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INSTRUMENTATION AND CONTROL PRACTICES IN
U. S. A. REPROCESSING PLANTS

L. A. Jobe

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PREFACE

The information contained in this report was presented at the Colloquy on Instrumentation in Plants for the Chemical Reprocessing of Irradiated Fuels, held in Paris, France on November 22-23, 1963. It is now being issued as an IDO report because the proceedings of that colloquy are not generally available.

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L. A. Jobe

A B S T R A C T

Instrumentation and control practices at the Hanford Plant, the Idaho Chemical Processing Plant, the Oak Ridge National Laboratory, and the Savannah River Plant are surveyed. Process variables are grouped, and the instruments which have been developed for use in measuring and controlling these variables are tabulated. Included in this tabulation are instruments for both in-line analytical and direct or inferential measurements. Several control loops are discussed from the systems engineering standpoint. The use of data loggers and computers is discussed briefly.

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INSTRUMENTATION AND CONTROL PRACTICES IN U. S. A. REPROCESSING PLANTS

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I. SUMMARY

A systems-oriented survey of instrumentation and control practices in U. S. A. reprocessing plants shows the relationship between the process, the measurement, and the control blocks in an integrated closed loop system. The design of instrumentation in these plants was originally based on steady state operations and on being able to satisfactorily adjust controller settings during plant operation. Current design of instrument and control systems, however, is being directed toward selection of proper instrumentation while utilizing the relationships between the various blocks of the closed loop system, which is illustrated in the following diagram.

The process block is the most difficult component of these closed loop systems to evaluate or describe mathematically. Reprocessing plants employ various processes depending upon the number and type of fuel elements handled. The Hanford Plant and the Savannah River Plant (SRP) employ a Purex process; the Idaho Chemical Processing Plant (ICPP) is a multi-process plant. The dynamic characteristics of the process block depend upon the process chosen and upon the size and design of the equipment used.

The measurement block covers a wide range of types of instruments used for the measurement of either primary or secondary variables. While much effort has been expended toward measurement of primary variables by in-line analytical instruments, most process control still relies upon measurement of secondary variables. In-line analytical instruments which have been developed to measure the concentration of fissionable or fission product materials include: alpha monitors, colorimeter-photometers, gamma absorptiometers, gamma monitors, neutron absorptiometers, neutron monitors, neutron multiplication detectors, pH monitors, and polarographs. These instruments are used for supervisory control and have not been incorporated into closed loop control. In addition, analytical control of the various operations is still largely accomplished by laboratory analysis of spot samples. Instruments for measurement of secondary variables have required less development. Typical secondary variables which are measured include: capacitance, conductivity, density, flow, interface level, liquid level, pressure, and temperature.

For the control block, pneumatic controllers, generally of the miniature graphic panel type, continue to predominate for most control instrumentation at all reprocessing sites in the United States. Miniature electronic instrumentation has been installed on some new facilities at the several sites. Control valves, however, remain almost exclusively pneumatic. Simple computing modules, available in both pneumatic and electronic lines, are finding such applications as computing true liquid level of a solution subject to density changes and correcting gas flows for pressure and temperature changes. Data loggers and computers have proved to be useful tools at the Hanford Laboratories and the Oak Ridge National Laboratory.

Probably the most significant future advances in instrumentation and control of chemical reprocessing plants will be achieved through further utilization of the systems engineering viewpoint and various process dynamics approaches. The application of the mathematical tools of systems engineering to the solution of control problems is best exemplified by the design of a control system for an electrically heated UO_3 calciner at Hanford and a cascade control system for a thermosiphon evaporator at the ICPP.

II. INTRODUCTION

Instrumentation is generally recognized as necessary in the chemical processing industry for measuring and controlling operating variables. Until about five years ago the design of instrumentation in both the chemical and petroleum industries, although highly instrumented, was based on assuming steady state operations and modifying the instrumentation during the plant operation. This technique was adopted for the conventional instrumentation employed for the original chemical reprocessing plants for irradiated fuels in the United States. Special analytical in-line instruments were in their infancy; furthermore, the radiation fields unique to the nuclear industry prevented direct application of available in-line instrumentation in chemical reprocessing plants. Although some recently developed instruments have been installed, adoption of further in-line instrumentation, data loggers, and computers to replace existing instrumentation is difficult to justify economically. On the other hand, new plants can make an optimum choice from currently available instrumentation.

Analytical control of the various operations in chemical reprocessing plants is still accomplished to a great extent by drawing spot samples for laboratory analysis. Remote manipulation techniques, which must be used for handling radioactive samples in transport and in the laboratory, result in up to a four hour delay in analytical results⁽¹⁾. Because of this delay, feed and catch tanks must be supplied for each unit operation in a process, with the product from each operation being retained until the analysis is available. In many cases duplicate hold tanks must be provided to maintain continuous operation of a plant.

