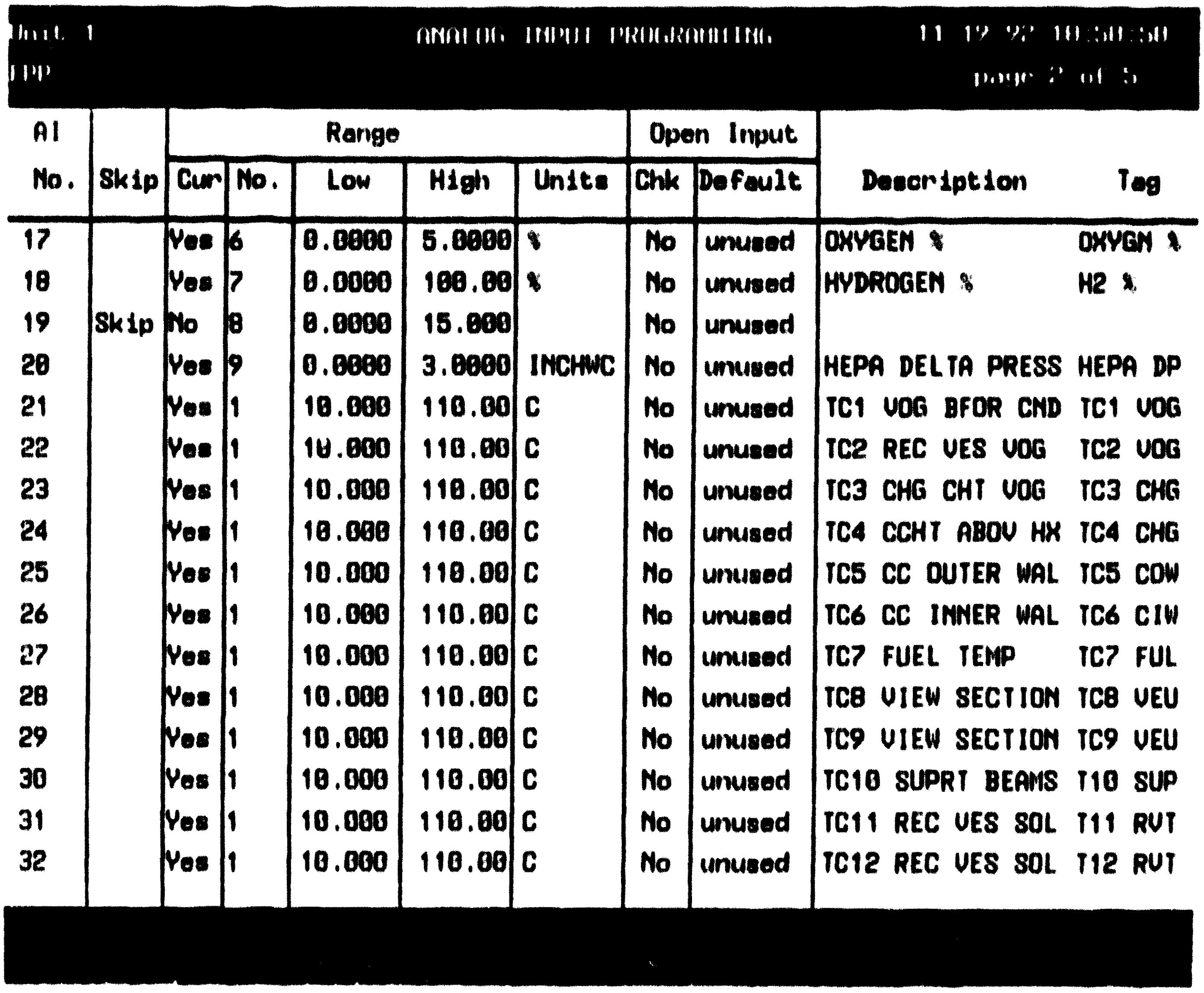
3. J. F. Krupa, JFK-2-84 to J. D. Christian, "Density Equation for Fluorinel B/Cd Flowsheet Product", dated March 6, 1984.

4. Hydrofluoric Acid reference manual. Copyright 1978, Allied Chemical Corporation.

5. J. A. Murphy, Murp-1-89 to J. A. Rindfleisch, "Flowsheet Changes for FDP Campaign 44", dated March 8, 1989, (confidential attachment).

6. J. A. Murphy, Murp-2-89 to C. B. Millet, "Preliminary Raffinate Solution Compositions for Present FDP and Upgrade GFD Dissolution Processes," dated April 30, 1990.

7. J. D. Christian, D. B. Illum, and J. A. Murphy, Metal Electrodes for Continuous Amperometric Measurement of Free Hydrofluoric Acid In Complexed Acidic Solutions, Talanta, 37, 651-654 (June 1990). (Note that data and equations in this paper for the Ti-Pd electrode are for the Ti-0.2% Pd nominal composition; the composition stated, Ti-2% Pd, is incorrect.)



Unit 1 ANALOG INPUT PROGRAMMING 11 12 92 10:50:50
IPP page 2 of 5

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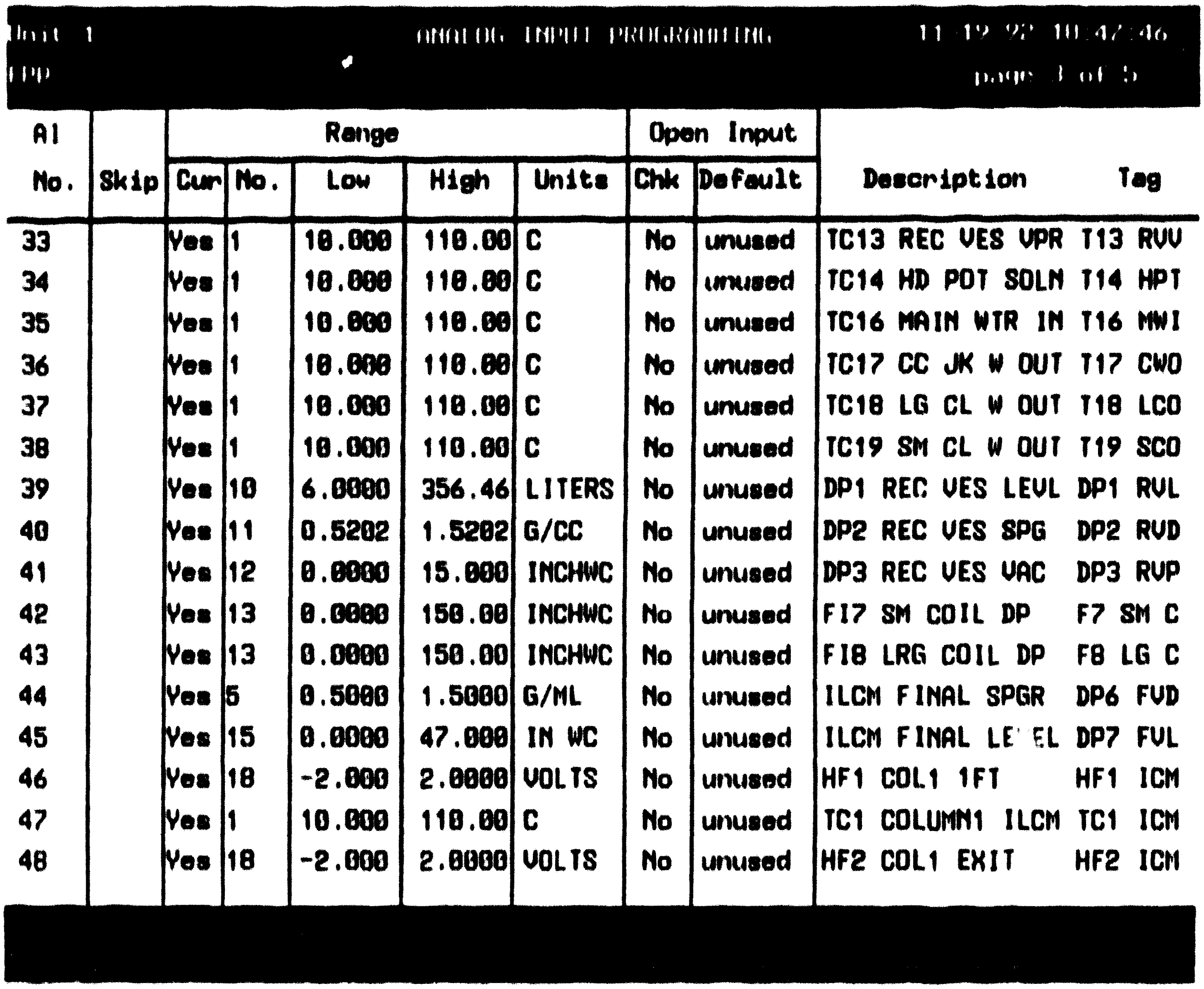
19	Skip	No	8	0.0000	15.000		No	unused		
20		Yes	9	0.0000	3.0000	INCHWC	No	unused	HEPA DELTA PRESS	HEPA DP
21		Yes	1	10.000	110.00	C	No	unused	TC1 VOG BFOR CND	TC1 VOG
22		Yes	1	10.000	110.00	C	No	unused	TC2 REC VES VOG	TC2 VOG
23		Yes	1	10.000	110.00	C	No	unused	TC3 CHG CHT VOG	TC3 CHG
24		Yes	1	10.000	110.00	C	No	unused	TC4 CCHT ABOV HX	TC4 CHG
25		Yes	1	10.000	110.00	C	No	unused	TC5 CC OUTER WAL	TC5 COW
26		Yes	1	10.000	110.00	C	No	unused	TC6 CC INNER WAL	TC6 CIW
27		Yes	1	10.000	110.00	C	No	unused	TC7 FUEL TEMP	TC7 FUL
28		Yes	1	10.000	110.00	C	No	unused	TC8 VIEW SECTION	TC8 VEU
29		Yes	1	10.000	110.00	C	No	unused	TC9 VIEW SECTION	TC9 VEU
30		Yes	1	10.000	110.00	C	No	unused	TC10 SUPRT BEAMS	T10 SUP
31		Yes	1	10.000	110.00	C	No	unused	TC11 REC VES SOL	T11 RVT
32		Yes	1	10.000	110.00	C	No	unused	TC12 REC VES SOL	T12 RVT



Unit 1 ANALOG INPUT PROGRAMMING 11 19 92 10:47:46

IPP page 3 of 5

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35		Yes	1	10.000	110.00	C	No	unused	TC16 MAIN WTR IN	T16 MWI
36		Yes	1	10.000	110.00	C	No	unused	TC17 CC JK W OUT	T17 CWO
37		Yes	1	10.000	110.00	C	No	unused	TC18 LG CL W OUT	T18 LCO
38		Yes	1	10.000	110.00	C	No	unused	TC19 SM CL W OUT	T19 SCO
39		Yes	10	6.0000	356.46	LITERS	No	unused	DP1 REC VES LEVL	DP1 RVL
40		Yes	11	0.5202	1.5202	G/CC	No	unused	DP2 REC VES SPG	DP2 RVD
41		Yes	12	0.0000	15.000	INCHWC	No	unused	DP3 REC VES VAC	DP3 RVP
42		Yes	13	0.0000	150.00	INCHWC	No	unused	FI7 SM COIL DP	F7 SM C
43		Yes	13	0.0000	150.00	INCHWC	No	unused	FI8 LRG COIL DP	F8 LG C
44		Yes	5	0.5000	1.5000	G/ML	No	unused	ILCM FINAL SPGR	DP6 FVD
45		Yes	15	0.0000	47.000	IN WC	No	unused	ILCM FINAL LEVEL	DP7 FVL
46		Yes	18	-2.000	2.0000	VOLTS	No	unused	HF1 COL1 1FT	HF1 ICM
47		Yes	1	10.000	110.00	C	No	unused	TC1 COLUMN1 ILCM	TC1 ICM
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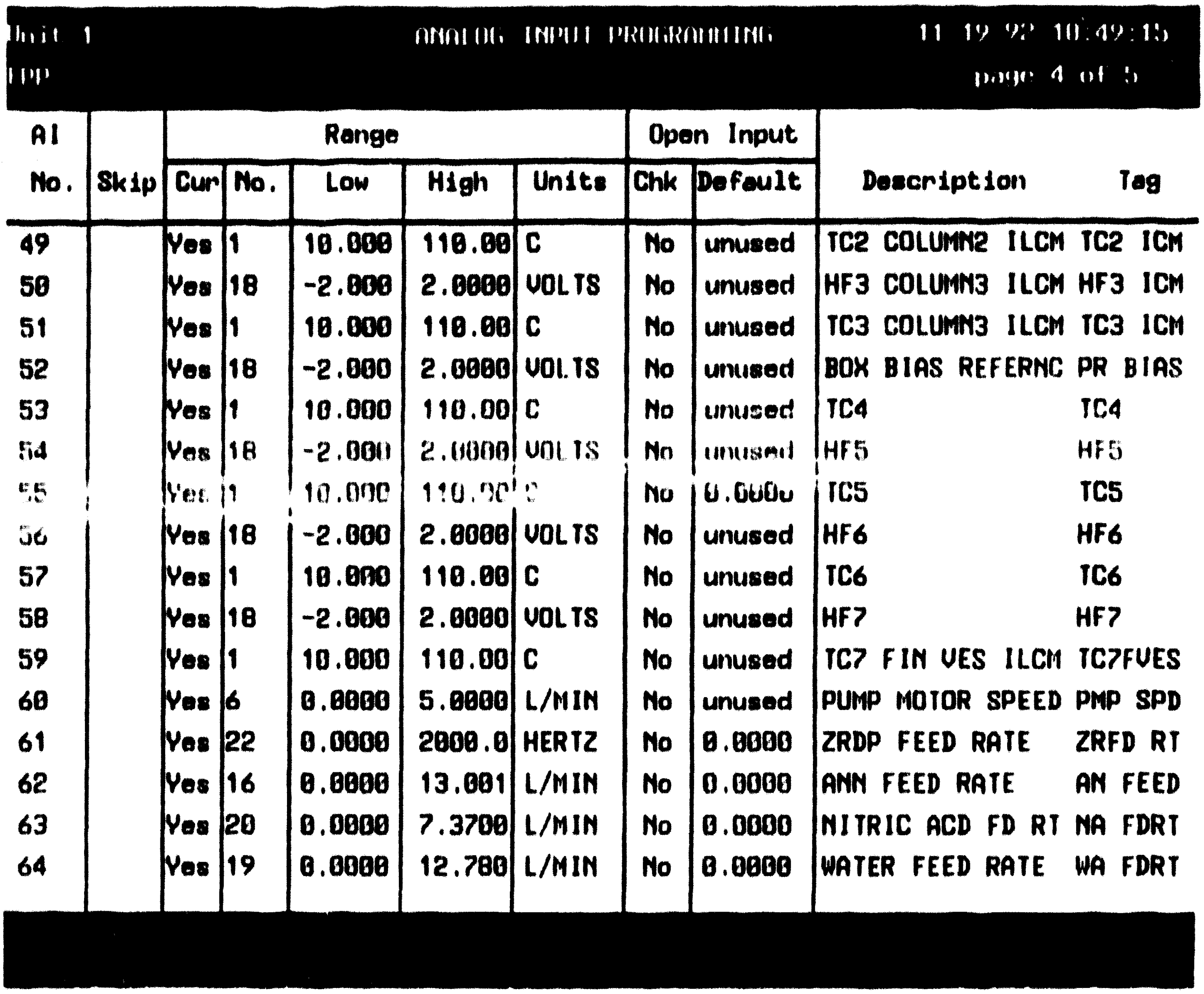

Unit 1 ANALOG INPUT PROGRAMMING 11 19 92 10:49:15

IDP page 4 of 5

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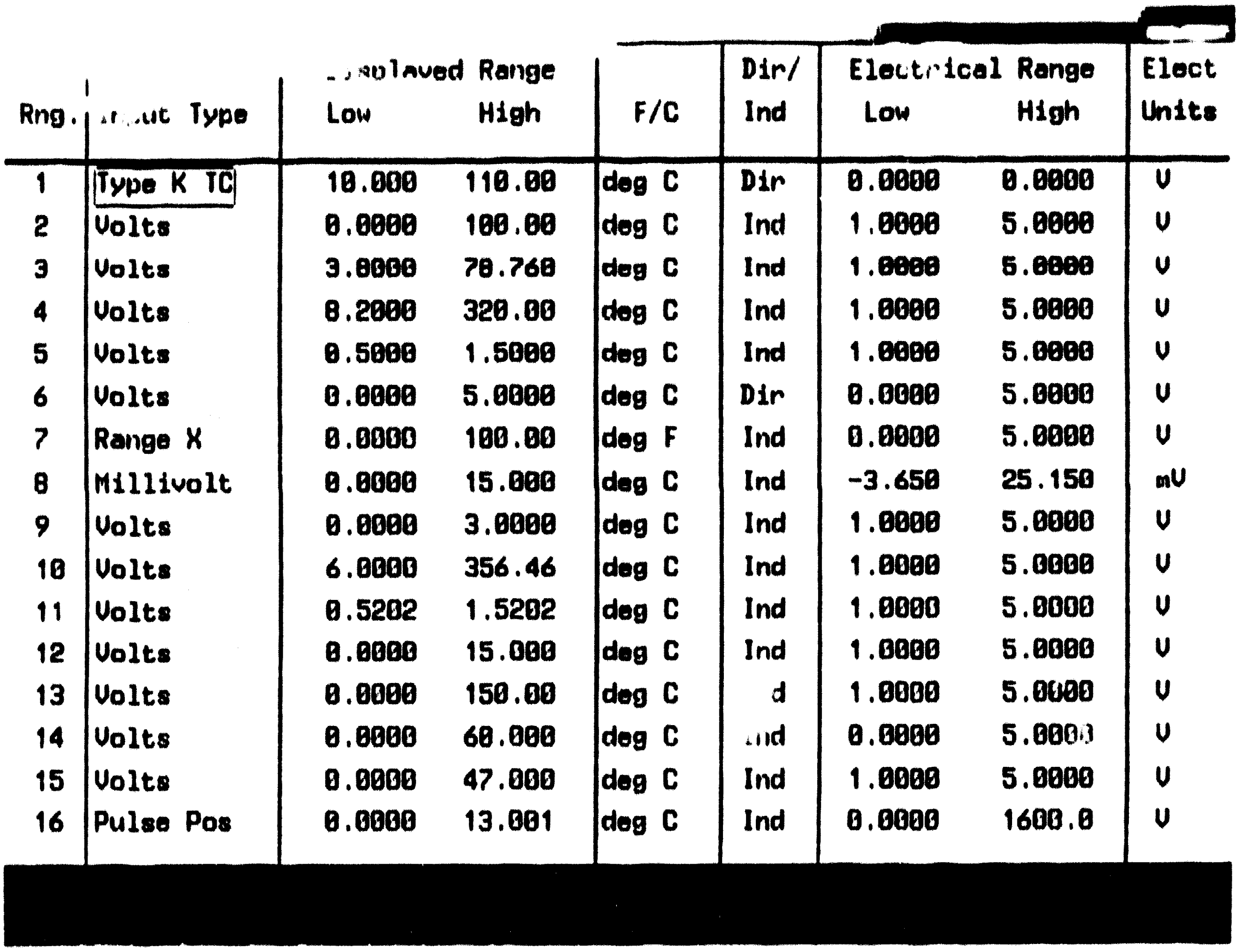
51		Yes	1	10.000	110.00	C	No	unused	TC3 COLUMN3 ILCM	TC3 ICM
52		Yes	18	-2.000	2.0000	VOLTS	No	unused	BOX BIAS REFERNC	PR BIAS
53		Yes	1	10.000	110.00	C	No	unused	TC4	TC4
54		Yes	18	-2.000	2.0000	VOLTS	No	unused	HF5	HF5
55		Yes	1	10.000	110.00	C	No	0.0000	TC5	TC5
56		Yes	18	-2.000	2.0000	VOLTS	No	unused	HF6	HF6
57		Yes	1	10.000	110.00	C	No	unused	TC6	TC6
58		Yes	18	-2.000	2.0000	VOLTS	No	unused	HF7	HF7
59		Yes	1	10.000	110.00	C	No	unused	TC7 FIN VES ILCM	TC7FVES
60		Yes	6	0.0000	5.0000	L/MIN	No	unused	PUMP MOTOR SPEED	PMP SPD
61		Yes	22	0.0000	2000.0	HERTZ	No	0.0000	ZRDP FEED RATE	ZRFD RT
62		Yes	16	0.0000	13.001	L/MIN	No	0.0000	ANN FEED RATE	AN FEED
63		Yes	20	0.0000	7.3700	L/MIN	No	0.0000	NITRIC ACD FD RT	NA FDRT
64		Yes	19	0.0000	12.780	L/MIN	No	0.0000	WATER FEED RATE	WA FDRT



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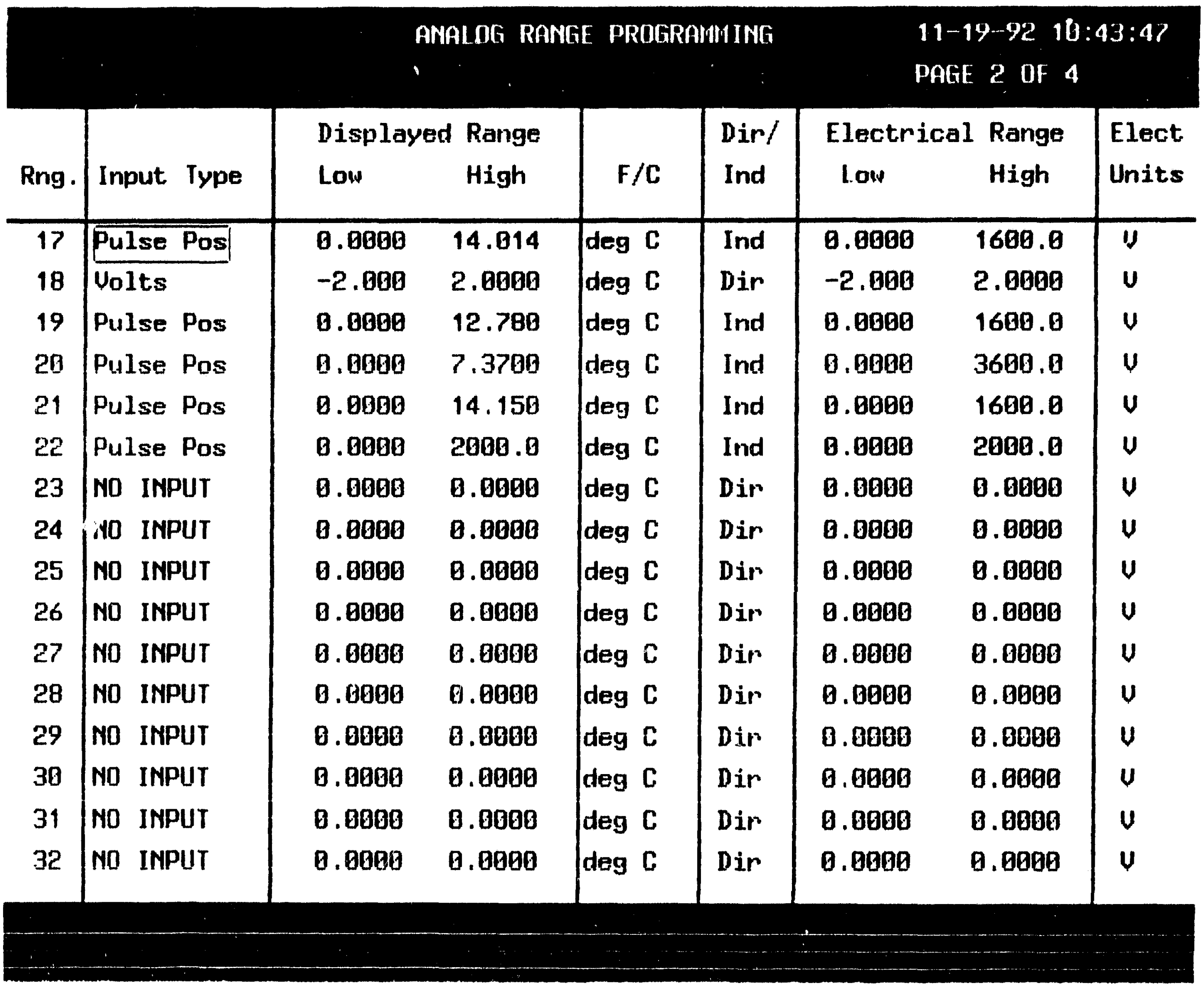
2	Volts	0.0000	100.00	deg C	Ind	1.0000	5.0000	V
3	Volts	3.0000	70.760	deg C	Ind	1.0000	5.0000	V
4	Volts	0.2000	320.00	deg C	Ind	1.0000	5.0000	V
5	Volts	0.5000	1.5000	deg C	Ind	1.0000	5.0000	V
6	Volts	0.0000	5.0000	deg C	Dir	0.0000	5.0000	V
7	Range X	0.0000	100.00	deg F	Ind	0.0000	5.0000	V
8	Millivolt	0.0000	15.000	deg C	Ind	-3.650	25.150	mV
9	Volts	0.0000	3.0000	deg C	Ind	1.0000	5.0000	V
10	Volts	6.0000	356.46	deg C	Ind	1.0000	5.0000	V
11	Volts	0.5202	1.5202	deg C	Ind	1.0000	5.0000	V
12	Volts	0.0000	15.000	deg C	Ind	1.0000	5.0000	V
13	Volts	0.0000	150.00	deg C	Ind	1.0000	5.0000	V
14	Volts	0.0000	60.000	deg C	Ind	0.0000	5.0000	V
15	Volts	0.0000	47.000	deg C	Ind	1.0000	5.0000	V
16	Pulse Pos	0.0000	13.001	deg C	Ind	0.0000	1600.0	V