Optimization of radiochemical reprocessing plants can best be achieved by adopting an overall systems engineering viewpoint as represented in Figure 1⁽²⁾, which is taken from an instrumentation

Figure 1. Systems Engineering Diagram

research and development program written for the ICPP in 1957⁽³⁾. In this system a process, including the chemical kinetics, heat, mass, and momentum transfer, instrumentation, and control, is treated as a single problem handled by a process dynamics (i.e., unsteady state) technique. An example of this philosophy as applied to a solvent extraction column, the heart of today's chemical reprocessing plants, is illustrated in Figure 2⁽³⁾.

A quick survey of the several component parts and the entire "closed loop" used in United States reprocessing plants forms the basis for this report.

Figure 2. Schematic Diagram of Extraction Column Control System

III. THE PROCESS BLOCK

The process block of Figure 1 is the most difficult component of the entire loop to describe mathematically. It comprises a number of interrelated unit operations and reactions; its mathematical description is thus affected by the complexity of the process and by the characteristics of the actual equipment used in carrying out these operations and reactions.

Reprocessing plants in the United States employ various processes depending on the number and type of fuel elements handled. Figure 3 shows the Purex two-cycle process employed at Hanford⁽⁴⁾ except for the initial processing steps of fuel element dejacketing and the initial nitric acid dissolution. An important feature of this process affecting overall instrumentation and control of the plant is the recycle of second cycle aqueous raffinate to the first cycle extraction column. As shown in Figure 3, alpha, gamma, and neutron monitors are used extensively in this process.

A Purex process is also employed at the Savannah River Plant, but there the aqueous raffinate is not recycled. Also, at the Savannah River Plant, mixer-settlers are used whereas pulse columns are employed at Hanford.

Figure 3. Flow Diagram for Purex Process at Hanford

The Idaho Chemical Processing Plant (ICPP) is a multipurpose re-processing plant. Figure 4 is a flow diagram for the TBP (tributyl phosphate) first extraction cycle and for the Hexone second and third extraction cycles. Figure 5 shows an air-pulsed extraction column which was developed at the ICPP⁽⁵⁾.

Figure 4. Simplified Flowsheet for Continuous Processing of Aluminum-Uranium Fuels at ICPP

Figure 5. Air-Pulsed Extraction Column

IV. THE MEASUREMENT BLOCK

Variables

Process control requires the measurement of either primary or secondary variables. Primary control depends upon the ability to measure continuously a primary variable such as chemical composition (for recovery) or radioactivity (for decontamination). While much effort has been expended in the last decade toward measurement of primary variables, most process control still relies upon measurement of secondary variables. The most common of these secondary variables and typical uses are given in the following list:

1. Density for controlling steam input to evaporators.
2. Interface level for controlling flow rates to or from extraction columns.
3. Flow rate for maintaining constant feed rates and, in some cases, for ratioing the flow of a second stream.
4. Liquid level for controlling flows from constant head tanks and evaporators. In some cases liquid level can be used for dual control of an evaporator during start-up.

5. Acidity or alkalinity for controlling the addition of various reagents to a process.
6. Pressure for regulating air bleed rate to off-gas systems and air rate to air lifts.
7. Temperature for regulating steam input to evaporators and cooling water to condensers.

There are, of course, many other variables that can be used for control or to divert a stream to a hold tank when a process characteristic reaches an alarm setting.

Measuring Instruments

In general, measuring instruments can be divided into two categories: "in-line" analytical instruments and (non-analytical) process-characteristic measuring instruments. Characteristics common to both types of instruments are given in Table I, an expansion of the Cooper and Wallings Table⁽⁷⁾. In Table I, several ranges are shown for the same type of instrument to indicate that two or more instruments with different ranges have been developed. Review papers by Alkire⁽⁸⁾, Axtmann⁽⁹⁾, Pleasance⁽¹⁰⁾, and Wilson⁽¹⁾ discuss many of these instruments.

Considerable effort has been expended in the last decade to develop highly specialized instruments to monitor continuously certain variables. These instruments, commonly referred to as "in-line" instruments, can measure properties such as chemical or electrical equivalence, refractive index, radioactive characteristics, nuclear resonance, spectral characteristics, acidity, and temperature. In-line instruments not only monitor, but, in many cases, can detect and correct process deviations in a fraction of the time required for a laboratory analysis.

The following in-line instruments, included in Table I, are capable of yielding analytical results in terms of specific activities or concentrations of a desired element and thus may be called primary variable measuring instruments.

1. Alpha Monitor: Figure 6 shows an alpha monitor developed at Hanford⁽⁸⁾ and suitable for measuring plutonium in a medium concentration range of 0.05 to 10 g Pu/l. Solid-state silicon detectors have recently been reported^(14,15) as being highly sensitive to alpha radiation while rejecting the effect of a 50 R/hr gamma field. A low level alpha counter with less than 0.001 counts/minute background can be used for bioassay samples, while another unit has monitored very high activity streams.

Figure 6. Scintillating Glass Alpha Detector

TABLE I
SUMMARY OF IN-LINE PROCESS INSTRUMENTATION

INSTRUMENT AND PRINCIPLE OF OPERATION	VARIABLES DETERMINED	RELIABILITY	STATE OF DEVELOPMENT	ACCURACY	RANGE OF INSTRUMENTS USED	REFERENCE TO INSTRUMENT USE
Alpha Monitor: Counts pulses from phototube activated by visible light from interaction of alpha particles with zinc sulphide phosphor in direct contact with solutions. The solid state silicon wafer type of alpha monitor is sensitive to alpha while discriminating against high gamma radiation.	Total alpha activity from various emitters: Pu concentrations at medium range (in aqueous solutions). Traces of alpha activity in waste streams. High levels of alpha activity in process solutions.	Good; system is simple. Suitable for in-canyon installation.	Number of units in operation; no sampling. Work needed on organic streams and better phosphors. This type of detector has been prototyped.	± 20%	0.05 to 10 g Pu/l; detection limit of 0.01 g/l	6,11
	Low level for bio assays.				3.3×10^{-4} µCuries/ml	12
	High level for process streams.				5×10^3 to 1.5×10^8 dis/min-ml	13
						14 15
Alpha Printer: Same as alpha monitor except it measures, washes, and dries samples before counting.	Total alpha from various emitters; used principally for detection of Pu at low concentrations (in aqueous solutions).	Good at Hanford; system is a complex electrical mechanical device. Abandoned by SRF.	Development completed; effort required to simplify system. SRP has replaced all su ¹ units	± 15%	10^{-6} to 0.1 g Pu/l	16
					5×10^{-5} µCuries/ml	17
						18
Colorimeter: Measures current from phototube caused by absorption of visible light in solution; depends upon specific wavelength absorption of various substances; absorption is proportional to concentration.	Concentration of light absorbing materials: U, Pu, and dichromate concentrations.	Good; system is simple; subject to interference from other colored ions and decomposition products in organic materials.	Development of sampling cells and light sources fairly complete; full-stream monitors adapted for commercial units in use.	± 5% ± 20% ± 3% ± 1%	30 to 500 g U/l	7,10,20,28
					2 to 32 g Cr ₂ O ₇ ²⁻ /l	7,21
					26 to 55 g U/l	22,26
					0.1 to 70 g U/l and 0.1 to 10 g Pu/l	23
	U in the presence of Fe.				0.1 to 2 g U/l	25
	U in waste solutions.				0.0002 to 0.002 g U/l with added reagent	24
Dielectric Constant: Measures change of capacitance, with solution forming part of capacitance.	% TBP in diluent electrolyte in organic.	Failed at ORNL: Used in Hanford labs.	Development needed on instrumentation and interpretation.	± 0.5%	20-30% TBP	7
	Interface level.	Used routinely in column interface control.	Interface control needs development for high radiation fields and removal of metal deposits.	± 1/8"	0 to 48"	7,29
		Abandoned at SRF.				30
Conductivity: Measures resistance of solution with an a.c. bridge; conductivity is proportional to concentration of electrolyte.	Concentration of electrolyte in solution, eg, other measurements plus conductivity can determine concentration of acid and U in water.	Excellent; simple system; can be used directly in stream or vessel; no sampling needed.	Instruments commercially available; continuous in-line acidity measurements being made.	+ 7% < 0.03 M ± 0.2N	0.01 to 10 ⁶ ohms	8
					0.1 to 0.4M HNO ₃	31
	Can be used to measure liquid level also.				0.1 to 0.9 ohm ⁻¹ cm ⁻¹	45
Gamma Absorptiometer: Measures current from ion chamber or scintillation detector caused by gamma radiation from a fixed source; radiation is absorbed by solution components in proportion to concentration and mass absorption coefficient.	Concentration of heavy elements: U, Pu, and Th. Will not distinguish between these elements; practically unaffected by light element concentration.	Excellent; system is simple; can be installed in-canyon because maintenance is small.	Usable instruments complete in plants. Data logging gamma absorptiometer operated over a year in pilot plant.	± 7% ± 0.3% ± 0.5 g U/l	5 to 300 g U/l	32,33,34
					300 to 420 g U/l	35
					0 to 120 g U/l	36
Gamma Monitor: Measures current or pulses from multiplier phototube activated by light from NaI(Tl) scintillation crystal caused by interaction of gamma photons with crystal; can give total gamma, gamma energy spectrum, or a specific gamma energy.	Total gamma activity or gamma energy spectrum or a single gamma emitter. Can be used to detect presence and quantity of gamma emitters in solids, liquids, or gases.	Good; dependent on sampling and cell decontamination.	Well advanced; in-line unit being used with 400 channel analyzer.	± 15%	0.01 to 30 Curies/ml (Cs-137 or equiv.)	37,38
					10^{-5} to 10^{-3} Curies/ml	39
					0 to 1.2×10^4 c/min-ml	40
					5 decades of c/min	26,27
Neutron Monitor: Measures current or pulses from BF ₃ counters or other detectors caused by neutron interaction with light elements to produce alpha particles.	Neutrons produced by alpha-neutron reaction. Alpha emitters such as U, Pu, Am, Cm, produce neutrons when in solution in proportion to conc. Analysis obtained by(a,n) reaction with light elements O,F,N, etc. Useful for Pu and U-235.	Excellent; no sampling system; primary element reliable; maintenance low.	Extensive research and development needed. SRP has done considerable work. Calibration and shielding need thorough study. Neutron multiplication technique developed.	± 5% ± 5% ± 10%	~ 1.0 to 60 g Pu/l	7
					3 to 100 g/l	8
					0.1 to 100 g Pu/l	42
					1 to 3 g Pu/l	41
					0.5 to 12 g U-235/l	61
pH Monitor: Measures potential developed between glass and calomel electrode due to the hydrogen ion concentration; provision made for intermittent calibration against standard solutions.	Concentration of acid or base in aqueous solutions.	Good; system is fairly simple.	Completed for operation on sampled solutions. Number of continuous in-line instruments also installed.	± 0.1 pH	0 to 14 pH	8,10,47,62
Polarograph: Measures current from reduction of ion to lower valence state; current is proportional to the ionic concentration of reducible species.	Concentration of various ions in aqueous solutions; especially useful for small concentrations of U.	Good, but system is quite complex; U measurement subject to interference from organics, iron, etc.	Development of plant unit well along; not suitable for full-stream monitor; cyclic but automatic. Prototype installed in raw waste system.	± 20%	0.02 to 6 g U/l	10
					0.01 to 10 g U/l	8
					0.002 to 0.02 g U/l	54
					0.005 to 0.1 g U/l	55
					0.02 to 2 g U/l	63
Reduction-Oxidation: Measures interelectrode potential between measuring and reference electrodes.	Valence state particularly for Pu. Ionic concentration, eg, Cl.	Probably as good as pH.	Instrumentation exists; a considerable amount of work needed on application and interpretation of data.			
Turbidimeter: Measures light transmitted; variation of colorimeter.	Aqueous phase dispersed in organic and vice versa.	Probably equal to colorimeter.	Instrument exists; work needed on interpretation of data and application. Sampling problem.	0.2%	0-1% organic	60