ANALOG RANGE PROGRAMMING 11-19-92 10:43:47

PAGE 2 OF 4

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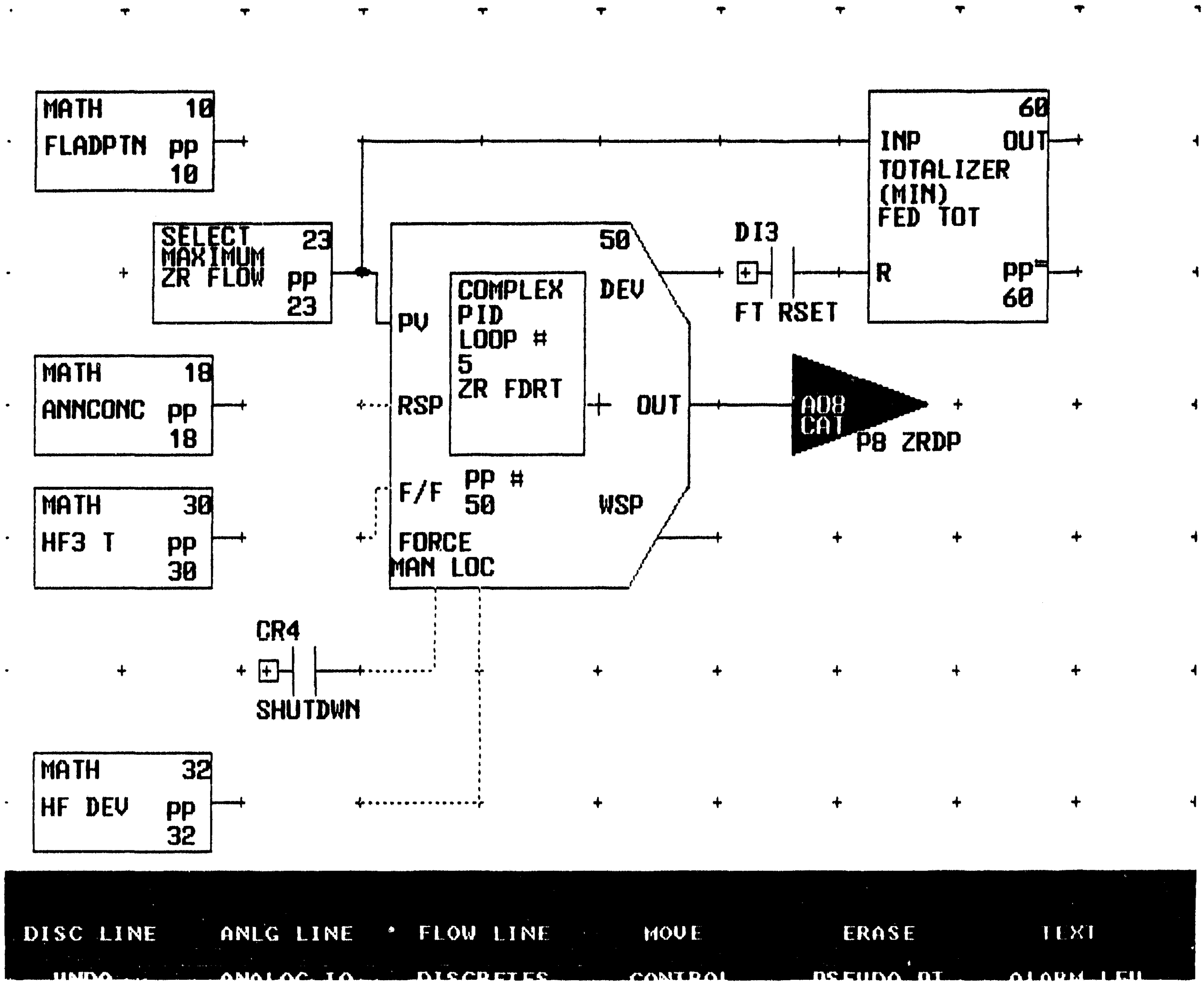
18	Volts	-2.000	2.0000	deg C	Dir	-2.000	2.0000	V
19	Pulse Pos	0.0000	12.780	deg C	Ind	0.0000	1600.0	V
20	Pulse Pos	0.0000	7.3700	deg C	Ind	0.0000	3600.0	V
21	Pulse Pos	0.0000	14.150	deg C	Ind	0.0000	1600.0	V
22	Pulse Pos	0.0000	2000.0	deg C	Ind	0.0000	2000.0	V
23	NO INPUT	0.0000	0.0000	deg C	Dir	0.0000	0.0000	V
24	NO INPUT	0.0000	0.0000	deg C	Dir	0.0000	0.0000	V
25	NO INPUT	0.0000	0.0000	deg C	Dir	0.0000	0.0000	V
26	NO INPUT	0.0000	0.0000	deg C	Dir	0.0000	0.0000	V
27	NO INPUT	0.0000	0.0000	deg C	Dir	0.0000	0.0000	V
28	NO INPUT	0.0000	0.0000	deg C	Dir	0.0000	0.0000	V
29	NO INPUT	0.0000	0.0000	deg C	Dir	0.0000	0.0000	V
30	NO INPUT	0.0000	0.0000	deg C	Dir	0.0000	0.0000	V
31	NO INPUT	0.0000	0.0000	deg C	Dir	0.0000	0.0000	V
32	NO INPUT	0.0000	0.0000	deg C	Dir	0.0000	0.0000	V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pp 23
MATH 18
ANNCONC pp 18
MATH 30
HF3 T pp 30
MATH 32
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50
PV
RSP
F/F
FORCE
MAN LOC
COMPLEX PID LOOP # 5 ZR FDRT
PP # 50
DEV
OUT
WSP
DI3
FT RSET
60
INP
OUT
TOTALIZER (MIN) FED TOT
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PP
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AO8 CAT
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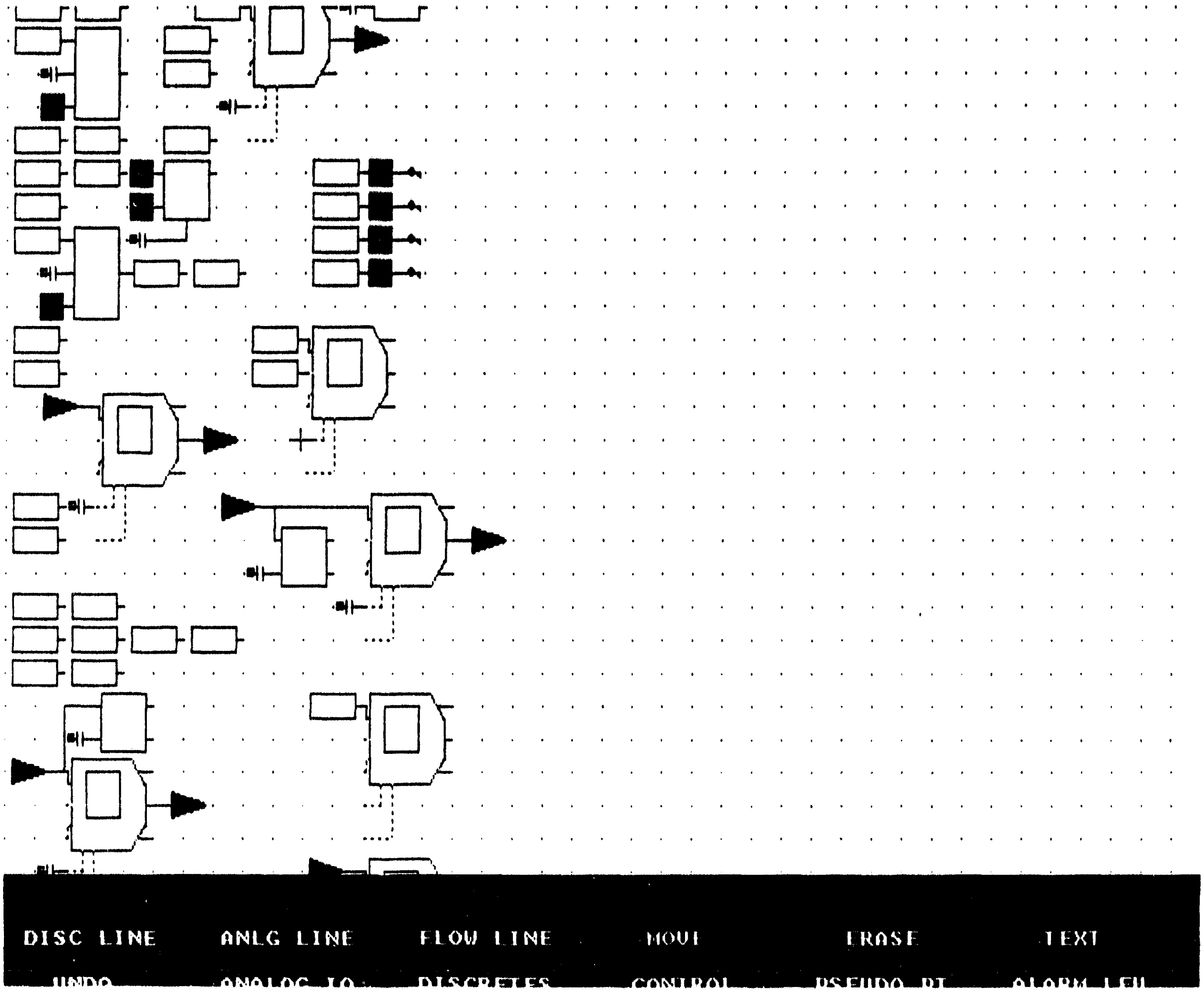
SHUTDWN
DISC LINE
ANLG LINE
FLOW LINE
MOVE
ERASE
TEXT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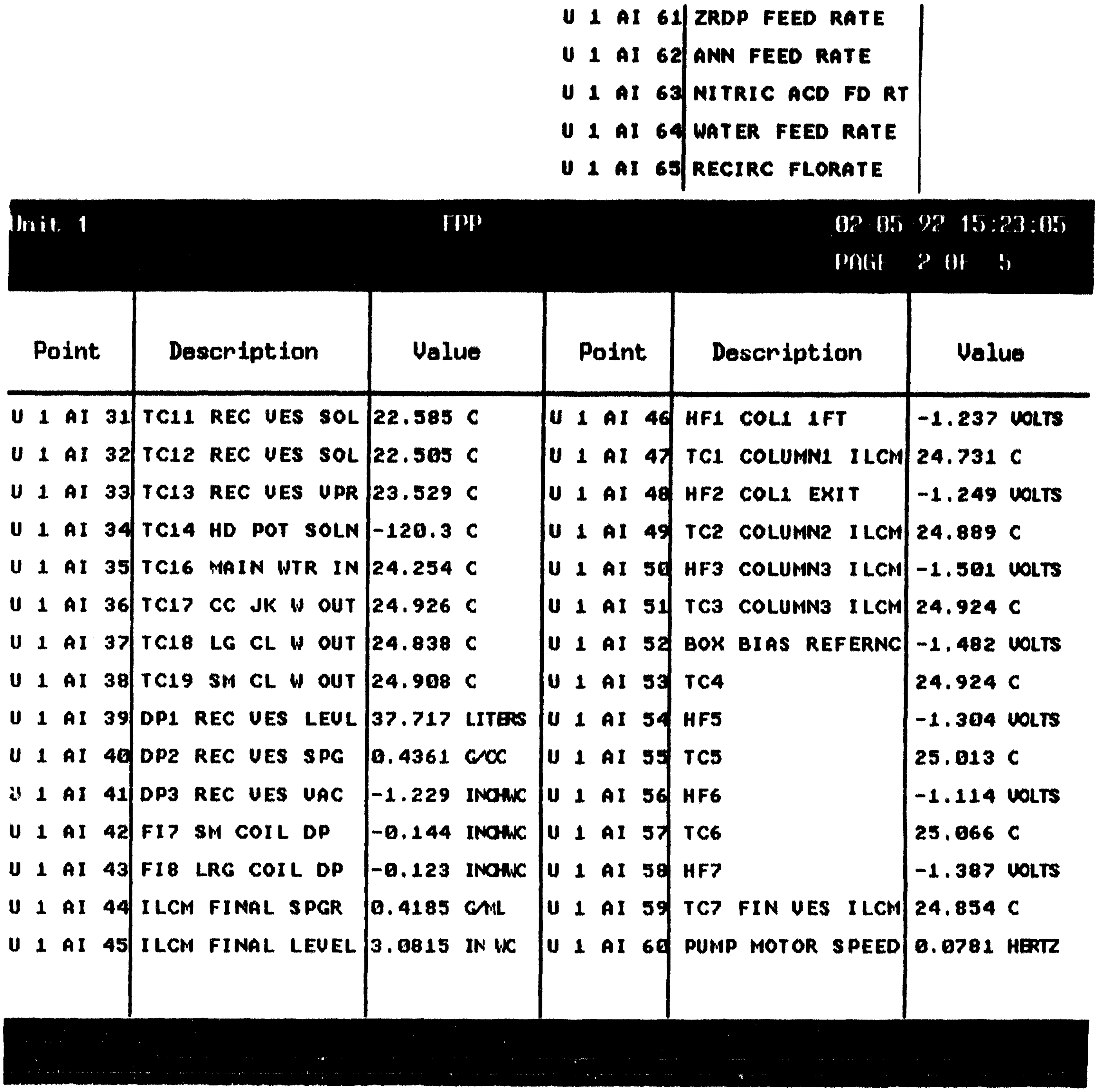
CONTROL
PSEUDO PT
ALARM LEV

Attachment II
DAC-11-92
November 5, 1992

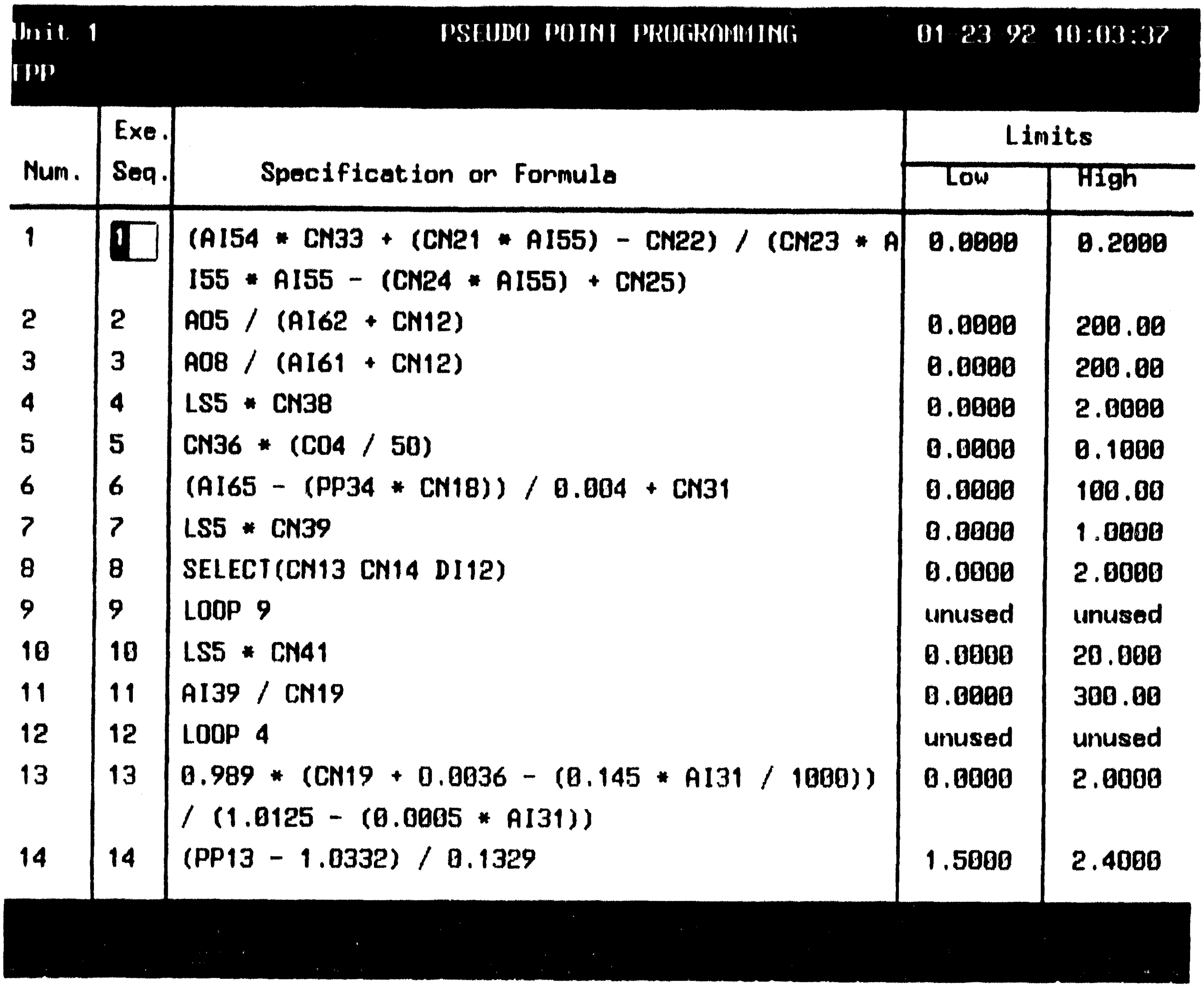


U 1 AI 61	ZRDP FEED RATE
U 1 AI 62	ANN FEED RATE
U 1 AI 63	NITRIC ACD FD RT
U 1 AI 64	WATER FEED RATE
U 1 AI 65	RECIRC FLORATE

Unit 1 FPP 02 05 92 15:23:05
PAGE 2 OF 5

Point	Description	Value	Point	Description	Value
U 1 AI 31	TC11 REC VES SOL	22.585 C	U 1 AI 46	HF1 COL1 1FT	-1.237 VOLTS
U 1 AI 32	TC12 REC VES SOL	22.505 C	U 1 AI 47	TC1 COLUMN1 ILCM	24.731 C
U 1 AI 33	TC13 REC VES VPR	23.529 C	U 1 AI 48	HF2 COL1 EXIT	-1.249 VOLTS
U 1 AI 34	TC14 HD POT SOLN	-120.3 C	U 1 AI 49	TC2 COLUMN2 ILCM	24.889 C
U 1 AI 35	TC16 MAIN WTR IN	24.254 C	U 1 AI 50	HF3 COLUMN3 ILCM	-1.501 VOLTS
U 1 AI 36	TC17 CC JK W OUT	24.926 C	U 1 AI 51	TC3 COLUMN3 ILCM	24.924 C
U 1 AI 37	TC18 LG CL W OUT	24.838 C	U 1 AI 52	BOX BIAS REFERNC	-1.482 VOLTS
U 1 AI 38	TC19 SM CL W OUT	24.908 C	U 1 AI 53	TC4	24.924 C
U 1 AI 39	DP1 REC VES LEVL	37.717 LITERS	U 1 AI 54	HF5	-1.304 VOLTS
U 1 AI 40	DP2 REC VES SPG	0.4361 G/CC	U 1 AI 55	TC5	25.013 C
U 1 AI 41	DP3 REC VES VAC	-1.229 INCHWC	U 1 AI 56	HF6	-1.114 VOLTS
U 1 AI 42	FI7 SM COIL DP	-0.144 INCHWC	U 1 AI 57	TC6	25.066 C
U 1 AI 43	FI8 LRG COIL DP	-0.123 INCHWC	U 1 AI 58	HF7	-1.387 VOLTS
U 1 AI 44	ILCM FINAL SPGR	0.4185 G/ML	U 1 AI 59	TC7 FIN VES ILCM	24.854 C
U 1 AI 45	ILCM FINAL LEVEL	3.0815 IN WC	U 1 AI 60	PUMP MOTOR SPEED	0.0781 HERTZ

PP 1	(HF) AT T PR 5	
PP 2	ANN FLOW RATIO	Calculate expected value to use for plugged meter detection
PP 3	ZR FEED FLO RATO	Similar calc for Zr feed
PP 4	ADAPT TUNE L2ANM	PID tuning value proportional to feedrate
PP 5	REM SP LP2 ANMID	Scale dfrom outer loop CO4 transfers SP to loop 2 mid cascade
PP 6	FRCBCK L6 FANOUT	Solving for CO to set initial value for final ANN loop 6
PP 7	ADP TUN L6FANOUT	Value proportional to feedrate similar to #4
PP 8	AN SP FDBCK FCTR	0,1 selector driven by DI12. On sets to 1 to include FB in AN flow
PP 9	LOOP	
PP 10	FLOW ADAPT I TUN	Flow proportional PID setting
PP 11	FEED VES LEVEL	Gauge DP adjusted for SPG gives liters
PP 12	LOOP4 ILCM HEAT	
PP 13	SPGR AT 25C ILCM	Temp correction for SPG provided by JAM, uses const SPG to elim noise
PP 14	ZR CONC ILCM	JAM eqn giving [Zr] from SPG for feedforward control