Figure 7. Uranium Photometer

2. Colorimeter-Photometer: A uranium photometer, developed at the ICPP, and its principle of operation is shown in Figure 7(2). This unit is suitable for measuring intermediate ranges of uranium concentration, 0 to 2 g/l, and thus is different from the high concentration (30 to 500 g/l) photometers developed at Hanford(8), SRP(23), and ORNL(26). Figure 8(25) compares the absorption wave lengths of several potential interfering elements and indicates the potential photometric difficulties which may be encountered.

Figure 8. Absorption Spectra of Some Potential Interfering Ions

Figure 9 shows the transmission spectra of the glass filters chosen for uranium measurement at 425 millimicrons and standardization at 525 millimicrons. Figure 10 is a schematic diagram of the photometer measuring circuit.

Figure 9. Absorption Spectrum For Simulated Raffinate and Typical Transmission Spectra of Glass Filters.

Figure 10. Schematic Diagram of Measuring Circuit for Photometer

An indirect colorimetric procedure for the determination of enriched uranium from dilute waste solutions has been reported by SRP⁽²⁴⁾; this method is adaptable to in-line colorimetry.

3. Gamma Monitor: The unit shown in Figure 11, developed through the prototype stage⁽²⁶⁾, employs a novel way of canceling out background variations by sinusoidally varying the volume of solution seen by the detector, resulting in its seeing an alternating component dependent almost entirely on the activity of the sample stream. A similar pulsation is used in standardizing the instrument. At Hanford, continuous in-line units have been installed to measure the decontamination factor during each processing cycle.

Figure 11. Experimental In-Line Gamma Monitor

4. Gamma Absorptiometer: A gamma absorptiometer, coupled to a data logger⁽³⁶⁾, has been developed that is capable of analyzing an average of 30

samples per eight-hour day, measuring uranium concentrations of 0 to 60 and from 60 to 120 g/l with an accuracy of ± 0.5 g/l.

5. Neutron Monitor: A neutron monitor developed at SRP⁽⁴²⁾ for plutonium inventory control is shown in Figure 12. Early difficulties with reliability of the monitor were reduced by using a transistorized preamplifier and relocating all components except the pulse transformer net work away from the detector⁽⁴³⁾. A neutron absorptiometer (with a response time of five seconds) for on-stream analysis of boron was reported at the 1961 ISA Annual Conference⁽⁴⁴⁾. A neutron multiplication technique to indicate the buildup of U-235 in radiochemical separations plants has also been reported⁽⁶¹⁾.

Figure 12. A Typical Neutron Monitoring System

6. pH Monitor: At Hanford, commercially available electrodes with remote connectors have been adapted to the handling of high-impedance coaxial cables for remote installation in process vessels. These units are suitable for low radiation areas. The pH flow cell shown in Figure 13 also was developed at Hanford and has been installed directly in sample lines in higher radiation areas⁽⁸⁾. One pH monitor is being used to control acid addition to a recycled waste system, and others are being used for continuous in-line pH measurement⁽⁴⁾.

Figure 13. pH Flow Cell

7. Polarograph: Several commercial polarographs have been modified for use in the nuclear industry; however, no report of their use has been made. A modified polarograph cell is shown in Figure 14.

Figure 14. Sectional View of a Polarograph Cell

The following secondary variables, although not having the analytical capabilities of the primary variables previously discussed, are the common process variables measured and controlled in plant installations.

1. Capacitance: Capacitance probes have been used at Hanford routinely for column interface control, and at the ICPP as limit alarms. Information from SRP, however, indicates that capacitance probes are easily fouled by metallic film and therefore are not satisfactory⁽³⁰⁾. Fouling problems have also been encountered at the ICPP. These probes are satisfactory, however, for clean fluids. Stainless steel sheathed probes are available for normal corrosive service.
2. Conductivity: An in-line conductivity instrument has been in use at Hanford to measure acidity⁽⁴⁾. Procedures using conductance plus density and conductances at two accurately controlled dilutions have been reported from the ICPP⁽⁴⁵⁾ as suitable for analyzing for stainless steel nitrates in electrolytic dissolver product solutions.
3. Density: Solution density is usually determined by air-purge bubblers; an example is shown in Figure 15. When air in the system cannot be tolerated, either a rotameter with a special float and inductance coil transmitter⁽⁴⁶⁾, or a device which transmits the change in buoyant force to a strain-gage load cell, as shown in Figure 16, can be employed.

Figure 15. Typical Columns Showing Instrument Probes for Determining Density, Liquid Level, and Interface

Figure 16. In-Line Density Monitor

4. Flow: A variety of flow instruments are in use at the various processing sites. In several situations at Hanford and the ICPP, where pressure drop is critical, electromagnetic flowmeters have been installed. Some difficulties have been encountered at the ICPP with these meters when used in conjunction with miniature secondary receiver-transmitters. On the other hand, during simultaneous testing, one magnetic flowmeter with a miniature secondary has been far superior in accuracy, linearity, reproductibility, and hysteresis to two different brands of rotameters having pneumatic transmitters.
5. Interface Level: Dip-tube probes are generally used to determine interface levels. However, dip tubes require frequent purging and recalibration for top interface applications and have been found to be unsatisfactory for determining bottom interface levels. A remote float-type liquid interface controller, which can be used to control either top or bottom interfaces has been reported from Hanford⁽⁴⁸⁾. This device utilizes an inductance coil transmitter. Numerous inductance coil interface level indicators of this type are now in use.
6. Level: Level measurements usually are obtained by air-purged dip tubes which indicate the pressure equivalent to the weight of the column of liquid. True liquid level indication results from dividing the column weight by the solution density. This principle has been incorporated into a control system for evaporator start-up⁽⁴⁹⁾. Float type units are less influenced by change in density than bubblers; however, they are subject to wear and are therefore not as desirable in nuclear installations.

V. THE CONTROLLER BLOCK

Conventional pneumatic controllers, generally of the miniature graphic panel type, continue to be utilized for most control instrumentation at all reprocessing sites in the United States. However, temperatures are recorded by large multipoint strip chart recording potentiometers.

A number of miniature electronic instrumentation lines have appeared in the United States during the last six years, and a few miniature electronic instruments have been installed on new facilities at the several sites. An advantage of electronic instrumentation is that it can be directly coupled to data loggers and computers. Electronic final control elements are not accepted yet as equals to their pneumatic counterparts, however. The conversion from electronic to pneumatic signals often restricts the adoption of electronic systems; one reason is that converter response characteristics often nullify any advantages of electronic systems.