Unit 1 PSEUDO POINT PROGRAMMING 01-23-92 10:03:37

FPP

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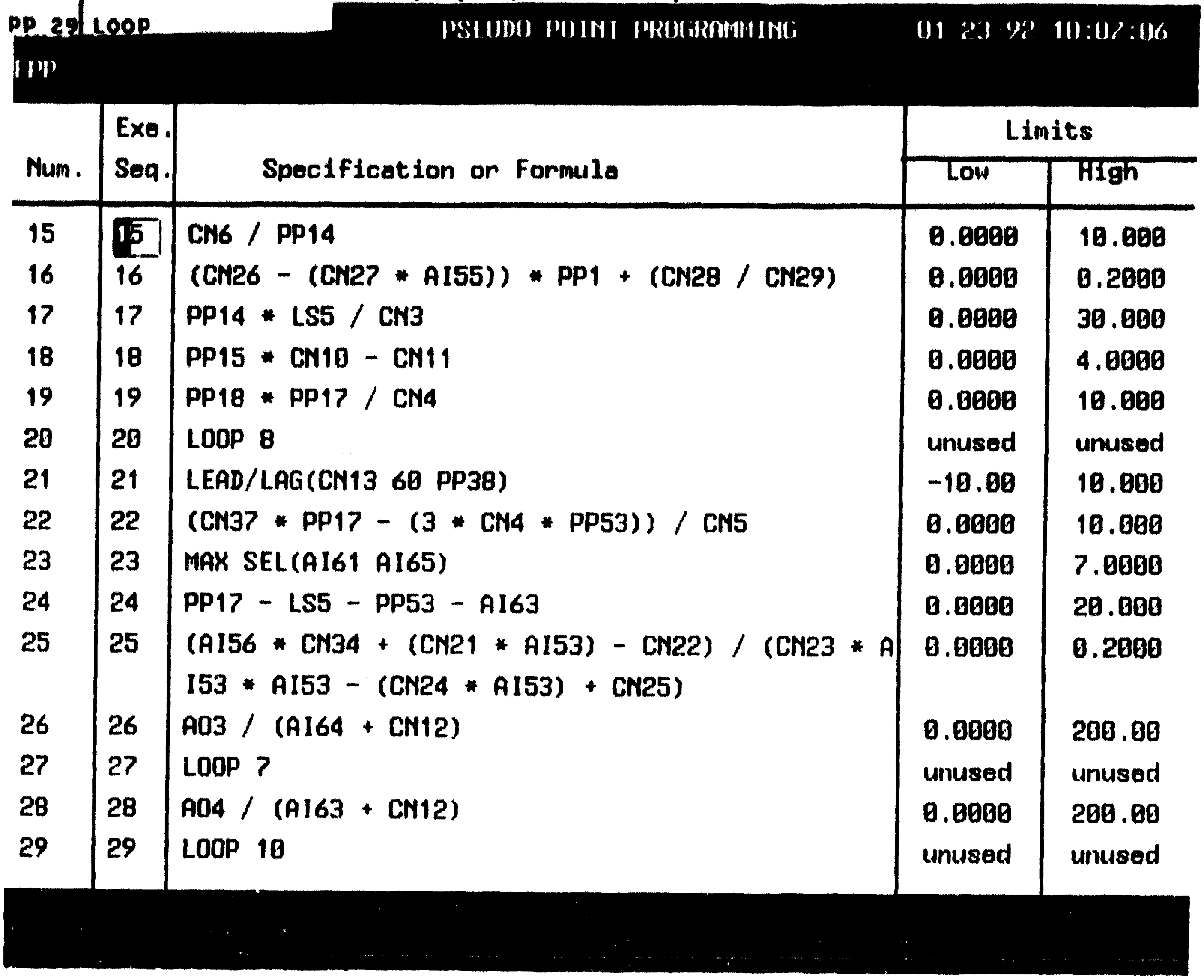
2	2	AO5 / (AI62 + CN12)	0.0000	200.00
3	3	AO8 / (AI61 + CN12)	0.0000	200.00
4	4	LS5 * CN38	0.0000	2.0000
5	5	CN36 * (CO4 / 50)	0.0000	0.1000
6	6	(AI65 - (PP34 * CN18)) / 0.004 + CN31	0.0000	100.00
7	7	LS5 * CN39	0.0000	1.0000
8	8	SELECT(CN13 CN14 DI12)	0.0000	2.0000
9	9	LOOP 9	unused	unused
10	10	LS5 * CN41	0.0000	20.000
11	11	AI39 / CN19	0.0000	300.00
12	12	LOOP 4	unused	unused
13	13	0.989 * (CN19 + 0.0036 - (0.145 * AI31 / 1000)) / (1.0125 - (0.0005 * AI31))	0.0000	2.0000
14	14	(PP13 - 1.0332) / 0.1329	1.5000	2.4000

PP 15	F/ZR RATIO ILCM	CN6 is fluoride estimate given by GFD run
PP 16	(HF) AT 35 PR 5	
PP 17	FIN VOLRATE ILCM	Zr balance gives target product flow: feed[Zr]*rate/target[Zr]
PP 18	ANN CONC ILCM	Linear estimate for product [Al] based on F/Zr
PP 19	ANN FD FWD DMND	ANN feedforward flow SP = prod [Al]*prod flow/[ANN]
PP 20	LOOP	
PP 21	FEED AVG RATE	Dampened rate of change of level reading is feedrate
PP 22	NA SETPOINTILCM	NO3 bal: NA flow=(prod NO3*flow-ANN contrib)/reagent [NA]
PP 23	ZRDP FLOW MAX SL	Selects the higher of 2 flowmeters in series allows for 1 to jam
PP 24	H2O SETPOINTILCM	Water setpoint is total target flow minus all other flows
PP 25	(HF) AT T PR 6	
PP 26	WATER FLOW RATIO	Water pump output to flowrate comparison for error detection
PP 27	LOOP	
PP 28	NITRIC FLOW RATO	Nitric pump output to flow comparison
PP 29	LOOP	

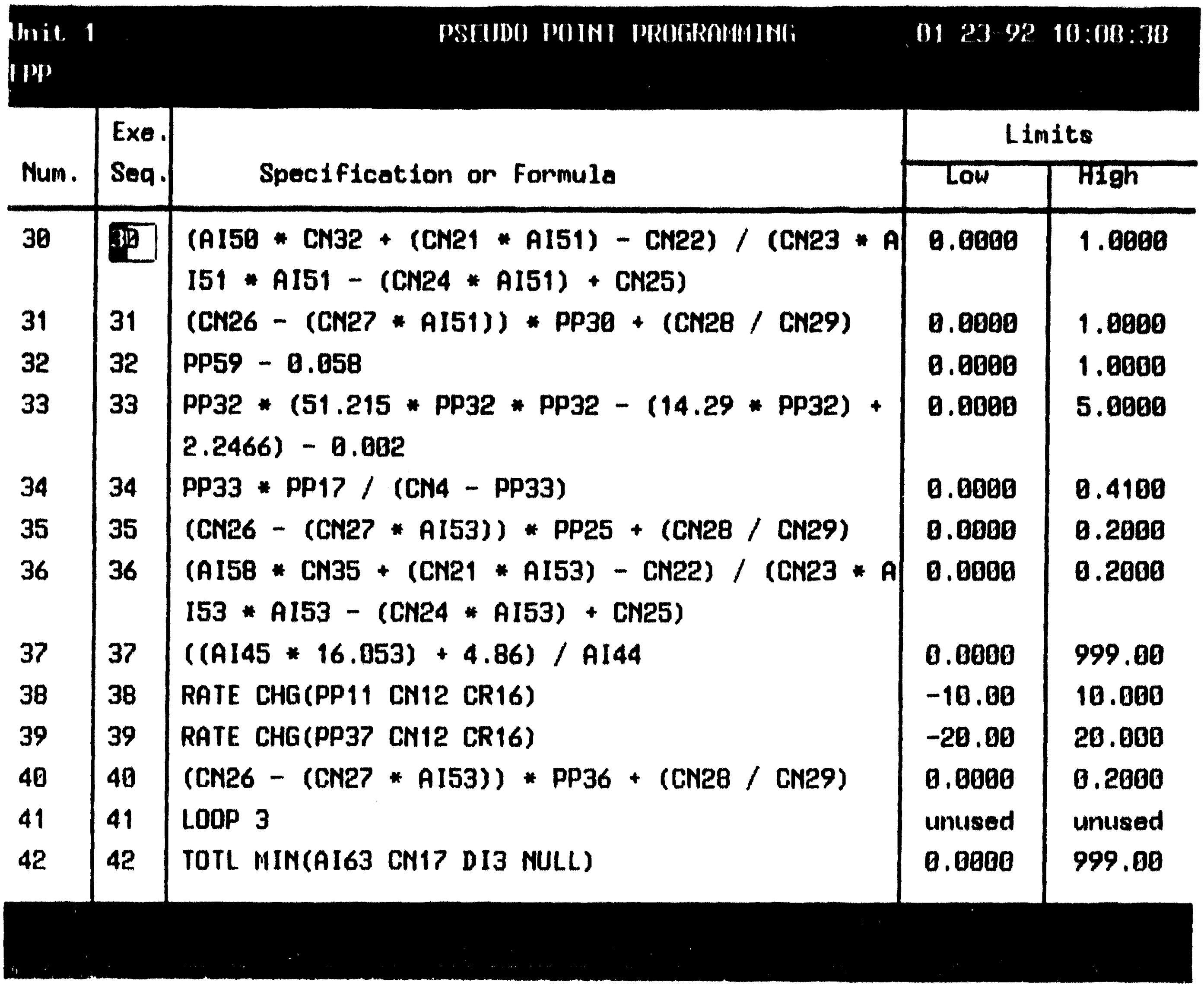


PSUDO POINT PROGRAMMING 01-23-92 10:07:06

PP

Num.	Exe. Seq.	Specification or Formula	Limits Low	Limits High
15	15	CN6 / PP14	0.0000	10.000
16	16	(CN26 - (CN27 * AI55)) * PP1 + (CN28 / CN29)	0.0000	0.2000
17	17	PP14 * LS5 / CN3	0.0000	30.000
18	18	PP15 * CN10 - CN11	0.0000	4.0000
19	19	PP18 * PP17 / CN4	0.0000	10.000
20	20	LOOP 8	unused	unused
21	21	LEAD/LAG(CN13 60 PP38)	-10.00	10.000
22	22	(CN37 * PP17 - (3 * CN4 * PP53)) / CN5	0.0000	10.000
23	23	MAX SEL(AI61 AI65)	0.0000	7.0000
24	24	PP17 - LS5 - PP53 - AI63	0.0000	20.000
25	25	(AI56 * CN34 + (CN21 * AI53) - CN22) / (CN23 * A I53 * AI53 - (CN24 * AI53) + CN25)	0.0000	0.2000
26	26	AO3 / (AI64 + CN12)	0.0000	200.00
27	27	LOOP 7	unused	unused
28	28	AO4 / (AI63 + CN12)	0.0000	200.00
29	29	LOOP 10	unused	unused

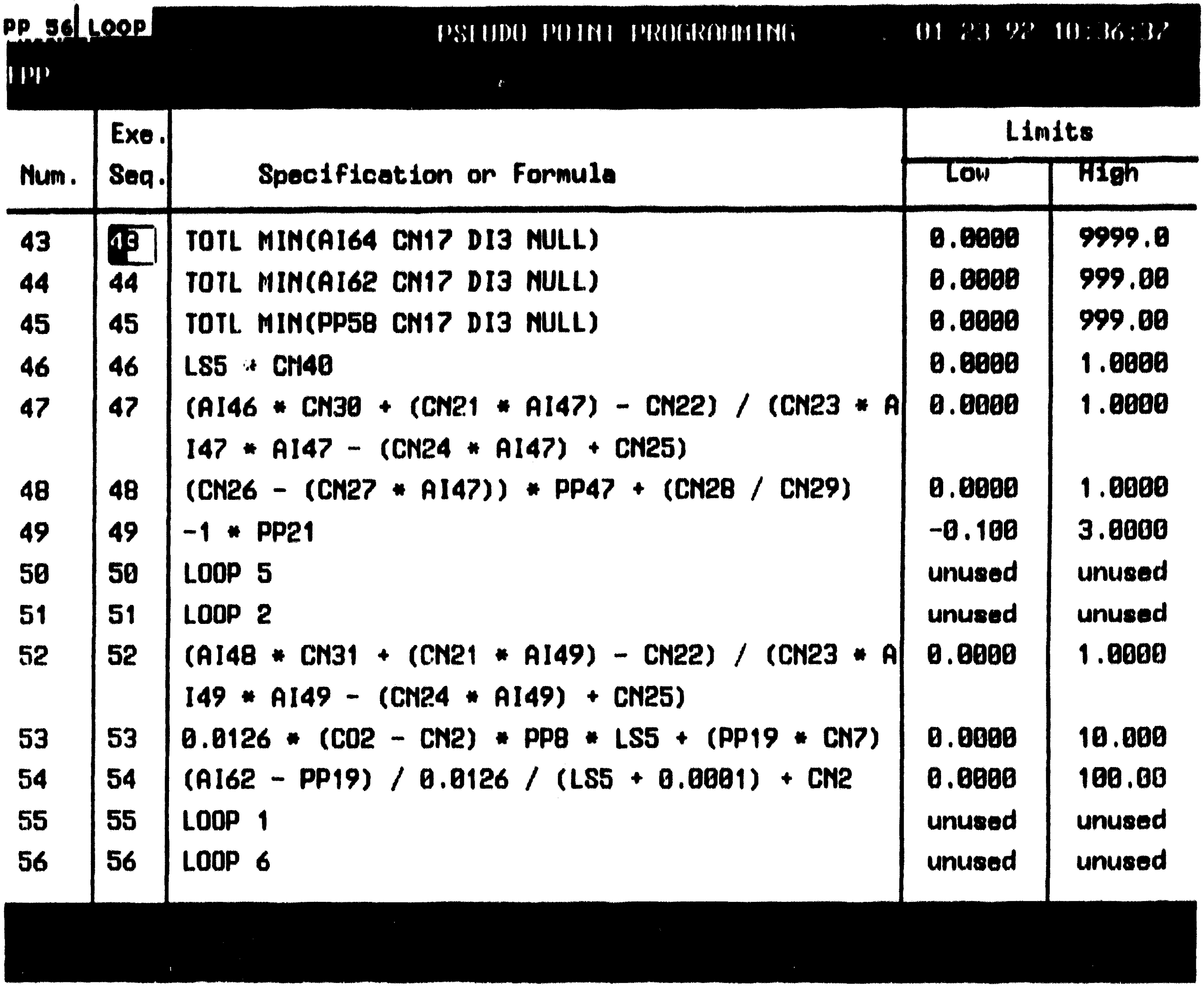
PP 30	(HF)PR3 T	
PP 31	(HF)PR3 35	
PP 32	(HF) DEVIATION	Probe 2 HF in excess of .058, set high to avoid xs AN during cycles
PP 33	Y FACTOR	ANN molar deficit calc from JAM gives final ANN correction
PP 34	AN ADJUST FLOW	Final ANN feedforward addition l/min
PP 35	(HF) AT 35 PR 6	
PP 36	(HF) AT T PR 7	
PP 37	FCV VOL CALC	Converts DP level and density to liters
PP 38	REC VES OUTFLOW	Level depletion rate is flowrate
PP 39	COL VES INFLOW	Level depletion rate is flowrate
PP 40	(HF) AT 35 PR 7	
PP 41	LOOP	
PP 42	NA FLOW TOTAL	Integrator for nitric acid flowrate



Unit 1 PSEUDO POINT PROGRAMMING 01 23 92 10:08:38
PP

Num.	Exe. Seq.	Specification or Formula	Limits Low	Limits High
30	30	(AI50 * CN32 + (CN21 * AI51) - CN22) / (CN23 * AI51 * AI51 - (CN24 * AI51) + CN25)	0.0000	1.0000
31	31	(CN26 - (CN27 * AI51)) * PP30 + (CN28 / CN29)	0.0000	1.0000
32	32	PP59 - 0.058	0.0000	1.0000
33	33	PP32 * (51.215 * PP32 * PP32 - (14.29 * PP32) + 2.2466) - 0.002	0.0000	5.0000
34	34	PP33 * PP17 / (CN4 - PP33)	0.0000	0.4100
35	35	(CN26 - (CN27 * AI53)) * PP25 + (CN28 / CN29)	0.0000	0.2000
36	36	(AI58 * CN35 + (CN21 * AI53) - CN22) / (CN23 * AI53 * AI53 - (CN24 * AI53) + CN25)	0.0000	0.2000
37	37	((AI45 * 16.053) + 4.86) / AI44	0.0000	999.00
38	38	RATE CHG(PP11 CN12 CR16)	-10.00	10.000
39	39	RATE CHG(PP37 CN12 CR16)	-20.00	20.000
40	40	(CN26 - (CN27 * AI53)) * PP36 + (CN28 / CN29)	0.0000	0.2000
41	41	LOOP 3	unused	unused
42	42	TOTL MIN(AI63 CN17 DI3 NULL)	0.0000	999.00

pp 43	WATER FLOW TOTAL	Integrator for water flow
pp 44	ANN FLOW TOT	Integrator for ANN flow
pp 45	ANN ADJ FLOW TOT	Integrator for final ANN flow
pp 46	LP2 ADAPTIVE RST	Flow proportional reset value for loop 2
pp 47	PR1 HF T	
pp 48	PR1 HF 35	
pp 49	FEED OUTFLOW	Sign change for level depletion rate
pp 50	ZR FD RATE LP 5	
pp 51	AN FB OUTR LOOP2	
pp 52	PR2 HF T	
pp 53	AN SETPT DEMAND	Scaled combine of FB (lp2 CO) & FF (PP19). CN7&8 are set to 0 or 1 for control
pp 54	FORCBK LP2 ANMID	ANN flow used to solve PP53 for CO2. Used for bumpless startup of LP2.
pp 55	LOOP	
pp 56	LOOP	

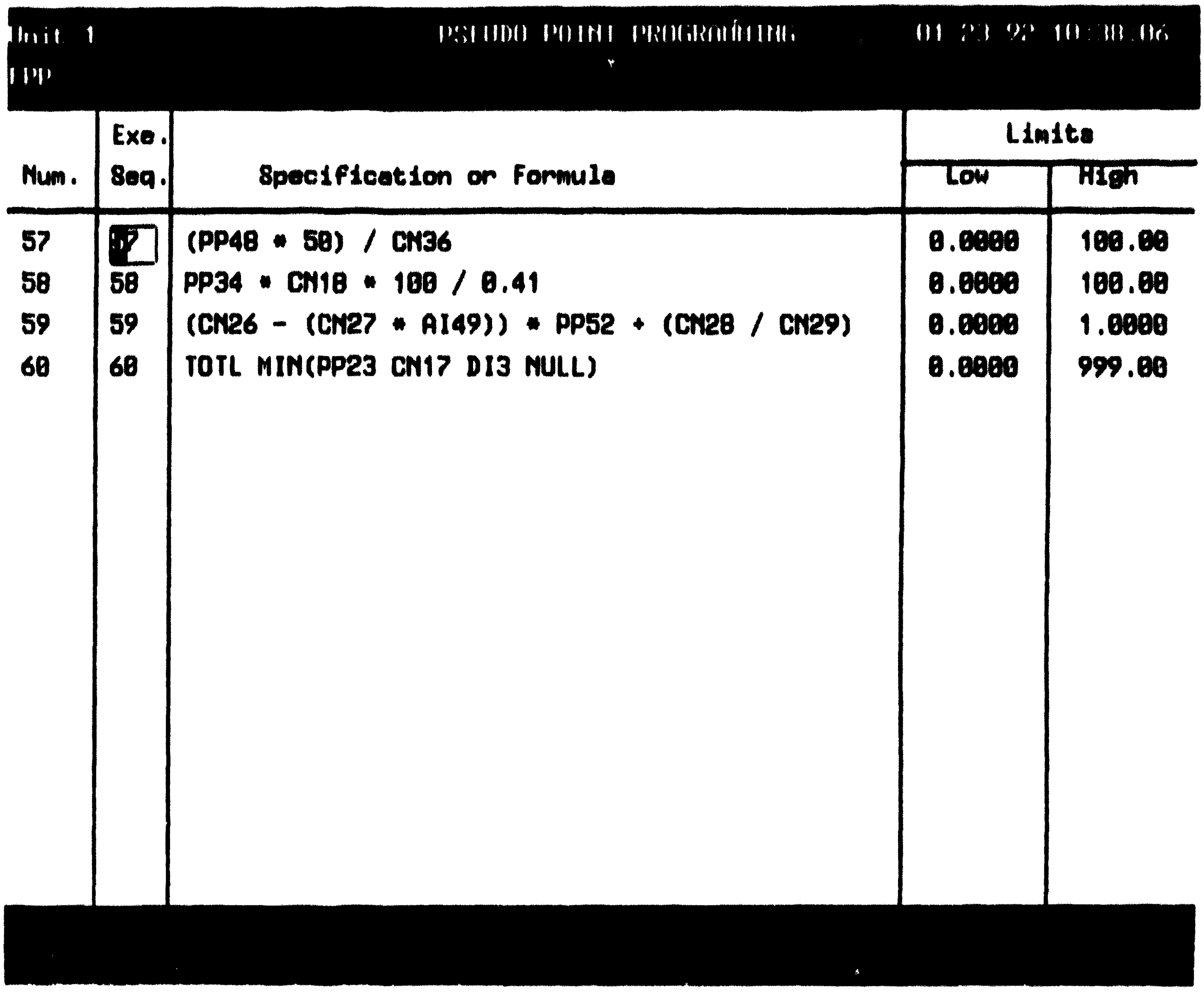


PSEUDO POINT PROGRAMMING 01 23 92 10:36:37

PP

Num.	Exe. Seq.	Specification or Formula	Limits Low	Limits High
43	43	TOTL MIN(AI64 CN17 DI3 NULL)	0.0000	9999.0
44	44	TOTL MIN(AI62 CN17 DI3 NULL)	0.0000	999.00
45	45	TOTL MIN(PP58 CN17 DI3 NULL)	0.0000	999.00
46	46	LS5 * CN40	0.0000	1.0000
47	47	(AI46 * CN30 + (CN21 * AI47) - CN22) / (CN23 * AI47 * AI47 - (CN24 * AI47) + CN25)	0.0000	1.0000
48	48	(CN26 - (CN27 * AI47)) * PP47 + (CN28 / CN29)	0.0000	1.0000
49	49	-1 * PP21	-0.100	3.0000
50	50	LOOP 5	unused	unused
51	51	LOOP 2	unused	unused
52	52	(AI48 * CN31 + (CN21 * AI49) - CN22) / (CN23 * AI49 * AI49 - (CN24 * AI49) + CN25)	0.0000	1.0000
53	53	0.0126 * (CO2 - CN2) * PP8 * LS5 + (PP19 * CN7)	0.0000	10.000
54	54	(AI62 - PP19) / 0.0126 / (LS5 + 0.0001) + CN2	0.0000	100.00
55	55	LOOP 1	unused	unused
56	56	LOOP 6	unused	unused

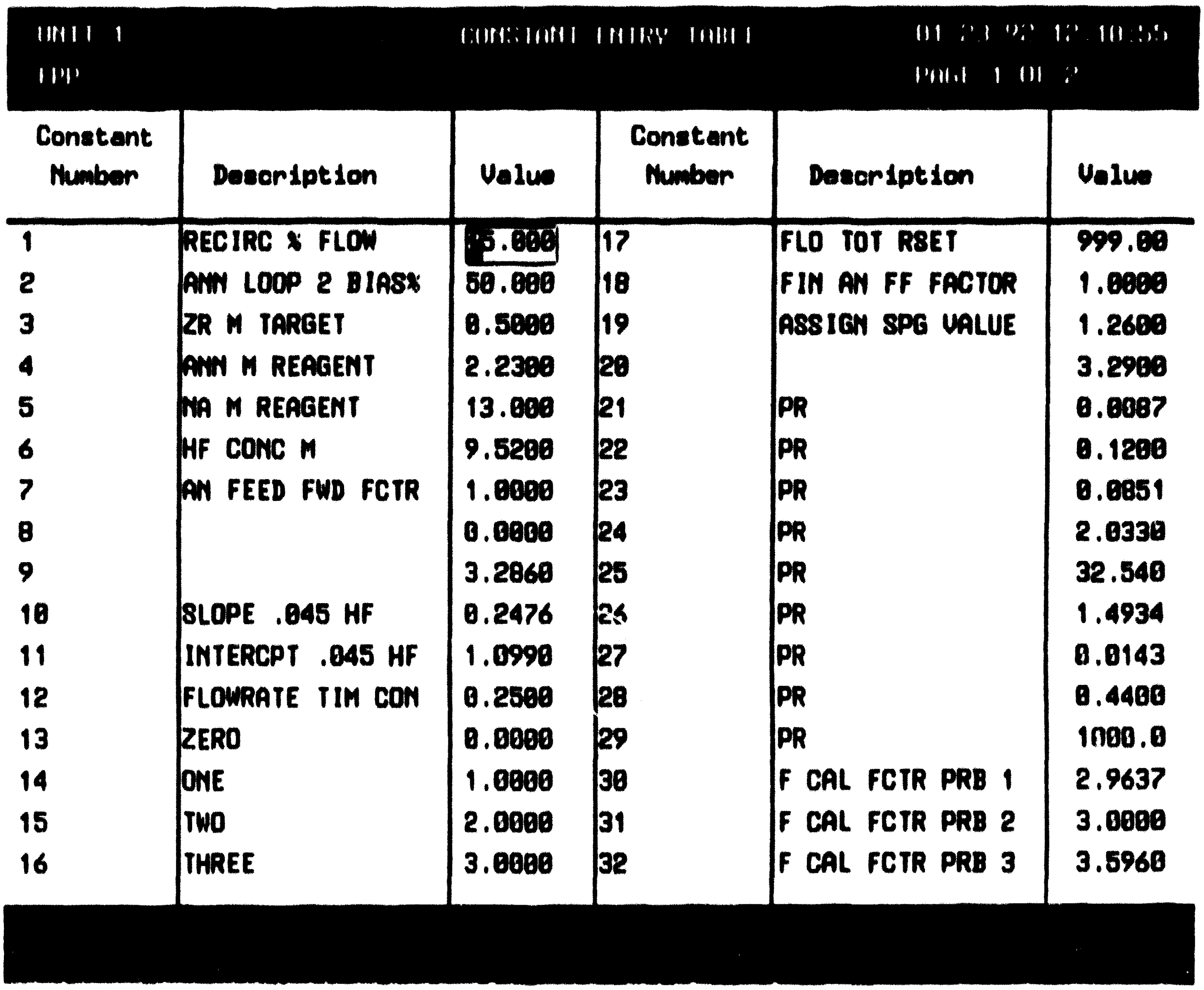
PP 57	FORCBK LP4 ANOUT	Solving for loop 4 initial output for bumpless cascade startup
PP 58	FAN x PUMP OUTPT	Scaled flow signal for final ANN flow pump output 0-100
PP 59	PR2 HF 35	
PP 60	FEED FLOW TOTAL	Integrator for feed flowrate



Unit 1 PSEUDO POINT PROGRAMMING 01 23 92 10:38:06

PPP

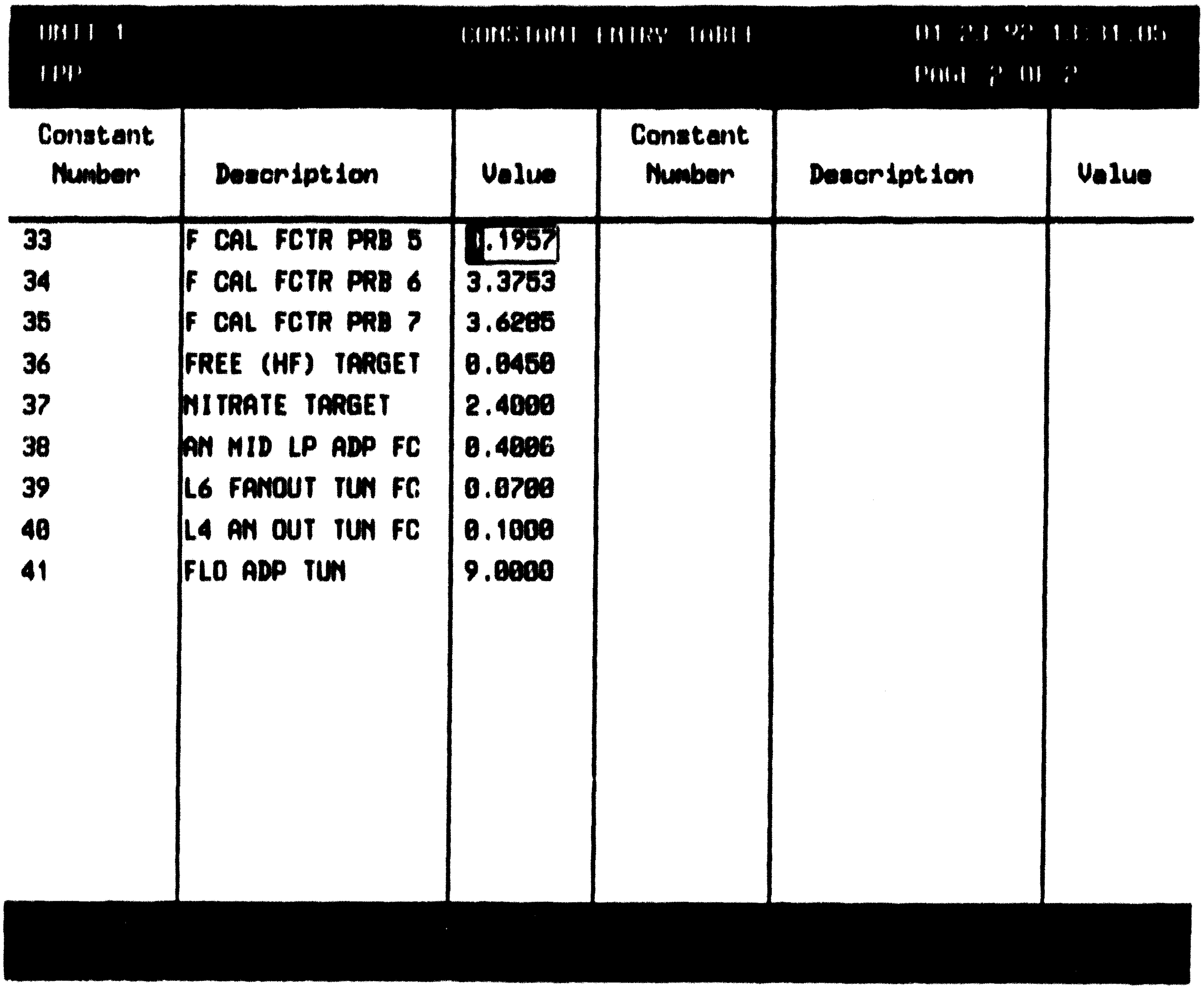
Num.	Exe. Seq.	Specification or Formula	Limits Low	Limits High
57	57	(PP48 * 50) / CN36	0.0000	100.00
58	58	PP34 * CN18 * 100 / 0.41	0.0000	100.00
59	59	(CN26 - (CN27 * AI49)) * PP52 + (CN28 / CN29)	0.0000	1.0000
60	60	TOTL MIN(PP23 CN17 DI3 NULL)	0.0000	999.00



UNIT 1 CONSTANT ENTRY TABLE 01 23 92 12:10:55

FPP PAGE 1 OF 2

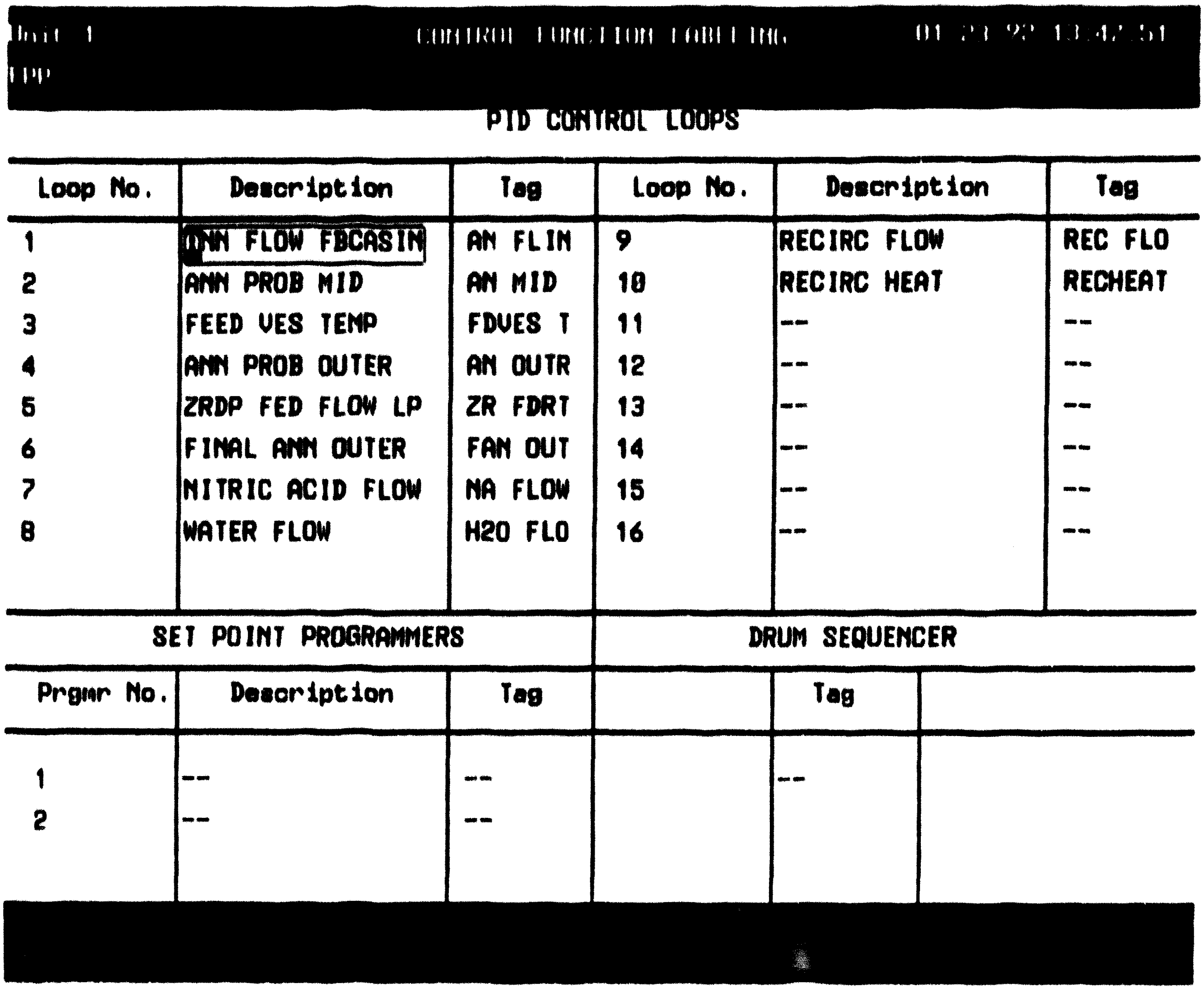
Constant Number	Description	Value	Constant Number	Description	Value
1	RECIRC % FLOW	[illegible]5.000	17	FLO TOT RSET	999.00
2	ANN LOOP 2 BIAS%	50.000	18	FIN AN FF FACTOR	1.0000
3	ZR M TARGET	0.5000	19	ASSIGN SPG VALUE	1.2600
4	ANN M REAGENT	2.2300	20		3.2900
5	NA M REAGENT	13.000	21	PR	0.0087
6	HF CONC M	9.5200	22	PR	0.1200
7	AN FEED FWD FCTR	1.0000	23	PR	0.0851
8		0.0000	24	PR	2.0330
9		3.2860	25	PR	32.540
10	SLOPE .045 HF	0.2476	26	PR	1.4934
11	INTERCPT .045 HF	1.0990	27	PR	0.0143
12	FLOWRATE TIM CON	0.2500	28	PR	0.4400
13	ZERO	0.0000	29	PR	1000.0
14	ONE	1.0000	30	F CAL FCTR PRB 1	2.9637
15	TWO	2.0000	31	F CAL FCTR PRB 2	3.0000
16	THREE	3.0000	32	F CAL FCTR PRB 3	3.5960



UNIT 1 CONSTANT ENTRY TABLE 01 23 92 13:31:05

EPP PAGE 2 OF 2

Constant Number	Description	Value	Constant Number	Description	Value
33	F CAL FCTR PRB 5	1.1957			
34	F CAL FCTR PRB 6	3.3753			
35	F CAL FCTR PRB 7	3.6285			
36	FREE (HF) TARGET	0.0450			
37	NITRATE TARGET	2.4000			
38	AN MID LP ADP FC	0.4006			
39	L6 FANOUT TUN FC	0.0700			
40	L4 AN OUT TUN FC	0.1000			
41	FLO ADP TUN	9.0000			



Unit 1 CONTROL FUNCTION LABELING 01 23 92 13:42:51
IPP

PID CONTROL LOOPS

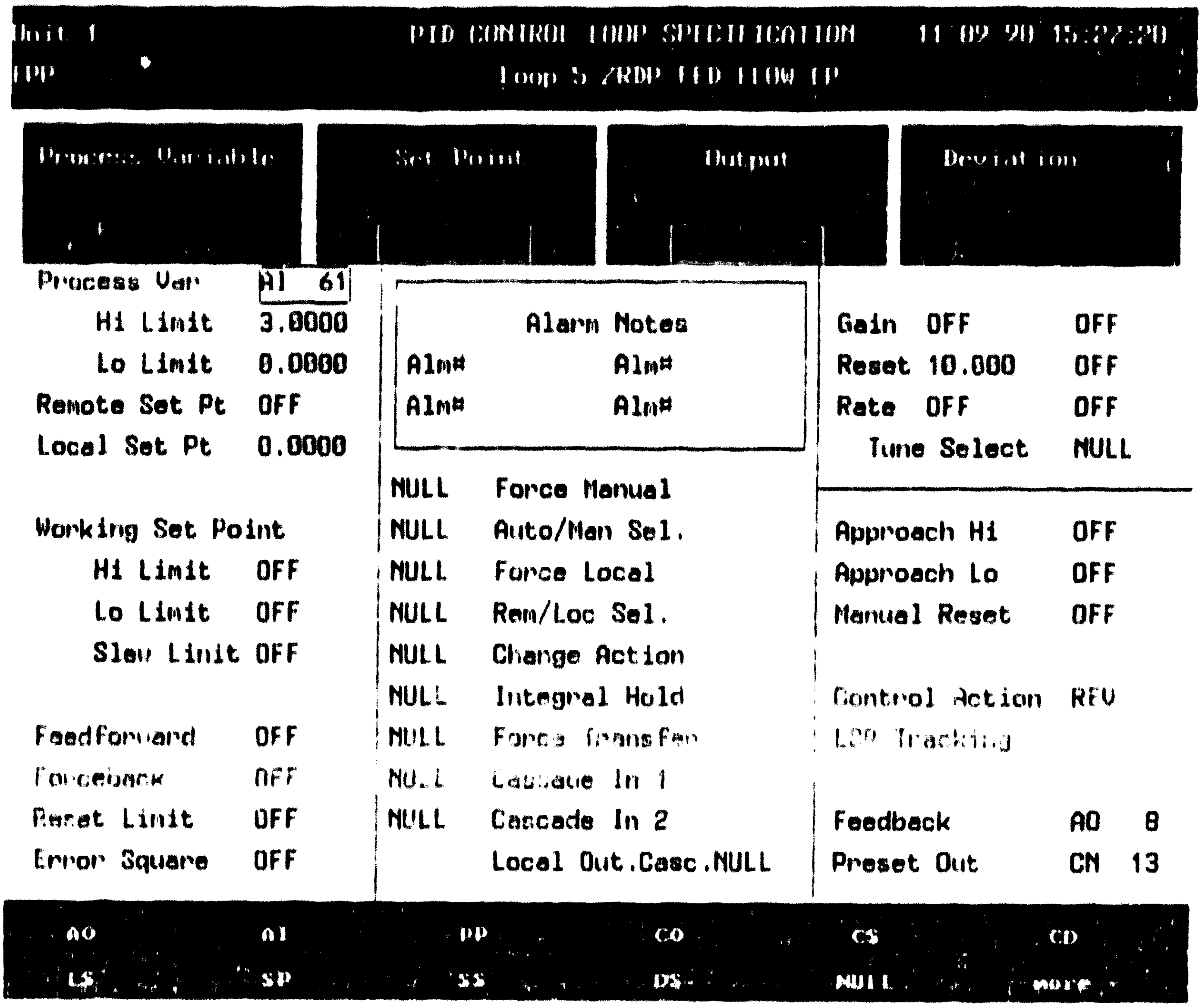
Loop No.	Description	Tag	Loop No.	Description	Tag
1	ANN FLOW FBCASIN	AN FLIN	9	RECIRC FLOW	REC FLO
2	ANN PROB MID	AN MID	10	RECIRC HEAT	RECHEAT
3	FEED VES TEMP	FDVES T	11	--	--
4	ANN PROB OUTER	AN OUTR	12	--	--
5	ZRDP FED FLOW LP	ZR FDRT	13	--	--
6	FINAL ANN OUTER	FAN OUT	14	--	--
7	NITRIC ACID FLOW	NA FLOW	15	--	--
8	WATER FLOW	H2O FLO	16	--	--

SET POINT PROGRAMMERS

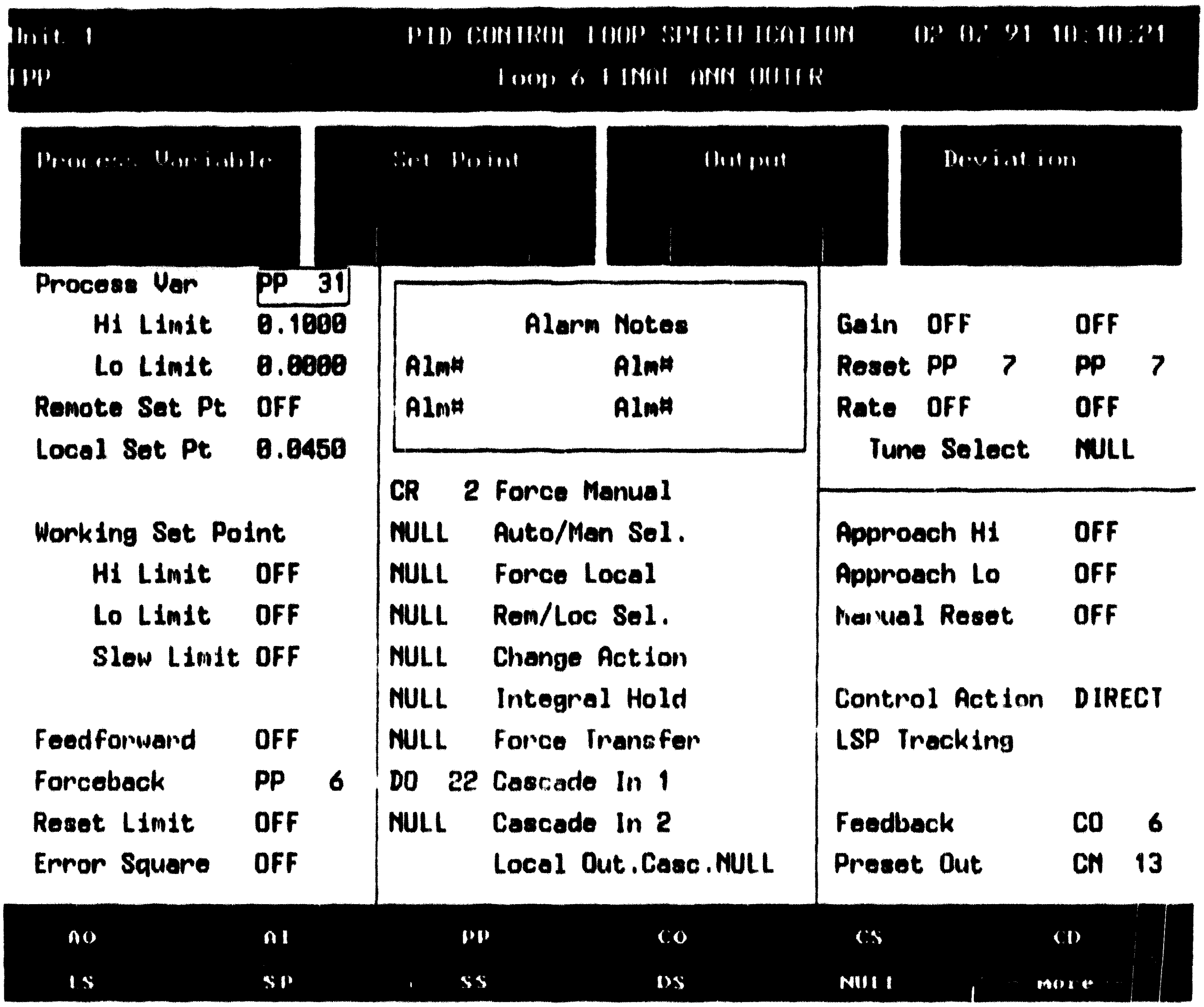
Prgmr No.	Description	Tag
1	--	--
2	--	--