Simple computing modules to perform addition, subtraction, averaging, multiplication, division, or square-root extraction operations are now available for both electronic and pneumatic instrumentation. Pneumatic computing relays are being used to compute true liquid level from the density of a solution and the pressure corresponding to the weight of the column of solution above a given location. In the start-up of evaporators, this true liquid level measurement is sometimes coupled with automatic switching from level to density control as soon as the desired density has been achieved. Other specific applications include: (1) ratioing relays for regulating back-pressure to the pressure weirs used to control raffinate flows from pulse columns and (2) electronic computing transformers to correct air flow rates for pressure and temperature changes.

VI. CONTROL LOOP SYSTEMS

Personnel at Hanford have been particularly active in applying the mathematical tools of systems engineering to the solution of control problems, as well as in the development and application of in-line instrumentation in their processing facilities. An example is the application of a closed-loop control system, developed with simulation techniques^(50,51), to the electrically heated UO_3 calciner furnace shown in Figure 17. Heat transfer equations for this calciner were derived using an assumed mathematical model which was then simulated on an analog computer; coefficients in the equations were adjusted until the

Figure 17. Continuous Calciner
Employed at Hanford

simulated and actual furnace curves were in agreement. A simulated three-mode controller was then added and closed-loop operation was tested on the analog computer. The end result was controller settings producing stable operation with minimum error and overshoot, both during start-up and system upset conditions.

Figure 18. Automatic Calciner Control System Block Diagram

The block diagram for this system is shown in Figure 18. Figure 19 shows the open loop transient response to a step change of input power of the exact furnace model and the closed loop response to a programmed start-up condition. Figure 20 shows the simulated closed-loop system response to ± 20 percent changes in heater supply voltage or in desired UO_2 powder temperature. The maximum error of shell temperature for the installed system was only $\pm 2.5^\circ F$ as contrasted with $\pm 7^\circ F$ for the simulated model.

Figure 19. Open and Closed-Loop Response

Figure 20. Simulated Closed-Loop Response to Changes in Voltage and Temperature.

The best example of systems engineering design at the ICPP is the control system for a first cycle product evaporator. This thermosiphon evaporator and the control system⁽⁵²⁾ are shown in Figure 21. In this system the steam flow to the evaporator is proportioned to the evaporator feed rate by a magnetic flowmeter-controller, and the ratio of these flow rates is adjusted by a density controller to maintain a specified density. The final representation of the evaporator system is that of a well agitated, steam jacketed kettle, as shown in Figure 22. The block diagram for this system is shown in Figure 23. The process transfer function was determined by a transient test of the evaporator, and, as shown in Figure 24, the evaporator can be represented by a single capacitance (first order) system. This evaporator control system has maintained proper control even when the evaporator feed rate varied abruptly between 20 percent and 115 percent of the mean flow rate⁽⁵³⁾.

Figure 21. Control System for First Cycle Product Evaporator

Figure 22. Final Representation of Evaporator System

Figure 23. Block Diagram for Evaporator System

Figure 24. Transient Test of First Cycle Product Evaporator

VII. DATA LOGGING AND USE OF COMPUTERS

Data loggers and process computers have been utilized with fuel re-processing to the greatest extent at Hanford and ORNL. The techniques employed at Hanford to develop mathematical models of various processes are outlined in Figure 25. Details of a program for developing a model of a solvent extraction system have previously been reported^(56, 57, 58). Briefly, a data logger has been used to reduce primary data, including stream compositions, flow rates, and other process parameters, to a digital form after which it is processed on an IBM 709 computer. Linear regression techniques have been used to determine coefficients of mathematical models.

At ORNL two data loggers have been used in development studies. A technique employed at ORNL for processing data is outlined in Figure 26⁽⁵⁹⁾. Briefly, the data reduction sequence consists of digitizing conventional instrumentation signals with an on-line automatic data logger and processing the data with an off-line digital computer. Conclusions resulting from ORNL experience with data loggers and computers are as follows:

1. A data logger has limited value unless it can be used in conjunction with a computer.
2. Up to 70 percent of computer operating time is utilized in reducing the data to usable form and then later converting it to useful engineering units.
3. Automatic data logging must be supplemented by manual data recording because data loggers break down occasionally.
4. On-line computation of data for process control was not justified for the Fluoride Volatility Pilot Plant.