DRUM SEQUENCER

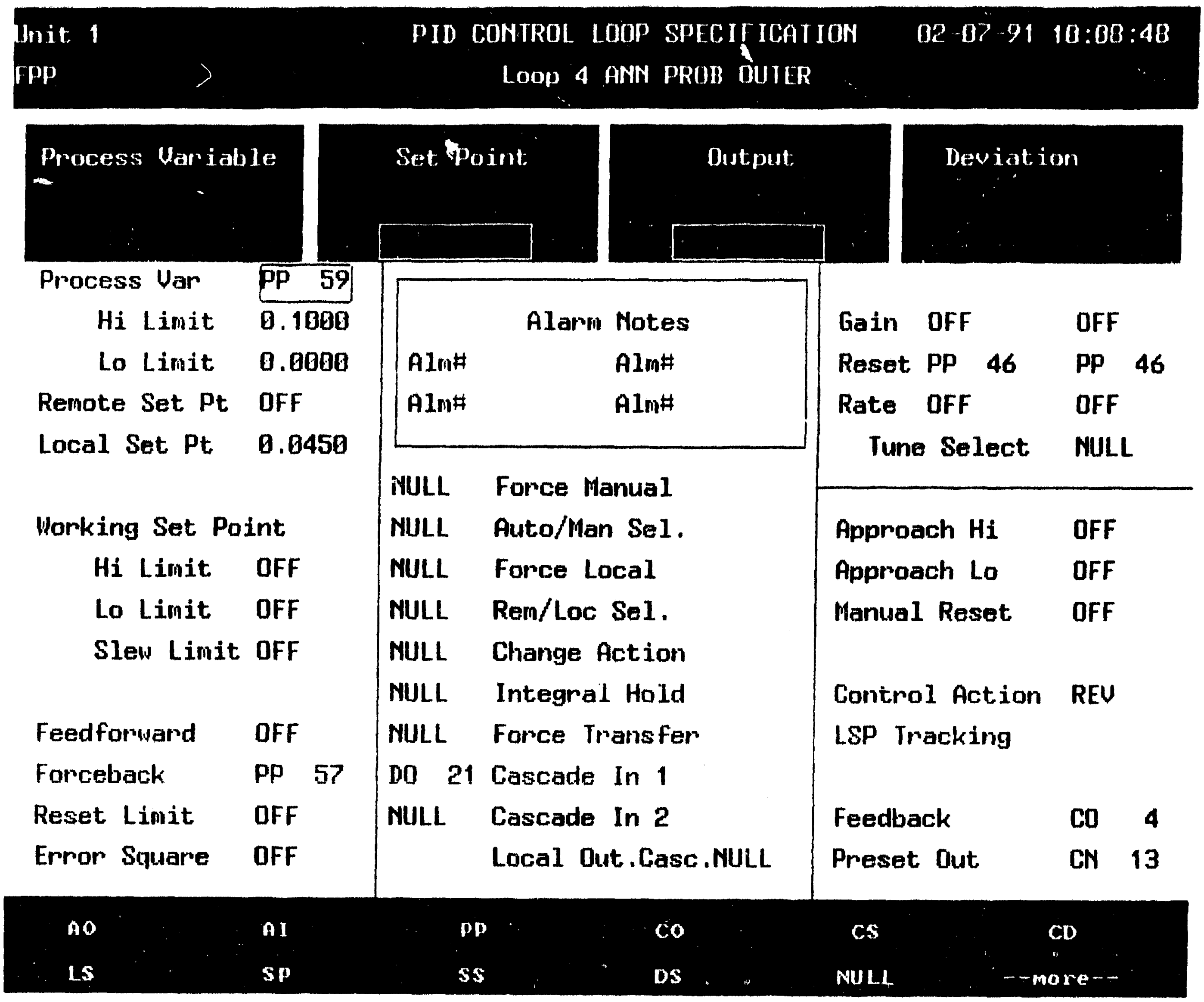
	Tag	
	--	



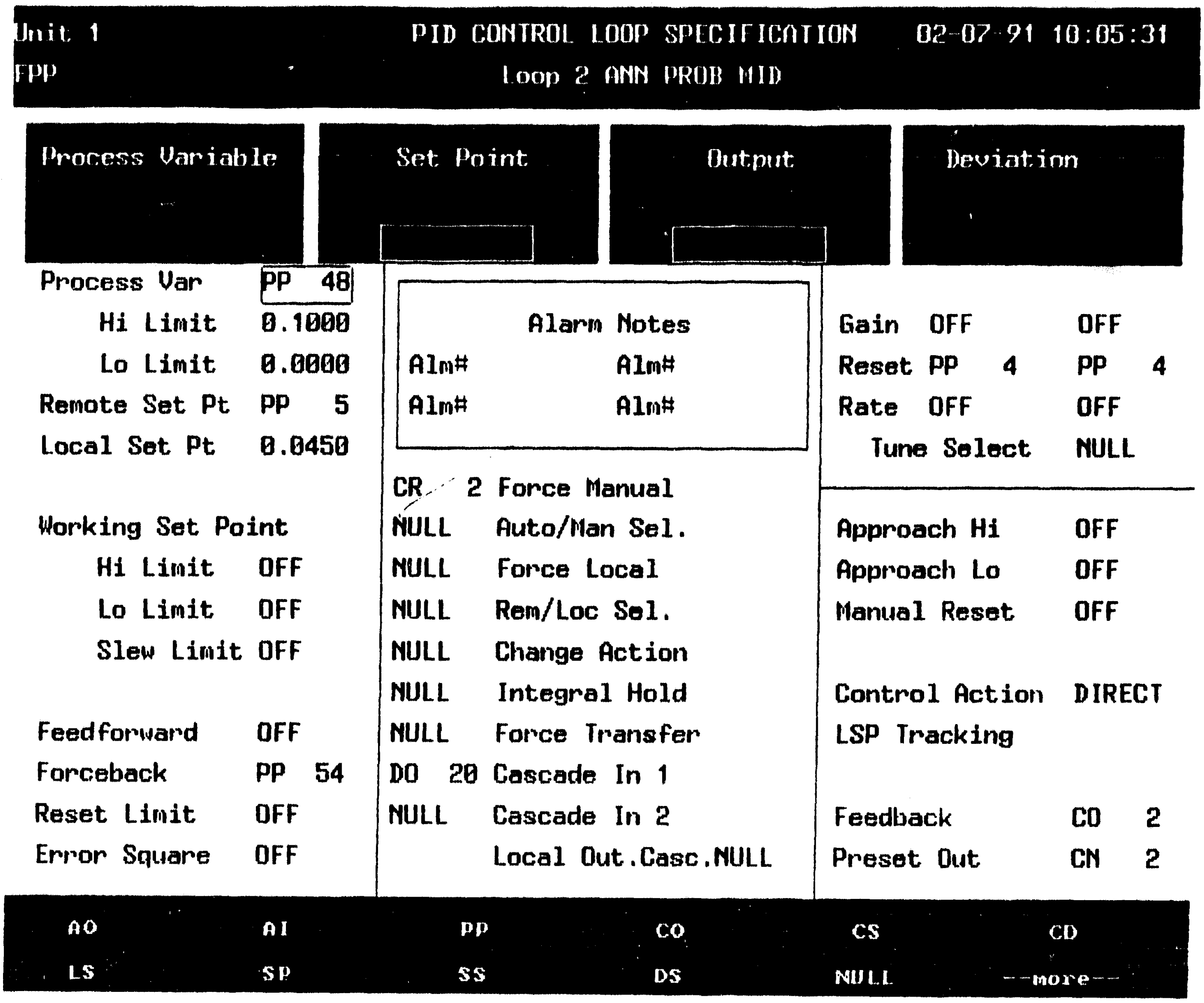
```
Unit 1                     PID CONTROL LOOP SPECIFICATION     11 09 90 15:27:20
FPP                              Loop 5 ZRDP FED FLOW FP

  Process Variable        Set Point          Output           Deviation

 Process Var   AI   61 |                            |
    Hi Limit   3.0000  |      Alarm Notes           | Gain   OFF        OFF
    Lo Limit   0.0000  | Alm#            Alm#       | Reset  10.000     OFF
 Remote Set Pt OFF     | Alm#            Alm#       | Rate   OFF        OFF
 Local Set Pt  0.0000  |                            |   Tune Select     NULL
                       | NULL   Force Manual        |
 Working Set Point     | NULL   Auto/Man Sel.       | Approach Hi       OFF
    Hi Limit   OFF     | NULL   Force Local         | Approach Lo       OFF
    Lo Limit   OFF     | NULL   Rem/Loc Sel.        | Manual Reset      OFF
    Slew Limit OFF     | NULL   Change Action       |
                       | NULL   Integral Hold       | Control Action    REV
 Feedforward   OFF     | NULL   Force Transfer      | LSP Tracking
 Forceback     OFF     | NULL   Cascade In 1        |
 Reset Limit   OFF     | NULL   Cascade In 2        | Feedback          AO   8
 Error Square  OFF     |        Local Out.Casc.NULL | Preset Out        CN  13

    AO        AI        PP        CO        CS        CD
    LS        SP        SS        DS        NULL      more
```



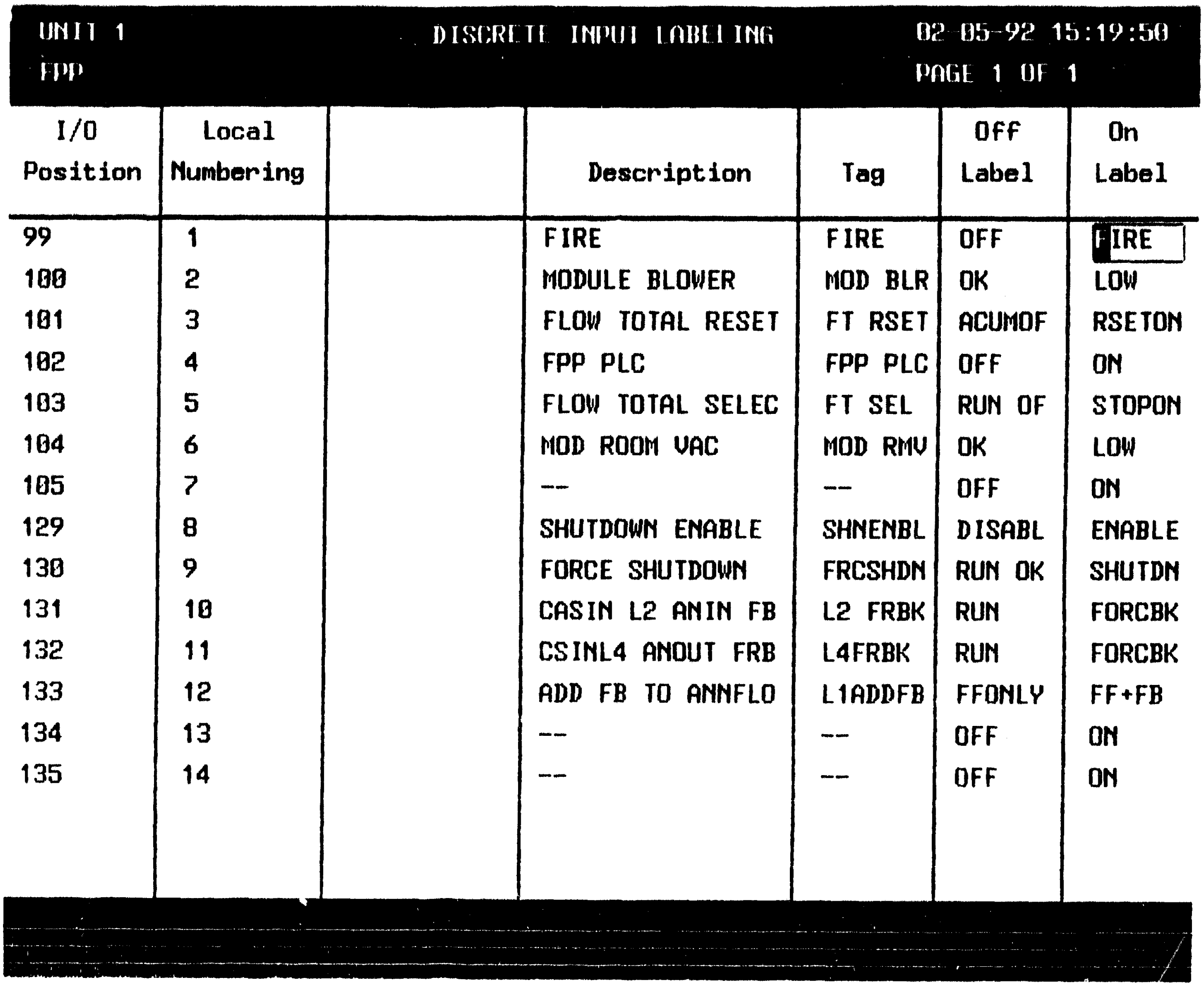
```
Unit 1                     PID CONTROL LOOP SPECIFICATION     02/07/91 10:10:21
FPP                              Loop 6 FINAL ANN OUTER

 Process Variable       Set Point            Output              Deviation

Process Var   PP  31 |                            |
    Hi Limit  0.1000 |        Alarm Notes         | Gain  OFF         OFF
    Lo Limit  0.0000 | Alm#           Alm#        | Reset PP   7      PP   7
Remote Set Pt OFF    | Alm#           Alm#        | Rate  OFF         OFF
Local Set Pt  0.0450 |                            |   Tune Select     NULL
                     | CR   2 Force Manual        |-------------------------
Working Set Point    | NULL   Auto/Man Sel.       | Approach Hi       OFF
    Hi Limit  OFF    | NULL   Force Local         | Approach Lo       OFF
    Lo Limit  OFF    | NULL   Rem/Loc Sel.        | Manual Reset      OFF
    Slew Limit OFF   | NULL   Change Action       |
                     | NULL   Integral Hold       | Control Action    DIRECT
Feedforward   OFF    | NULL   Force Transfer      | LSP Tracking
Forceback     PP   6 | DO  22 Cascade In 1        |
Reset Limit   OFF    | NULL   Cascade In 2        | Feedback          CO   6
Error Square  OFF    |        Local Out.Casc.NULL | Preset Out        CN  13

    AO          AI          PP          CO          CS          CD
    LS          SP          SS          DS          NULL        more
```



```
Unit 1                    PID CONTROL LOOP SPECIFICATION     02-07-91 10:08:48
FPP          >                   Loop 4 ANN PROB OUTER

Process Variable       Set Point            Output             Deviation

Process Var    PP  59                                    
    Hi Limit   0.1000            Alarm Notes             Gain   OFF       OFF
    Lo Limit   0.0000    Alm#              Alm#          Reset  PP  46    PP  46
Remote Set Pt  OFF       Alm#              Alm#          Rate   OFF       OFF
Local Set Pt   0.0450                                      Tune Select    NULL
                         NULL    Force Manual
Working Set Point        NULL    Auto/Man Sel.           Approach Hi      OFF
    Hi Limit   OFF       NULL    Force Local             Approach Lo      OFF
    Lo Limit   OFF       NULL    Rem/Loc Sel.            Manual Reset     OFF
    Slew Limit OFF       NULL    Change Action
                         NULL    Integral Hold           Control Action   REV
Feedforward    OFF       NULL    Force Transfer          LSP Tracking
Forceback      PP  57    DO  21  Cascade In 1
Reset Limit    OFF       NULL    Cascade In 2            Feedback         CO   4
Error Square   OFF               Local Out.Casc.NULL     Preset Out       CN  13

   AO          AI          PP          CO          CS          CD
   LS          SP          SS          DS          NULL        --more--
```

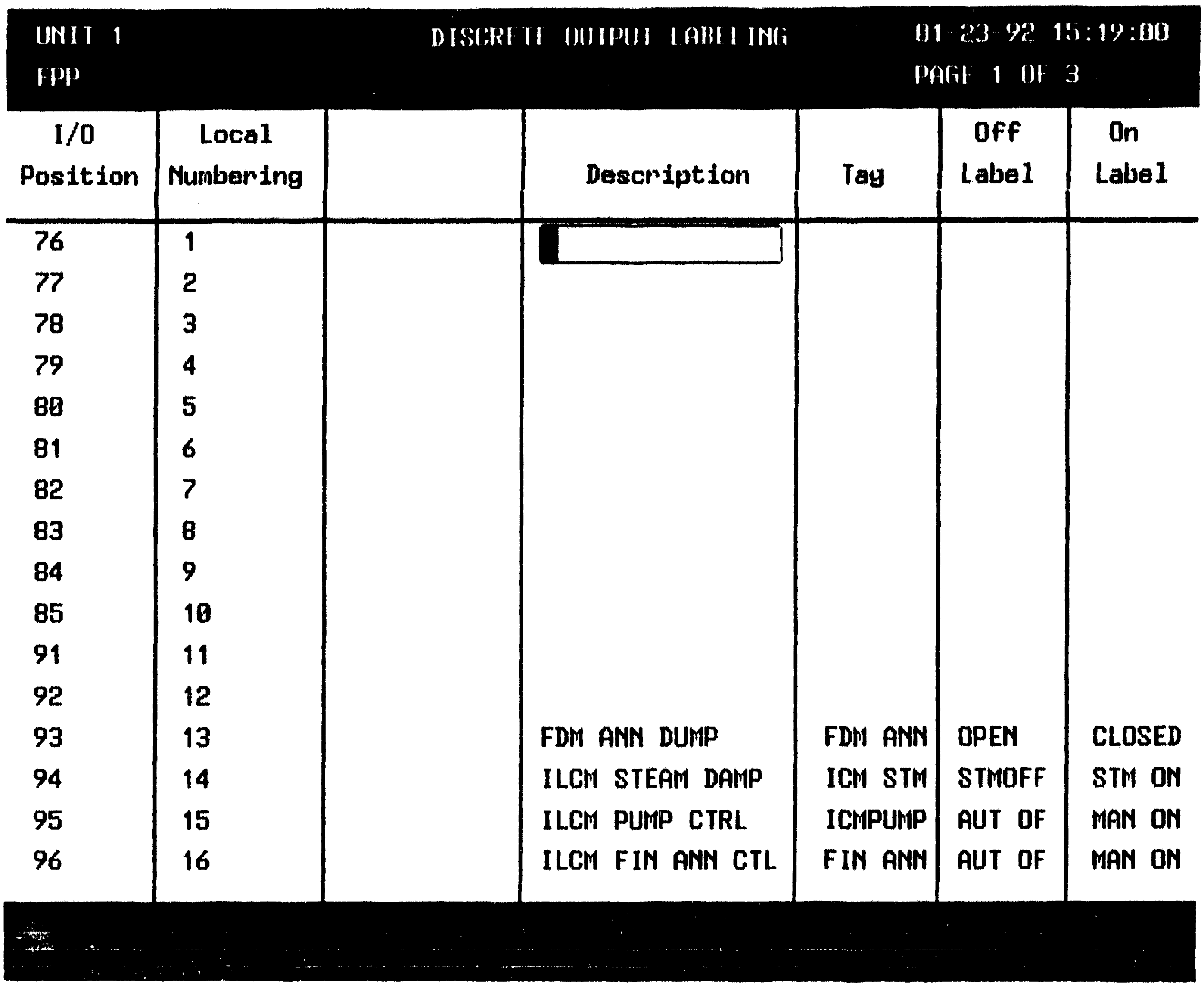


```
Unit 1                      PID CONTROL LOOP SPECIFICATION     02-07-91 10:05:31
FPP                               Loop 2 ANN PROB MID

 Process Variable       Set Point            Output             Deviation

 Process Var    PP  48
     Hi Limit   0.1000  |          Alarm Notes          |  Gain  OFF        OFF
     Lo Limit   0.0000  | Alm#               Alm#       |  Reset PP    4    PP   4
 Remote Set Pt  PP   5  | Alm#               Alm#       |  Rate  OFF        OFF
 Local Set Pt   0.0450  |                               |    Tune Select    NULL
                        CR    2 Force Manual
 Working Set Point      NULL    Auto/Man Sel.             Approach Hi      OFF
     Hi Limit   OFF     NULL    Force Local               Approach Lo      OFF
     Lo Limit   OFF     NULL    Rem/Loc Sel.              Manual Reset     OFF
     Slew Limit OFF     NULL    Change Action
                        NULL    Integral Hold             Control Action   DIRECT
 Feedforward    OFF     NULL    Force Transfer            LSP Tracking
 Forceback      PP  54  DO   20 Cascade In 1
 Reset Limit    OFF     NULL    Cascade In 2              Feedback         CO    2
 Error Square   OFF             Local Out.Casc.NULL       Preset Out       CN    2

    AO        AI        PP        CO        CS        CD
    LS        SP        SS        DS        NULL      --more--
```



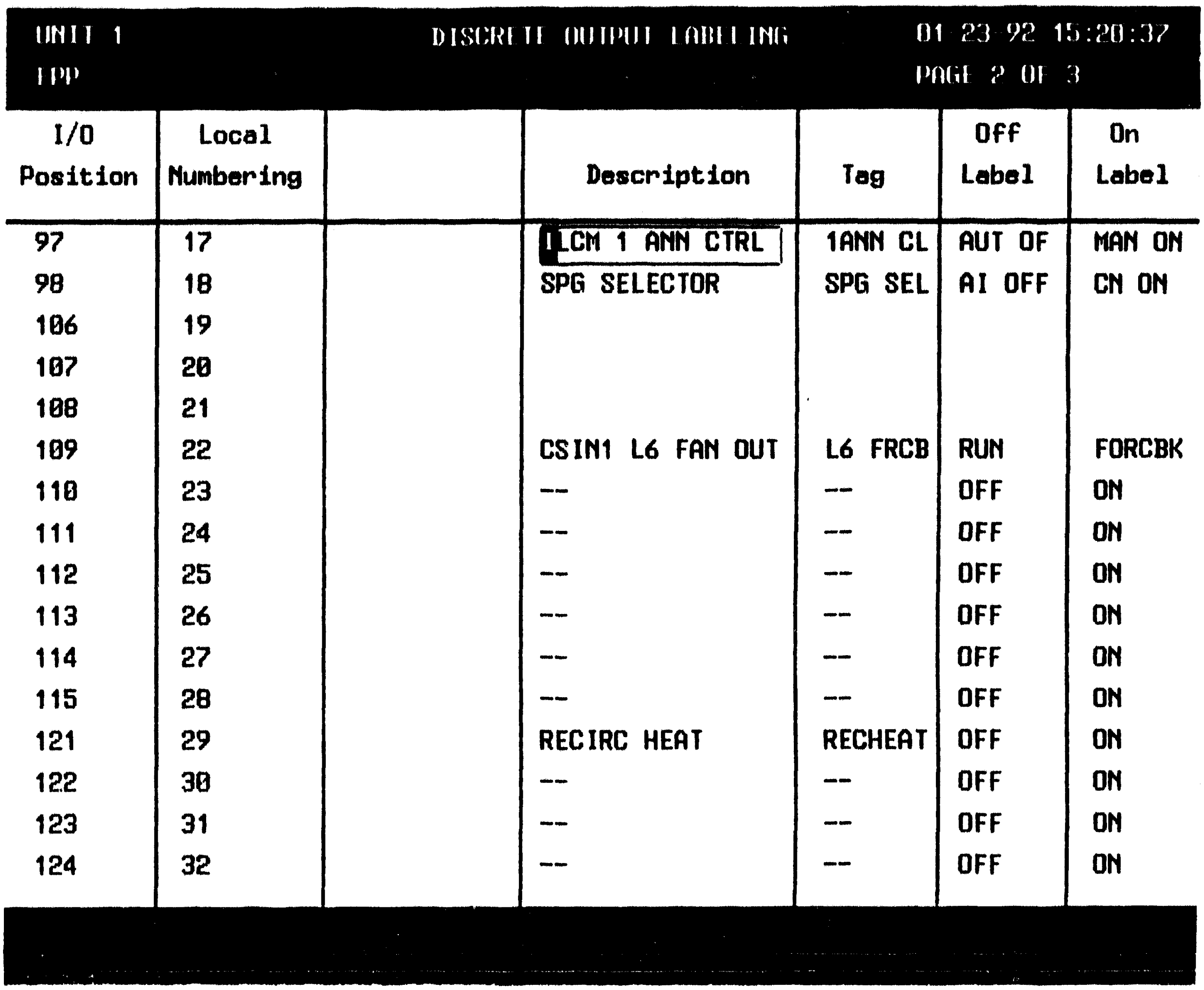
UNIT 1 DISCRETE INPUT LABELING 02-05-92 15:19:50
FPP PAGE 1 OF 1

I/O Position	Local Numbering		Description	Tag	Off Label	On Label
99	1		FIRE	FIRE	OFF	FIRE
100	2		MODULE BLOWER	MOD BLR	OK	LOW
101	3		FLOW TOTAL RESET	FT RSET	ACUMOF	RSETON
102	4		FPP PLC	FPP PLC	OFF	ON
103	5		FLOW TOTAL SELEC	FT SEL	RUN OF	STOPON
104	6		MOD ROOM VAC	MOD RMV	OK	LOW
105	7		--	--	OFF	ON
129	8		SHUTDOWN ENABLE	SHNENBL	DISABL	ENABLE
130	9		FORCE SHUTDOWN	FRCSHDN	RUN OK	SHUTDN
131	10		CASIN L2 ANIN FB	L2 FRBK	RUN	FORCBK
132	11		CSINL4 ANOUT FRB	L4FRBK	RUN	FORCBK
133	12		ADD FB TO ANNFLO	L1ADDFB	FFONLY	FF+FB
134	13		--	--	OFF	ON
135	14		--	--	OFF	ON