Figure 25. Hanford Techniques For Mathematical Model Development

Figure 26. ORNL Techniques For Data Reduction

VIII. LITERATURE CITED

1. Wilson, John N., Reducing Nuclear Power Costs With Improved Instruments, Paper Presented at 1962 Nuclear Congress, June 4-7, 1962, New York, New York.
2. McBride, J. A., Technical Programs and Projects at the Idaho Chemical Processing Plant, IDO-14555, April, 1961.
3. Schneider, Harry, Chemical Processing Instrumentation and Control Research Program, IDO-14420, September, 1957.
4. Harmon, M. K., Current Status of Solvent Extraction Processing of Irradiated Uranium Fuels, Paper Presented at 1962 Nuclear Congress, June 4-7, 1962, New York, New York.
5. Weech, M. E., R. S. P'Pool, D. K. MacQueen, Interim Report on the Development of an Air Pulser for Pulse Column Application, IDO-14559, September, 1961.
6. Parsegian, V. L., Instrument Needs of the Radiochemical Processing Plant, Nucleonics, pp. 38-53, January, 1950.
7. Cooper, V. R., M. T. Walling, Jr., Aqueous Processes for Separation and Decontamination of Irradiated Fuels, Second United Nations International Conference on the Peaceful Uses of Atomic Energy, Geneva, Switzerland, Vol. 17, pp 320-321, 1958.
8. Alkire, G. J., Continuous Analyzers for Processing Irradiated Uranium, Industrial and Engineering Chemistry, 52, p. 481, June 1960.
9. Axtmann, Robert C., Analytical Instrumentation Problems in Radiochemical Processes, Control Engineering, p. 94, January, 1959.
10. Pleasance, C. L., Continuous Analysis in Nuclear Processing, ISA Journal, 5, p. 39, June, 1958.
11. Upson, U. L., P. E. Brown, A Liquid-Contact Scintillation Alpha Counter, HW-49003 (Del), March, 1959.
12. Wingfield, E. C., An Alpha Monitor for Waste Streams, DP-197, January, 1957.
13. Pillinger, W. L., An Alpha Monitor for Process Streams, DP-302, July, 1958.
14. Cathey, L., Low Level Alpha Counting with Solid State Detectors, IRE Transactions on Nuclear Science NS-8, p. 10, October, 1961.
15. Cathey, L., W. J. Jenkins, The Operation of Solid-State Alpha Counters in Chemical Process Streams, IRE Transactions on Nuclear Science NS 9 : pp. 143-9, June, 1962.

- 16.. Hildreth, N. T., Design Criteria In-Line Alpha Monitor., HW-41525, 1956..
- 17.. Wingfield, E. C., P. R. Liller, A Monitor For Low-Level Radio-activity in Liquid Streams., DP-145, January, 1956..
- 18.. New, L. B., Private Communication, Savannah River Plant, October 23, 1963..
- 19.. Alkire, G. J., Technical Specifications for Uranium Colorimeters, HW-39823 (Rev. 1), 1956..
- 20.. Scott, F. A., An Analysis of the In-Line Uranium Photometer Data From Purex Hot Semi-Works --- Runs PX-2 Through PX-9, HW-40313, 1955..
- 21.. Dierks, R. D., Information For The Application of Dual-Filter Photometers for Continuous Analysis of Plant Streams, HW-50355, 1957..
- 22.. Prohaska, C. A., A Flow Colorimeter For Measuring Uranium Concentration in Process Streams, DP-229, August, 1957..
- 23.. Colvin, D. W., A Colorimeter for In-Line Analysis of Uranium and Plutonium Solutions, DP-461, March, 1960..
- 24.. Fulda, M. L., Determination of Enriched Uranium in Process Waste Streams, DP-610, August, 1961..
- 25.. Erickson, E. E., C. M. Slansky, A Continuous Photometer for Low Concentrations of Uranium in Aluminum Nitrate-Nitric Acid Solutions, IRE Transactions on Nuclear Science NS-8, p. 83, October, 1961..
- 26.. Landry, J. W., In-Line Instrumentation: Gamma Monitor, Uranium Colorimeter, ORNL-2978, September, 1960..
- 27.. Landry, J. W., In-Line Instrumentation: Gamma Monitor, Uranium Colorimeter, Preprint Number 3-LA-61, ISA Conference, Los Angeles, California, September 11-15, 1961..
- 28.. Scott, F. A., R. D. Dierks, Photometer for Continuous Determination of Uranium in Radioactive Process Streams, Analytical Chemistry, 32, p. 268, February, 1960..
- 29.. Simsen, C. A., HAPO Interface Monitor Model III Instruction and Maintenance Manual, HW-41691, February, 1956..
- 30.. Lovell, B. W., Personal Communication, Savannah River Plant, October 23, 1963..
- 31.. Colvin, D. W., An In-Line Conductivity Meter with Temperature Compensation, DP-361, 1959..