UNIT 1 DISCRETE OUTPUT LABELING 01-23-92 15:19:00
FPP PAGE 1 OF 3

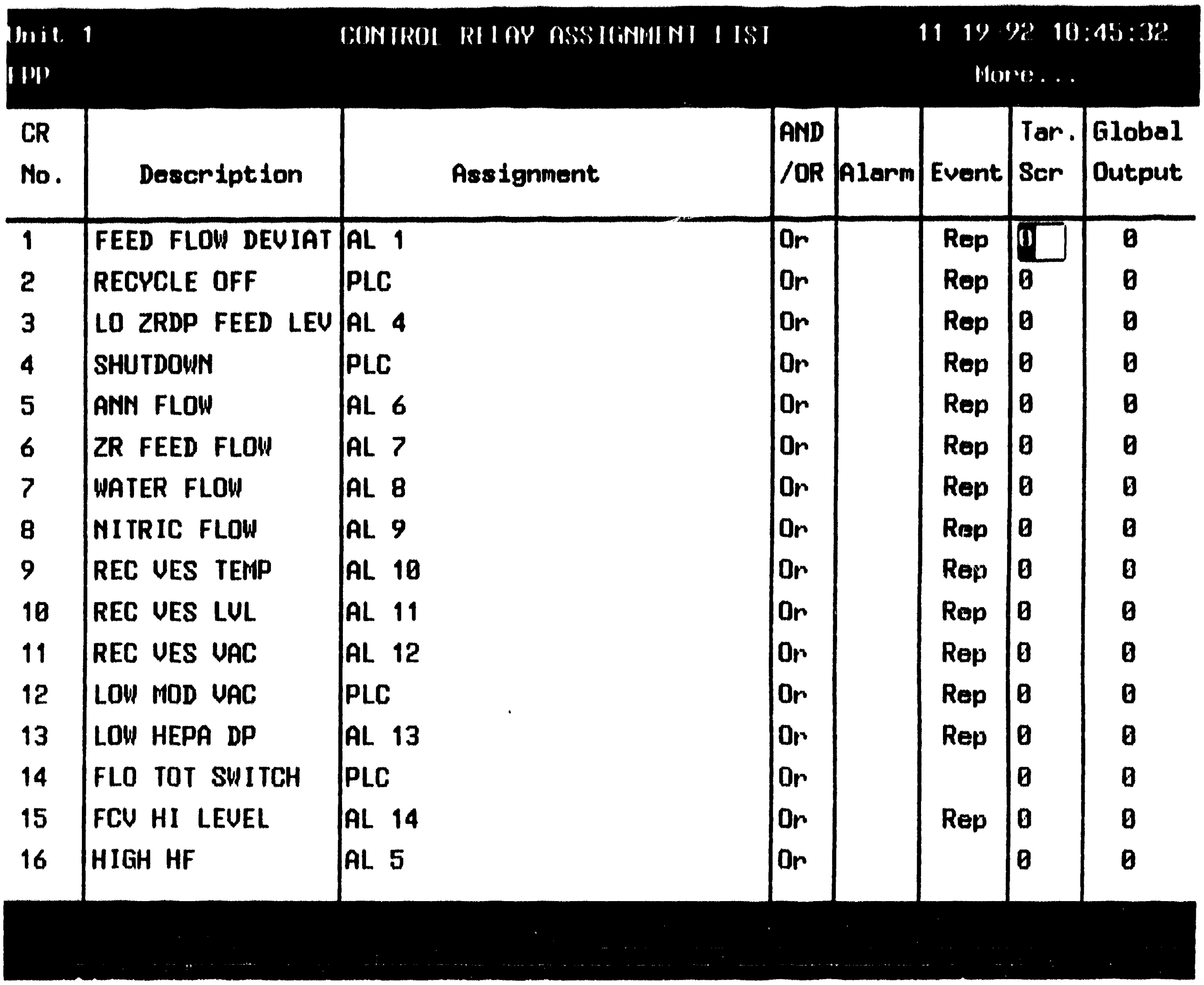
I/O Position	Local Numbering		Description	Tag	Off Label	On Label
76	1					
77	2					
78	3					
79	4					
80	5					
81	6					
82	7					
83	8					
84	9					
85	10					
91	11					
92	12					
93	13		FDM ANN DUMP	FDM ANN	OPEN	CLOSED
94	14		ILCM STEAM DAMP	ICM STM	STMOFF	STM ON
95	15		ILCM PUMP CTRL	ICMPUMP	AUT OF	MAN ON
96	16		ILCM FIN ANN CTL	FIN ANN	AUT OF	MAN ON



UNIT 1 DISCRETE OUTPUT LABELING 01-23-92 15:20:37

FPP PAGE 2 OF 3

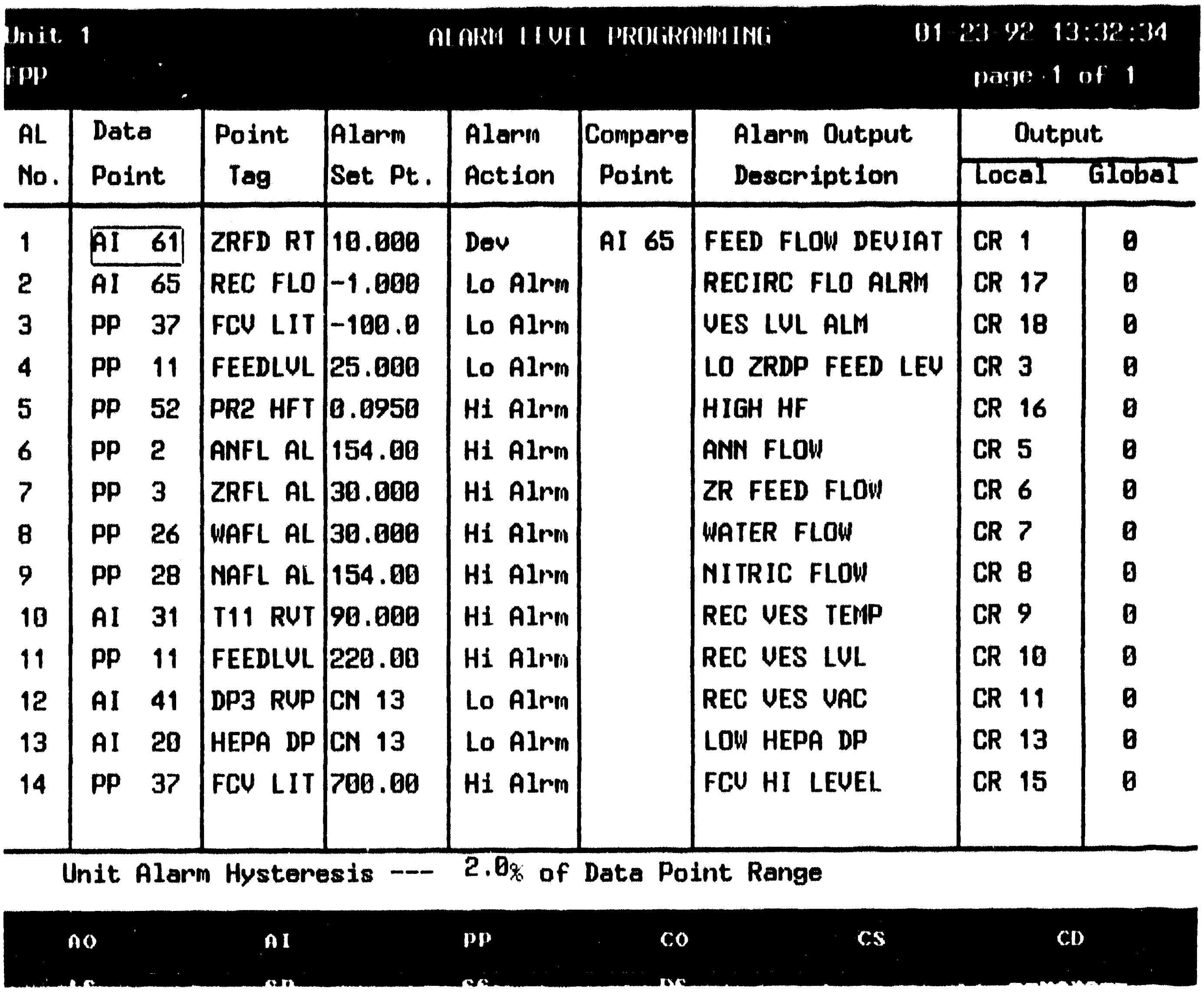
I/O Position	Local Numbering		Description	Tag	Off Label	On Label
97	17		ILCM 1 ANN CTRL	1ANN CL	AUT OF	MAN ON
98	18		SPG SELECTOR	SPG SEL	AI OFF	CN ON
106	19					
107	20					
108	21					
109	22		CSIN1 L6 FAN OUT	L6 FRCB	RUN	FORCBK
110	23		--	--	OFF	ON
111	24		--	--	OFF	ON
112	25		--	--	OFF	ON
113	26		--	--	OFF	ON
114	27		--	--	OFF	ON
115	28		--	--	OFF	ON
121	29		RECIRC HEAT	RECHEAT	OFF	ON
122	30		--	--	OFF	ON
123	31		--	--	OFF	ON
124	32		--	--	OFF	ON



Unit 1 CONTROL RELAY ASSIGNMENT LIST 11 19 92 10:45:32

IPP More...

CR No.	Description	Assignment	AND /OR	Alarm	Event	Tar. Scr	Global Output
1	FEED FLOW DEVIAT	AL 1	Or		Rep	0	0
2	RECYCLE OFF	PLC	Or		Rep	0	0
3	LO ZRDP FEED LEV	AL 4	Or		Rep	0	0
4	SHUTDOWN	PLC	Or		Rep	0	0
5	ANN FLOW	AL 6	Or		Rep	0	0
6	ZR FEED FLOW	AL 7	Or		Rep	0	0
7	WATER FLOW	AL 8	Or		Rep	0	0
8	NITRIC FLOW	AL 9	Or		Rep	0	0
9	REC VES TEMP	AL 10	Or		Rep	0	0
10	REC VES LVL	AL 11	Or		Rep	0	0
11	REC VES VAC	AL 12	Or		Rep	0	0
12	LOW MOD VAC	PLC	Or		Rep	0	0
13	LOW HEPA DP	AL 13	Or		Rep	0	0
14	FLO TOT SWITCH	PLC	Or			0	0
15	FCV HI LEVEL	AL 14	Or		Rep	0	0
16	HIGH HF	AL 5	Or			0	0

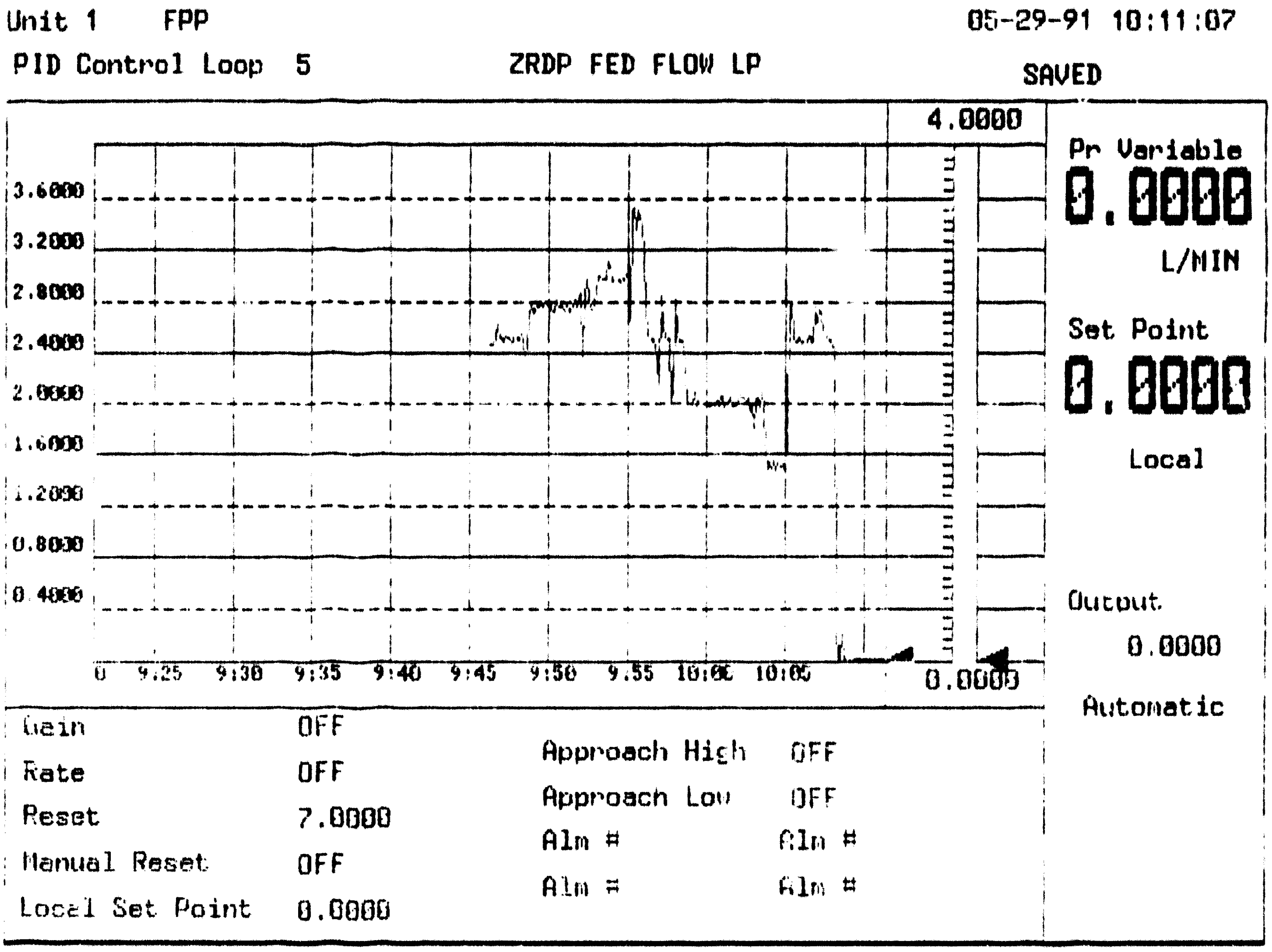


Unit 1 ALARM LEVEL PROGRAMMING 01 23 92 13:32:34

FPP page 1 of 1

AL No.	Data Point	Point Tag	Alarm Set Pt.	Alarm Action	Compare Point	Alarm Output Description	Output	
							Local	Global
1	AI 61	ZRFD RT	10.000	Dev	AI 65	FEED FLOW DEVIAT	CR 1	0
2	AI 65	REC FLO	-1.000	Lo Alrm		RECIRC FLO ALRM	CR 17	0
3	PP 37	FCV LIT	-100.0	Lo Alrm		VES LVL ALM	CR 18	0
4	PP 11	FEEDLVL	25.000	Lo Alrm		LO ZRDP FEED LEV	CR 3	0
5	PP 52	PR2 HFT	0.0950	Hi Alrm		HIGH HF	CR 16	0
6	PP 2	ANFL AL	154.00	Hi Alrm		ANN FLOW	CR 5	0
7	PP 3	ZRFL AL	30.000	Hi Alrm		ZR FEED FLOW	CR 6	0
8	PP 26	WAFL AL	30.000	Hi Alrm		WATER FLOW	CR 7	0
9	PP 28	NAFL AL	154.00	Hi Alrm		NITRIC FLOW	CR 8	0
10	AI 31	T11 RVT	90.000	Hi Alrm		REC VES TEMP	CR 9	0
11	PP 11	FEEDLVL	220.00	Hi Alrm		REC VES LVL	CR 10	0
12	AI 41	DP3 RVP	CN 13	Lo Alrm		REC VES VAC	CR 11	0
13	AI 20	HEPA DP	CN 13	Lo Alrm		LOW HEPA DP	CR 13	0
14	PP 37	FCV LIT	700.00	Hi Alrm		FCV HI LEVEL	CR 15	0

Unit Alarm Hysteresis --- 2.0% of Data Point Range



Unit 1 FPP

05-29-91 10:11:07

PID Control Loop 5

ZRDP FED FLOW LP

SAVED

Pr Variable 0.0000 L/MIN

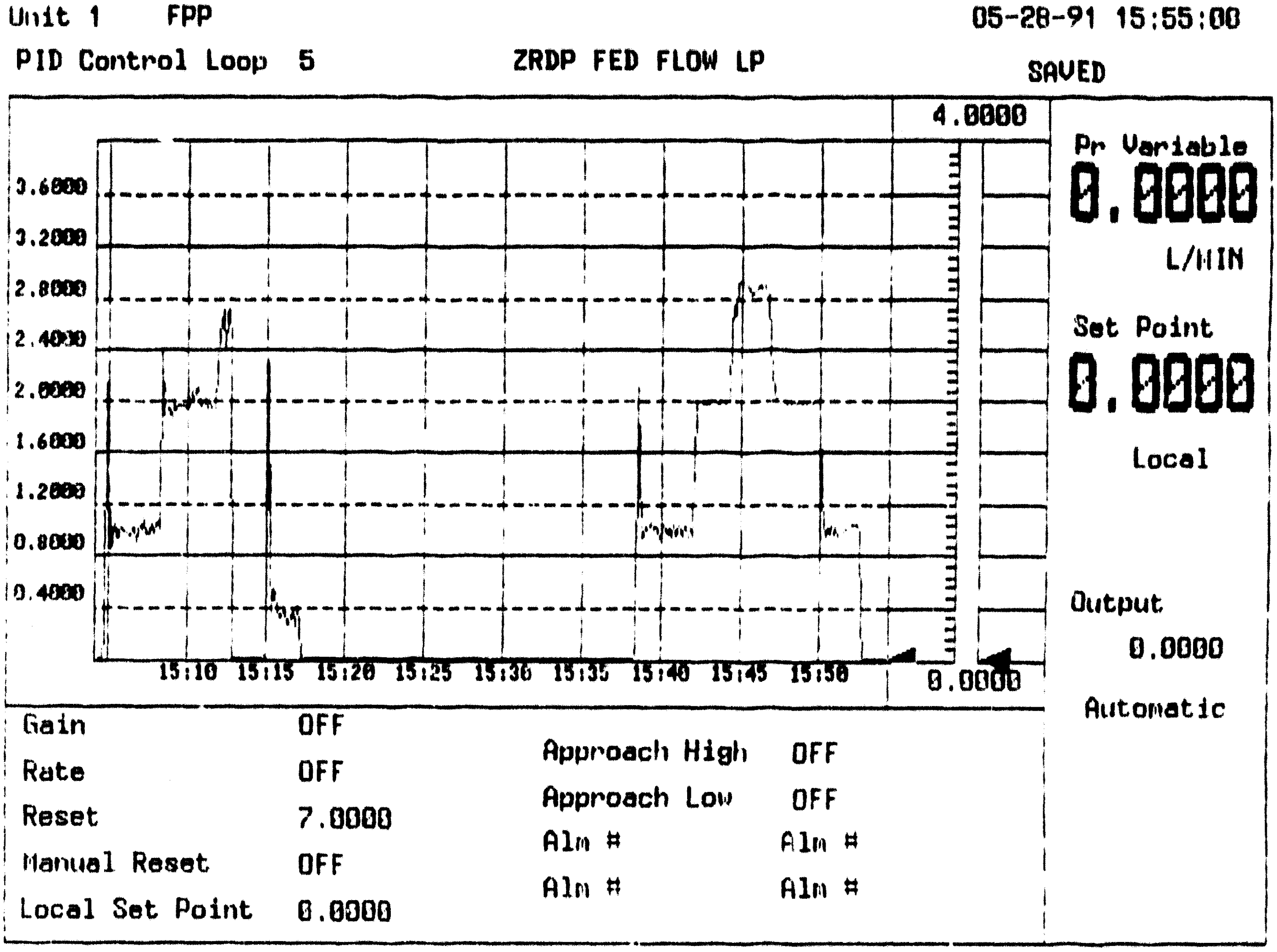
Set Point 0.0000 Local

Output 0.0000

Automatic

Gain	OFF
Rate	OFF
Reset	7.0000
Manual Reset	OFF
Local Set Point	0.0000

Approach High	OFF		
Approach Low	OFF		
Alm #		Alm #	
Alm #		Alm #	



Unit 1 FPP 05-28-91 15:55:00

PID Control Loop 5 ZRDP FED FLOW LP SAVED

Pr Variable 0.0000 L/MIN

Set Point 0.0000 Local

Output 0.0000

Automatic

Gain OFF

Rate OFF

Reset 7.0000

Manual Reset OFF

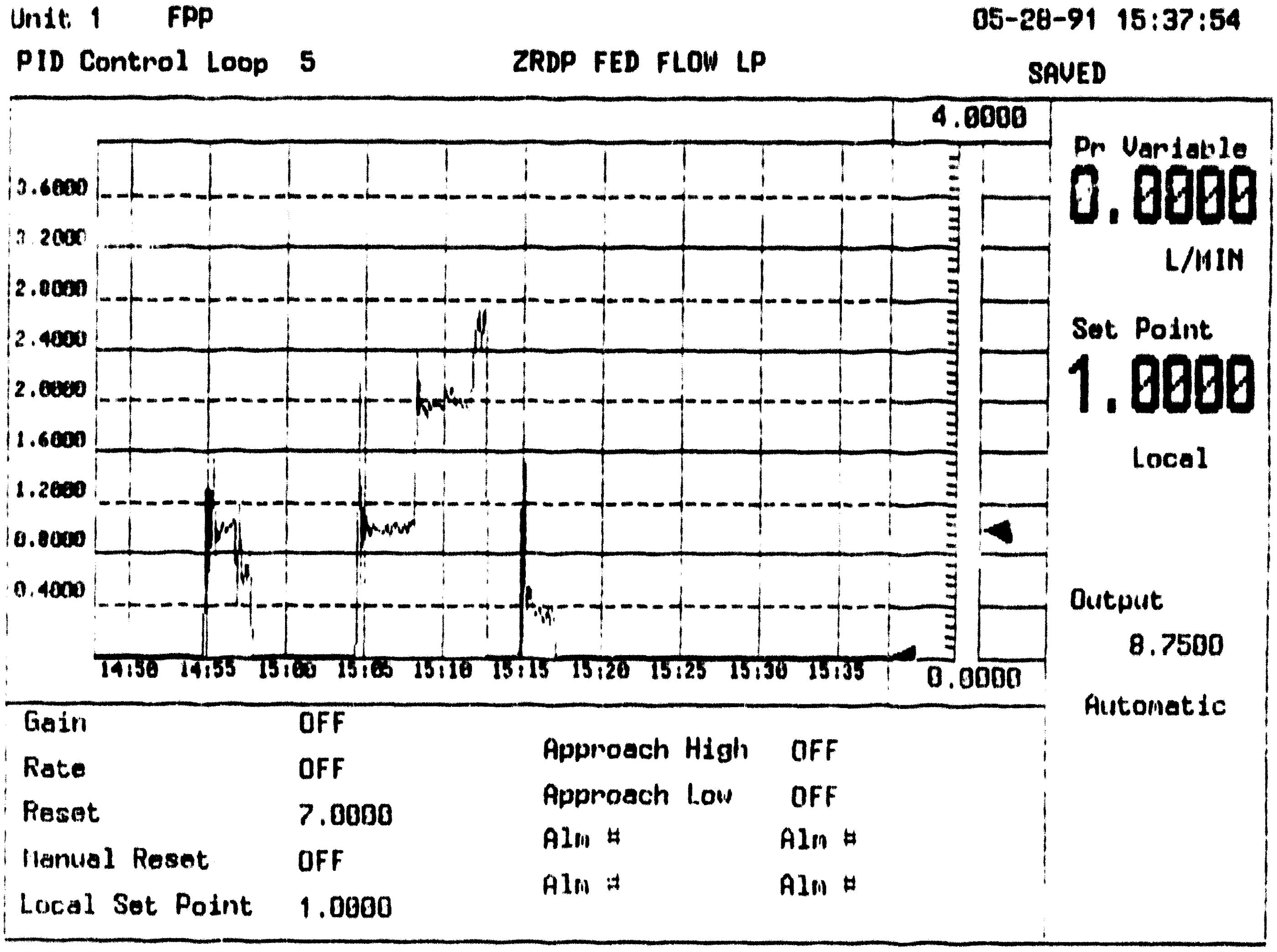
Local Set Point 0.0000

Approach High OFF

Approach Low OFF

Alm # Alm #

Alm # Alm #



Unit 1 FPP 05-28-91 15:37:54

PID Control Loop 5 ZRDP FED FLOW LP SAVED

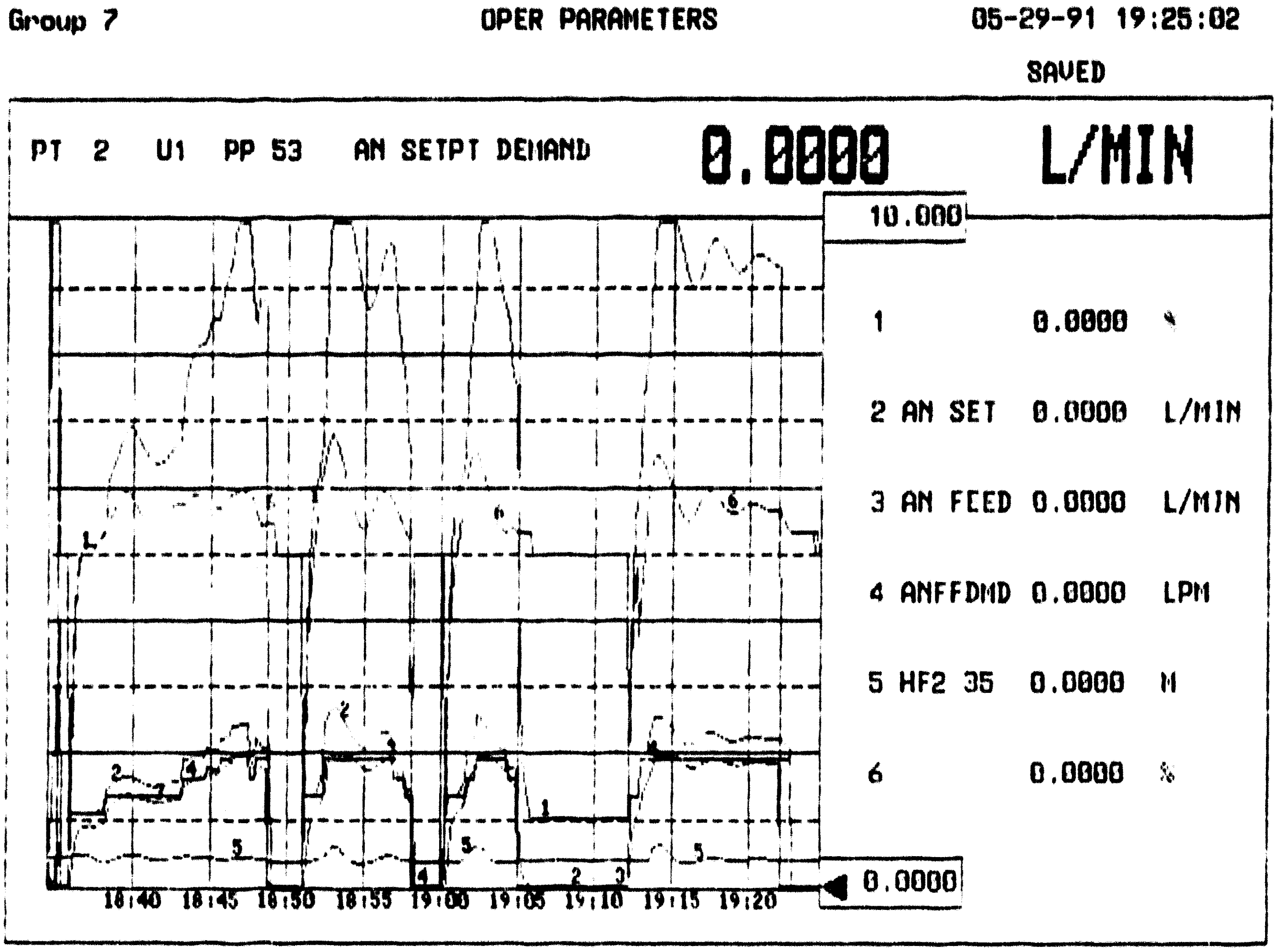
Pr Variable
0.0000
L/MIN

Set Point
1.0000
Local

Output
8.7500
Automatic

Gain	OFF
Rate	OFF
Reset	7.0000
Manual Reset	OFF
Local Set Point	1.0000

Approach High	OFF
Approach Low	OFF
Alm #	Alm #
Alm #	Alm #



Group 7 OPER PARAMETERS 05-29-91 19:25:02

SAVED

PT 2 U1 PP 53 AN SETPT DEMAND 0.0000 L/MIN

1 0.0000 %

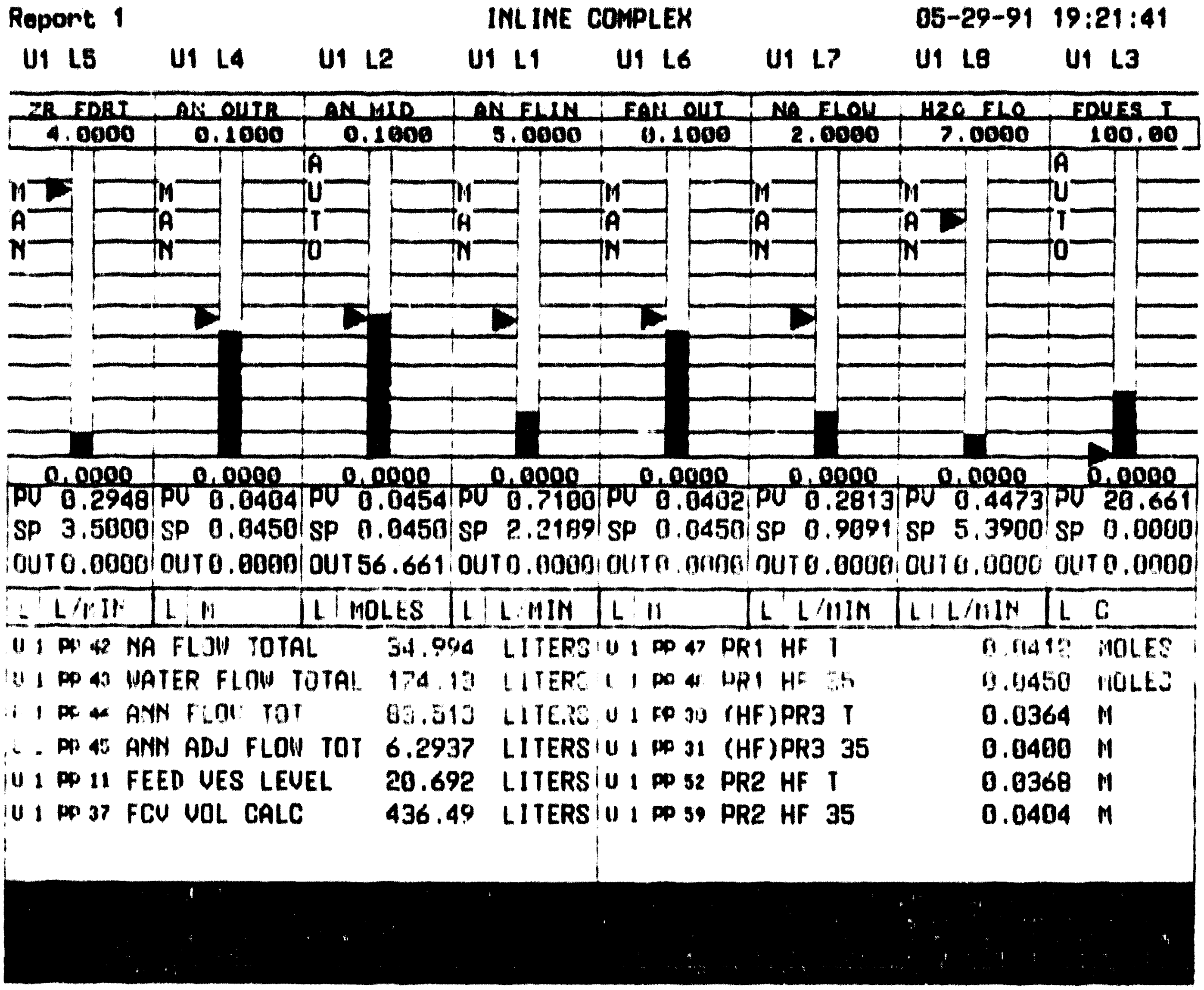
2 AN SET 0.0000 L/MIN

3 AN FEED 0.0000 L/MIN

4 ANFFDMD 0.0000 LPM

5 HF2 35 0.0000 M

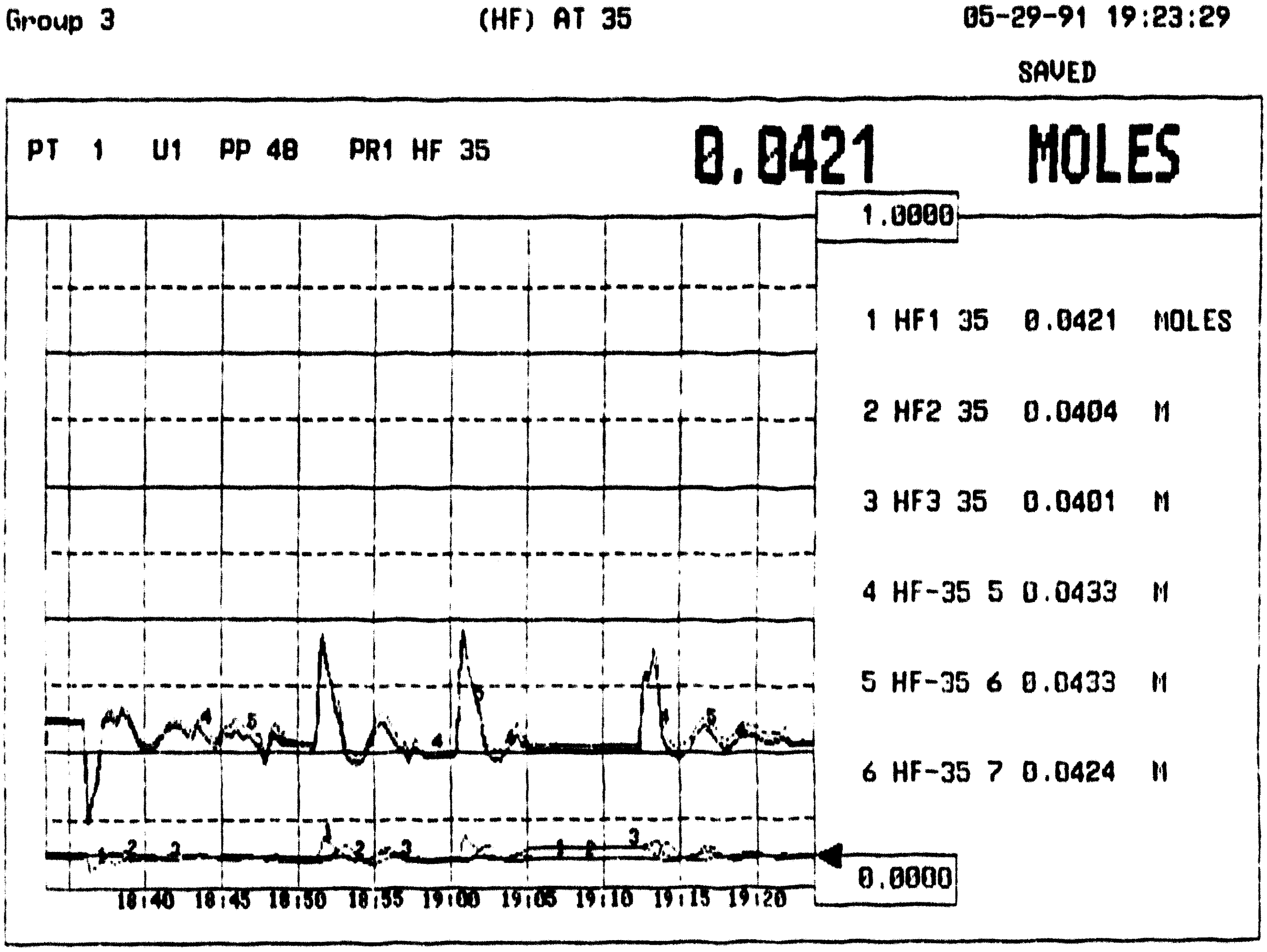
6 0.0000 %



Report 1 INLINE COMPLEX 05-29-91 19:21:41

U1 L5	U1 L4	U1 L2	U1 L1	U1 L6	U1 L7	U1 L8	U1 L3
ZR FDRT	AN OUTR	AN MID	AN FLIN	FAN OUT	NA FLOW	H2O FLO	FDVES T
4.0000	0.1000	0.1000	5.0000	0.1000	2.0000	7.0000	100.00
MAN	MAN	AUTO	MAN	MAN	MAN	MAN	AUTO
0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
PV 0.2948	PV 0.0404	PV 0.0454	PV 0.7100	PV 0.0402	PV 0.2813	PV 0.4473	PV 20.661
SP 3.5000	SP 0.0450	SP 0.0450	SP 2.2189	SP 0.0450	SP 0.9091	SP 5.3900	SP 0.0000
OUT 0.0000	OUT 0.0000	OUT 56.661	OUT 0.0000	OUT 0.0000	OUT 0.0000	OUT 0.0000	OUT 0.0000
L L/MIN	L M	L MOLES	L L/MIN	L M	L L/MIN	L L/MIN	L C

U 1 PP 42	NA FLOW TOTAL	34.994	LITERS	U 1 PP 47	PR1 HF T	0.0412	MOLES
U 1 PP 43	WATER FLOW TOTAL	174.13	LITERS	U 1 PP 46	PR1 HF 35	0.0450	MOLES
U 1 PP 44	ANN FLOW TOT	83.513	LITERS	U 1 PP 30	(HF)PR3 T	0.0364	M
U 1 PP 45	ANN ADJ FLOW TOT	6.2937	LITERS	U 1 PP 31	(HF)PR3 35	0.0400	M
U 1 PP 11	FEED VES LEVEL	20.692	LITERS	U 1 PP 52	PR2 HF T	0.0368	M
U 1 PP 37	FCV VOL CALC	436.49	LITERS	U 1 PP 59	PR2 HF 35	0.0404	M



Group 3 (HF) AT 35 05-29-91 19:23:29

SAVED

PT 1 U1 PP 48 PR1 HF 35 **0.0421 MOLES**

1 HF1 35 0.0421 MOLES

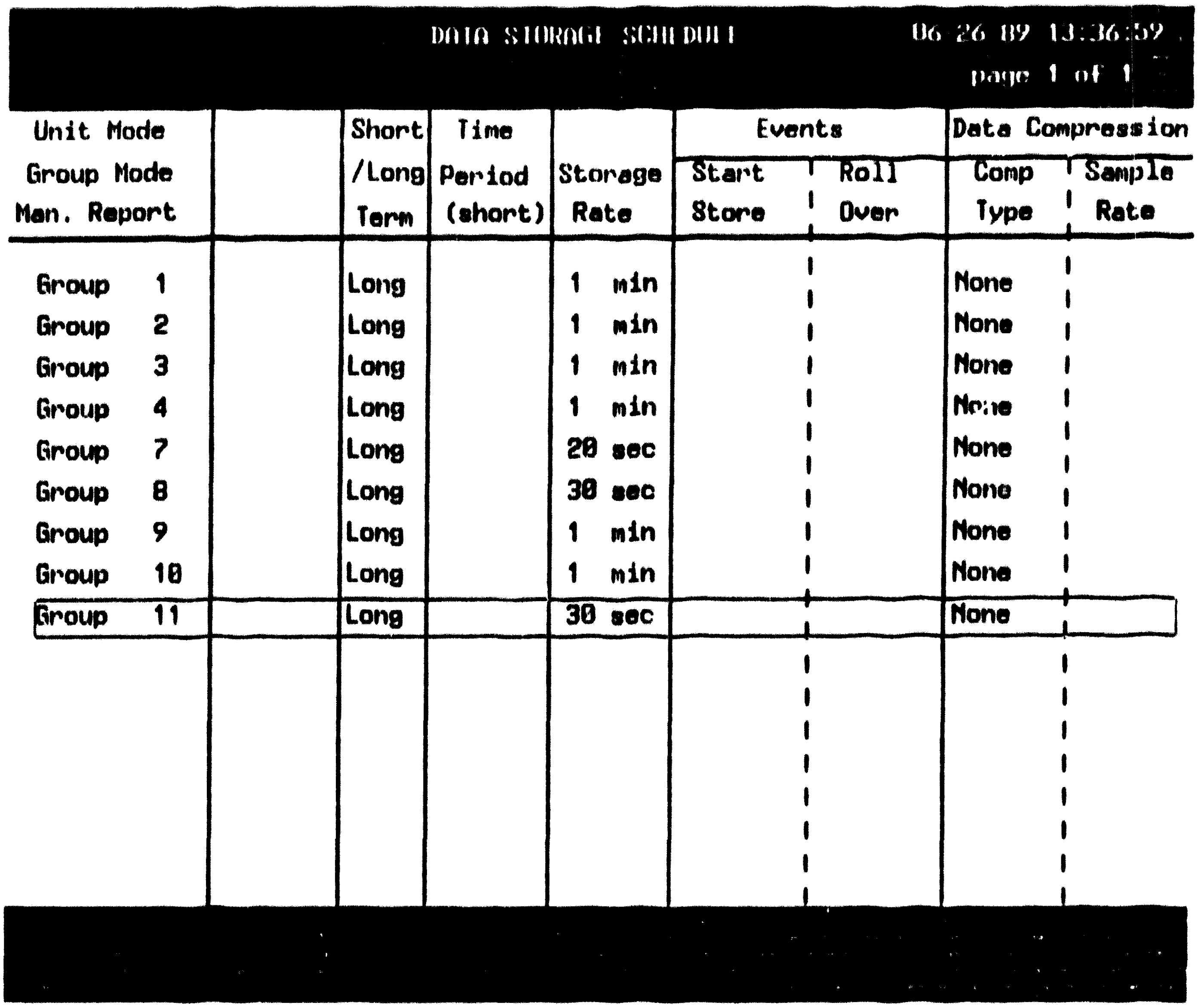
2 HF2 35 0.0404 M

3 HF3 35 0.0401 M

4 HF-35 5 0.0433 M

5 HF-35 6 0.0433 M

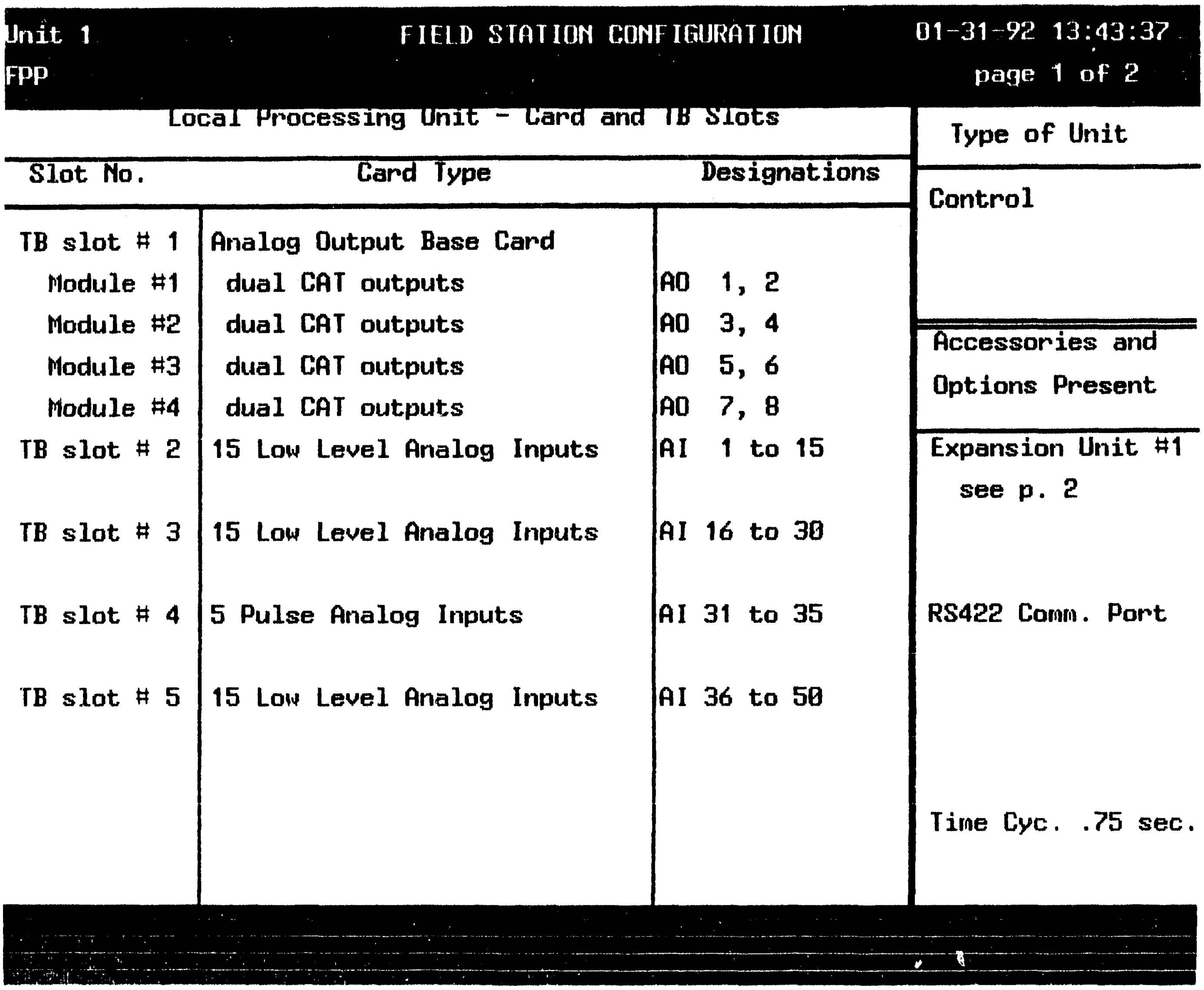
6 HF-35 7 0.0424 M



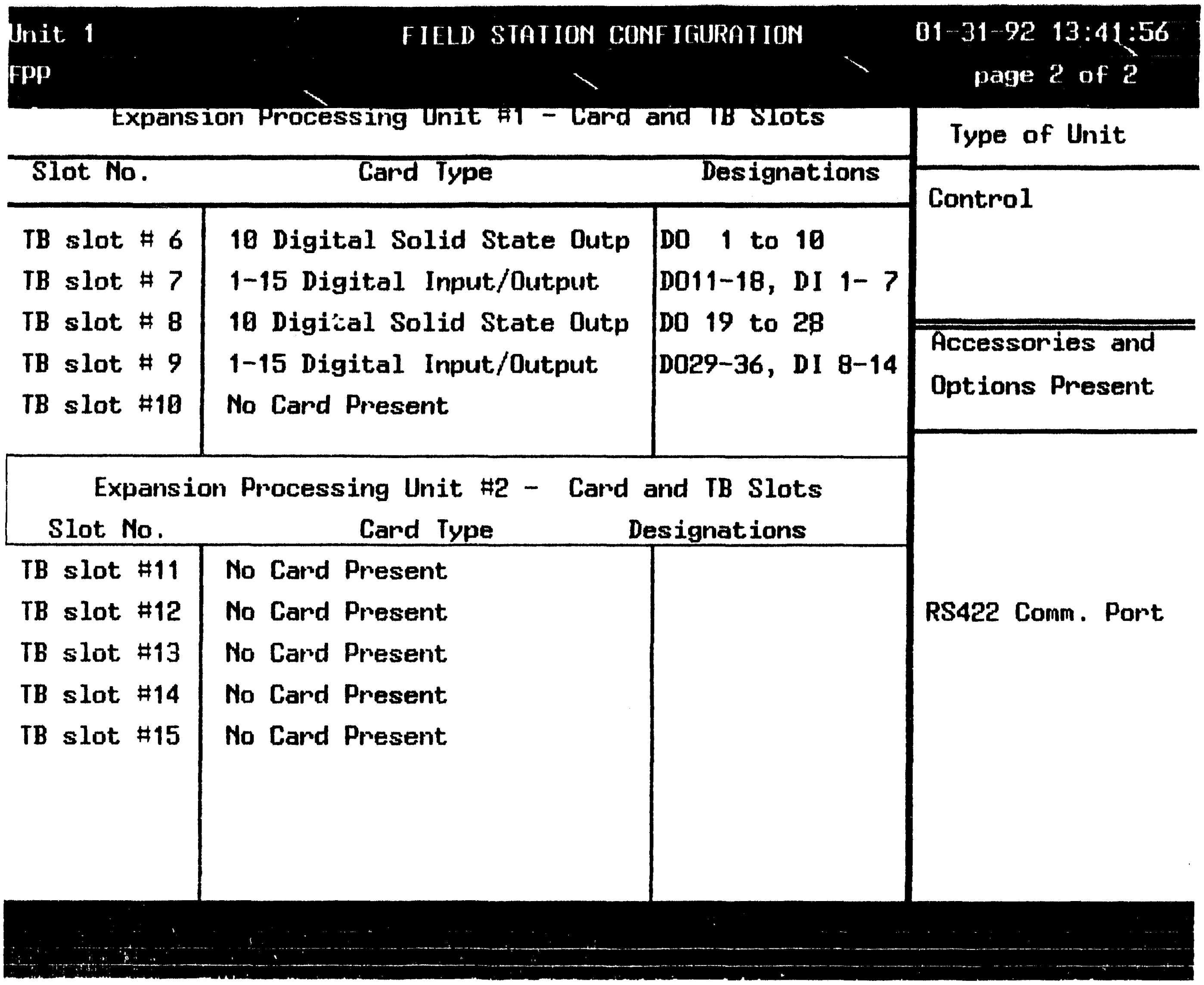
DATA STORAGE SCHEDULE 06 26 89 13:36:59

page 1 of 1

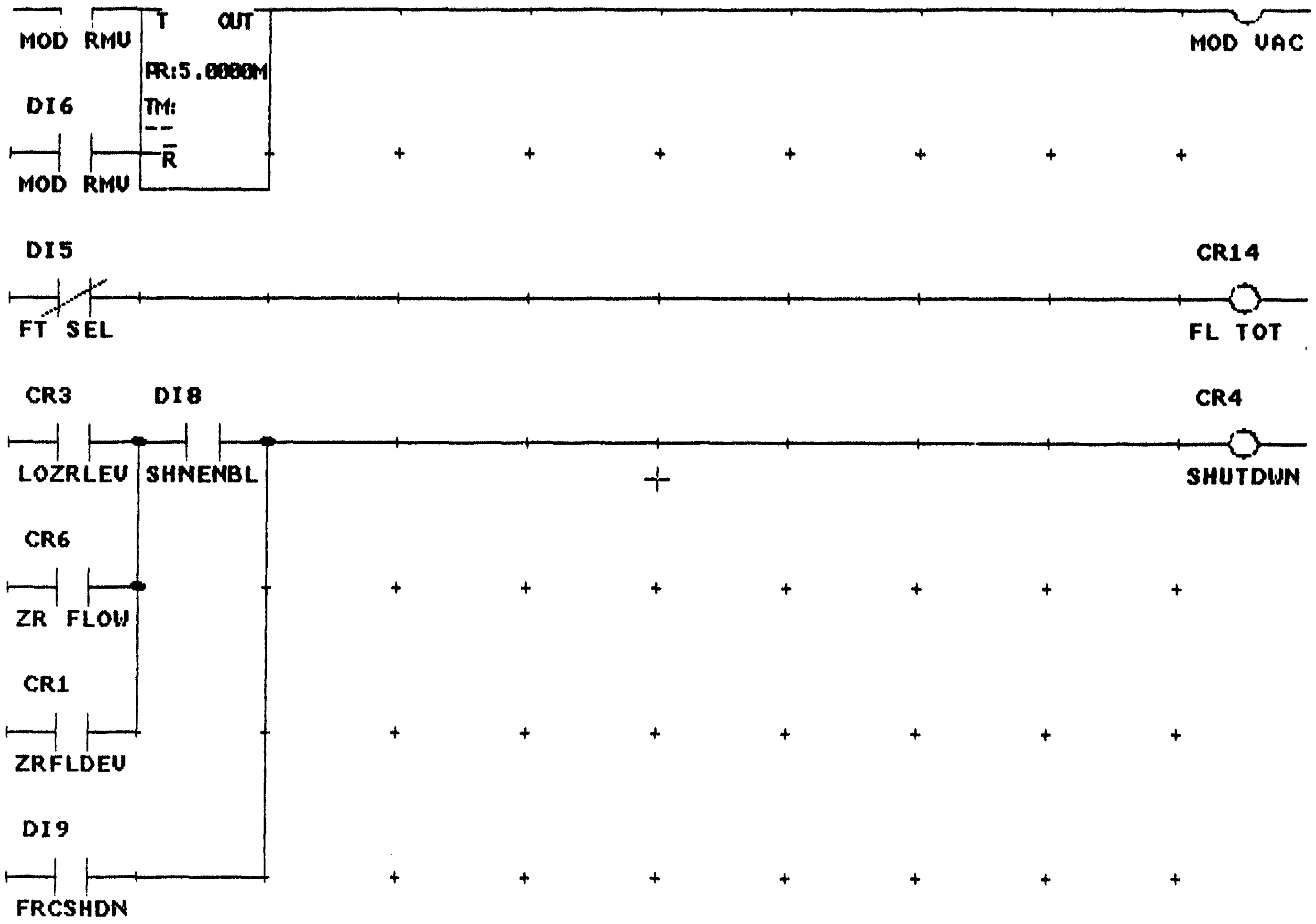
Unit Mode Group Mode Man. Report		Short /Long Term	Time Period (short)	Storage Rate	Events		Data Compression	
					Start Store	Roll Over	Comp Type	Sample Rate
Group 1		Long		1 min			None	
Group 2		Long		1 min			None	
Group 3		Long		1 min			None	
Group 4		Long		1 min			None	
Group 7		Long		20 sec			None	
Group 8		Long		30 sec			None	
Group 9		Long		1 min			None	
Group 10		Long		1 min			None	
Group 11		Long		30 sec			None	

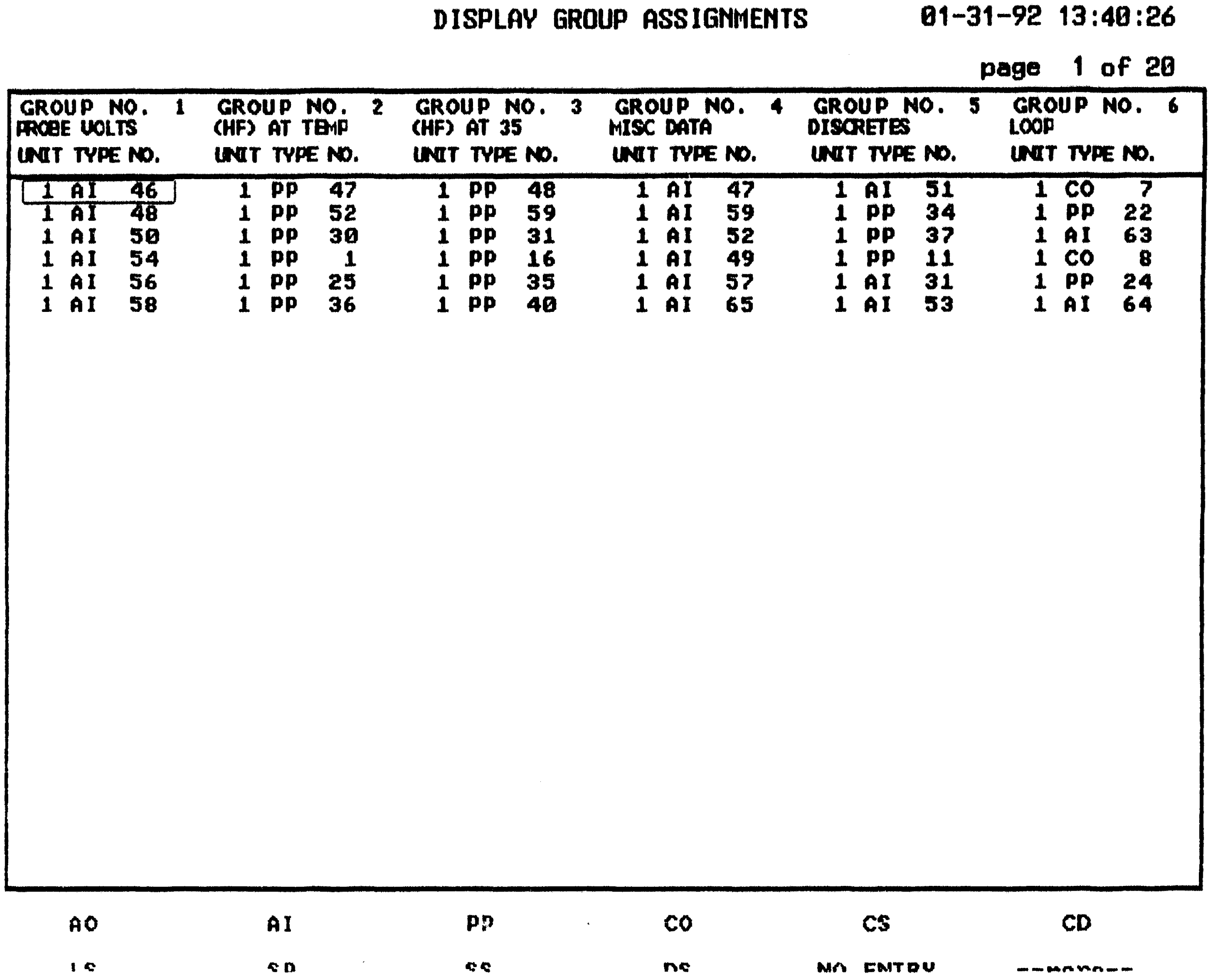


```
Unit 1                 FIELD STATION CONFIGURATION            01-31-92 13:43:37
FPP                                                                page 1 of 2
            Local Processing Unit - Card and TB Slots         |  Type of Unit
Slot No.              Card Type              Designations     |
                                                              |  Control
TB slot # 1 | Analog Output Base Card      |                  |
  Module #1 |  dual CAT outputs            |AO  1, 2          |
  Module #2 |  dual CAT outputs            |AO  3, 4          |
  Module #3 |  dual CAT outputs            |AO  5, 6          |  Accessories and
  Module #4 |  dual CAT outputs            |AO  7, 8          |  Options Present
TB slot # 2 | 15 Low Level Analog Inputs   |AI  1 to 15       |  Expansion Unit #1
            |                              |                  |    see p. 2
TB slot # 3 | 15 Low Level Analog Inputs   |AI 16 to 30       |
            |                              |                  |
TB slot # 4 | 5 Pulse Analog Inputs        |AI 31 to 35       |  RS422 Comm. Port
            |                              |                  |
TB slot # 5 | 15 Low Level Analog Inputs   |AI 36 to 50       |
            |                              |                  |
            |                              |                  |  Time Cyc. .75 sec.
```

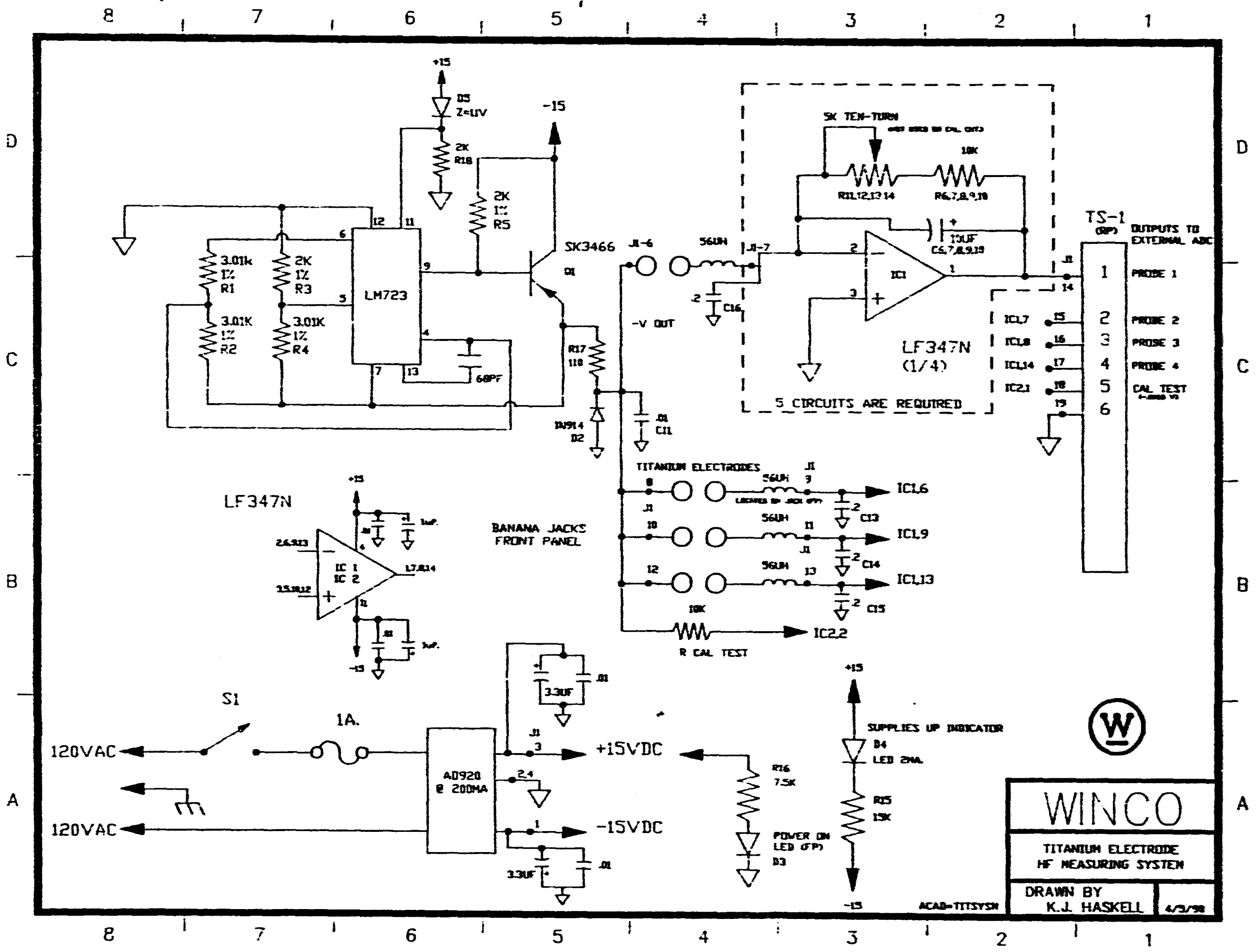


```
Unit 1                      FIELD STATION CONFIGURATION             01-31-92 13:41:56
FPP                                                                   page 2 of 2
       Expansion Processing Unit #1 - Card and TB Slots          Type of Unit
 Slot No.                 Card Type                Designations
                                                                  Control
 TB slot # 6   10 Digital Solid State Outp  DO  1 to 10
 TB slot # 7   1-15 Digital Input/Output    DO11-18, DI 1- 7
 TB slot # 8   10 Digital Solid State Outp  DO 19 to 28
 TB slot # 9   1-15 Digital Input/Output    DO29-36, DI 8-14     Accessories and
 TB slot #10   No Card Present                                    Options Present

     Expansion Processing Unit #2 -  Card and TB Slots
  Slot No.                Card Type           Designations
 TB slot #11   No Card Present
 TB slot #12   No Card Present                                    RS422 Comm. Port
 TB slot #13   No Card Present
 TB slot #14   No Card Present
 TB slot #15   No Card Present
```


MOD RMV
T OUT
PR:5.0000M
TM:
DI6
R
MOD RMV
MOD VAC
DI5
FT SEL
CR14
FL TOT
CR3
LOZRLEV
DI8
SHNENBL
CR4
SHUTDWN
CR6
ZR FLOW
CR1
ZRFLDEV
DI9
FRCSHDN



```
                         DISPLAY GROUP ASSIGNMENTS       01-31-92 13:40:26

                                                              page  1 of 20
GROUP NO.  1  GROUP NO.  2  GROUP NO.  3  GROUP NO.  4  GROUP NO.  5  GROUP NO.  6
PROBE VOLTS   (HF) AT TEMP  (HF) AT 35    MISC DATA     DISCRETES     LOOP
UNIT TYPE NO. UNIT TYPE NO. UNIT TYPE NO. UNIT TYPE NO. UNIT TYPE NO. UNIT TYPE NO.

  1  AI   46    1  PP   47    1  PP   48    1  AI   47    1  AI   51    1  CO    7
  1  AI   48    1  PP   52    1  PP   59    1  AI   59    1  PP   34    1  PP   22
  1  AI   50    1  PP   30    1  PP   31    1  AI   52    1  PP   37    1  AI   63
  1  AI   54    1  PP    1    1  PP   16    1  AI   49    1  PP   11    1  CO    8
  1  AI   56    1  PP   25    1  PP   35    1  AI   57    1  AI   31    1  PP   24
  1  AI   58    1  PP   36    1  PP   40    1  AI   65    1  AI   53    1  AI   64

   AO            AI            PP            CO            CS            CD
   LS            SP            SS            DS            NO ENTRY      --MORE--
```


8
7
6
5
4
3
2
1
D
C
B
A
+15
D5
Z=11V
2K
R18
-15
2K
1%
R5
SK3466
Q1
12
11
6
5
9
4
7
13
LM723
3.01k
1%
R1
2K
1%
R3
3.01K
1%
R2
3.01K
1%
R4
68PF
R17
110
1N914
D2
J1-6
56UH
J1-7
.2
C16
-V OUT
.01
C11
5K TEN-TURN
10K
R11,12,13,14
R6,7,8,9,10
10UF
C6,7,8,9,10
IC1
LF347N
(1/4)
5 CIRCUITS ARE REQUIRED
J1
14
IC1,7
IC1,8
IC1,14
IC2,1
15
16
17
18
19
TS-1
(RP)
OUTPUTS TO EXTERNAL ADC
PROBE 1
PROBE 2
PROBE 3
PROBE 4
CAL TEST
TITANIUM ELECTRODES
8
10
12
9
11
13
IC1,6
IC1,9
IC1,13
C13
C14
C15
10K
R CAL TEST
IC2,2
LF347N
IC 1
IC 2
2,6,9,13
3,5,10,12
1,7,8,14
1uF
BANANA JACKS FRONT PANEL
S1
1A.
120VAC
120VAC
AD920
@ 200MA
3
2,4
1
+15VDC
-15VDC
3.3UF
.01
R16
7.5K
POWER ON LED (FP)
D3
SUPPLIES UP INDICATOR
D4
LED 2MA.
R15
15K
ACAD=TITSYSM
WINCO
TITANIUM ELECTRODE
HF MEASURING SYSTEM
DRAWN BY
K.J. HASKELL
4/3/98

ILCM PRE-RUN CHECKOUT

Test run reagent loops on manual (15%) for water, ANN, Nitric acid, Final ANN. Be sure valves are open. Check for flow reading on correct turbine meter. Be as brief as possible.

Check turbine meter signal voltage on feed pump. Should be 0.6 to 4.7 volts. Run feed pump (briefly) in manual (8%) with valves closed to check direction of rotation.

Check TC's, tank levels, and densities for valid signals.

Check operation of probe loop recirculation pumps.

Check HF probe leads with precision resistor "probe" 2K to 50K ohms. Do not ground. Check for valid signals on proper channels on computer.

STARTUP CHECKLIST FOR ILCM OPERATION

Heat solution to desired temperature
Connect feed flowmeter to black cable #1
Connect TC's 1, 2, and 3 to sockets with same numbers.
Calibrate Probes

Record reagent tank volumes in logbook
Set HF target (CN36) .045
Set reagent concentrations for ANN (CN4) and Nitric Acid (CN5)
Set product Zr target (CN3)
Verify correct data tape is in DSU and storage of data to tape is allowed
Valid 25 degree C SPG (CN19), HF (CN6) in constant table.
Check calculated Zr and F/Zr values.
Unsave all trends. Save trends 3 and 7 on 5 minute interval.
Check Bar/Status 1 for all loops in manual.
Set CN7 (AN FF), CN18 (FIN AN FF) to 1; set switch 5 (AN FB) to 0.
Turn on all pumps: external control, check direction of rotation!
Fill reagent lines to addition point
Flow totalizer (DI3) reset, run (force on, unforce)
Turn on switches 3 and 4 (CAS IN 1 for cascade loops)
Set desired final tank level for shutdown event
Turn switches 1 and 2 off
Switch probe recirc loops from calibration solution to process solution.

OPERATION

Switch ANN (loop 1), water (loop 8), and Nitric acid (loop 7) to auto.
Loop 5 is feed. Set to auto and enter flow setpoint.
Watch for operation of these pumps.
Turn on switch 1 to enable shutdown once flow events are clear

Switch loop 2 to auto when a steady valid probe 1 reading is obtained (AI46, PP48),
Turn off switch 3 to forceback loop 2 (ANN first probe feedback - cascade middle loop)
Turn on switch 5 to add feedback control to ANN loop.

When a steady valid reading is obtained from probe 2 (AI48,PP59) switch loop 4 to automatic.
Toggle loop 2 to switch it to remote setpoint.
Turn off switch 4 to forceback loop 4 (ANN OUTER).

Monitor [HF] for approach to setpoint and all loop outputs for on-scale operation (not at 100%).

Shutdown at any time by turning switch 2 on.
Watch for feed depletion, product level, and high [HF] alarms.
Watch for reagent depletion.

SHUTDOWN

Normally shutdown occurs at 20 liters actual feed volume (adjusted for SPG) to leave a heel.
Enter 0 setpoint for loop 5 (Zr feed). Set to manual and enter 0 output.
Check Bar/status for flow controller outputs = 0. Switch to manual and set output at zero.
Check that pump rotation has stopped.
Turn off steam.
Record reagent volumes.
Print any desired screens.

DATE FILMED

11/22/93

END