32. Miller, D. G., R. E. Connally, A Gamma Absorptiometer for the In-Line Determination of Plutonium or Uranium, HW-36788, June, 1955.
33. Connally, R. E., U. L. Upson, P. E. Brown, and F. O. Brauer, Uranium Analysis by Gamma Absorptiometry, HW-54438, 1958.
34. Alkire, G. J., Information for Application of Gamma Absorptiometers for Chemical Processes, HW-45664, 1956.
35. Thurnau, D. H., A Gamma Absorptiometer for the Continuous Analysis of Solutions of Heavy Metal Salts, DP-249, November, 1957.
36. Wilburn, N. P., W. L. Nicholson, A Data Logging Gamma Absorptiometer for Routine Uranium Analysis, ISA Proc. 17, Paper No. 60.1.62, 1962.
37. Leboeuf, M. B., R. E. Connally, U. F. Upson, A Continuous Gamma Activity Monitor for the Product Stream of the Uranium Recovery Plant, HW-29348, 1953.
38. Leboeuf, M. B., R. E. Connally, L. F. Miller, Dual Function System for Gamma Radiation Monitoring in an Extraction Process Pilot Plant, HW-33901, 1954.
39. Wingfield, E. C., A Beta-Gamma Monitor for Liquid Streams, DP-177, September, 1956.
40. Wingfield, E. C., A Gamma Monitor for Liquid Streams, DP-342, December, 1958.
41. Dierks, R. D., The Development of the HLO Pipeline Plutonium Monitor, HW-57662, 1958.
42. Kropp, W. A., Neutron Monitor for Plutonium Inventory Control, DP-267, January, 1958.
43. Lapsley, A. C., Neutron Monitor for Inaccessible Locations, DP-362, April, 1959.
44. Pleasance, C. L., A Neutron Absorptiometer for the On-Stream Analysis of Boron, Preprint Number 29-LA-61, Fall Instrument-Automation Conference and Exhibit, September 11-15, 1961, Los Angeles, California.
45. Pearson, D. P., The In-Line Analysis of Electrolytic Dissolver Solutions for Nitric Acid and Salt Concentrations, IDO-14603, March, 1963.
46. Huck, C. E., Density Monitor For Purex HA Column, HW-56674, July, 1958.
47. Michelson, C. E., pH Monitoring of Metal Recovery Neutralized Waste, HW-33148, September, 1954.

48. Hahn, K. J., H. M. Jones, A Remotable Float-Type Liquid Interface Controller, HW-55166, February, 1958.
49. Bower, J. R., Chemical Processing Technology Quarterly Progress Report, IDO-14599, p. 32, July-September, 1962.
50. Huck, C. E., The Application to a UO₂ Calciner Furnace of a Closed Loop Control System Developed Using Simulation Techniques, HW-SA-2433, April, 1962.
51. Huck, C. E., How an Analog Computer Helped Design a Control System, ISA Journal, 10, pp. 57-62, April, 1963.
52. Bower, J. R., Chemical Processing Technology Quarterly Progress Report, IDO-14574, pp. 36-38, July-September, 1961.
53. Bower, J. R., Chemical Processing Technology Quarterly Progress Report, IDO-14583, pp. 35-37, October-December, 1961.
54. Propst, R. C., In-Line Polarography of Uranium in Process Waste, DP-572, June, 1961.
55. Propst, R. C., A Recording Polarograph for Routine Analyses, DP-618, August, 1961.
56. Wilburn, N. P., R. D. Dierks, Program For the Development of A Mathematical Model of a Solvent Extraction System, HW-64623, April, 1960.
57. Wilburn, N. P., R. D. Dierks, Techniques for the Development of a Mathematical Model of a Solvent Extraction System, HW-SA-2010, May, 1961.
58. Wilburn, N. P., Mathematical Determination of Concentration Profiles in Two Phase Continuous Countercurrent Extractors, HW-SA-3139, August, 1963.
59. Moncrief, E. C., M. C. Hill, Evaluation of Automatic Data Processing in the Fluoride Volatility Pilot Plant, ORNL-3371, January, 1963.
60. Colvin, D. W., A Turbidimeter for In-Process Analysis of Organic Contamination, DP-178, October, 1956.
61. Lapsley, A. C., Detection of U²³⁵ Buildup in Separations Processes by Neutron Multiplication, DP-533, December, 1960.
62. Hahn, K. J., K. Koyama, An Industrial-Type Flow Cell for Monitoring pH, Industrial and Engineering Chemistry, 49, No. 11, p 63A, 1957.
63. Alkire, G. J., et al., Plant-Type Polarographic System for Determining Uranium in Radioactive Waste Streams, Analytical Chemistry, 30, p. 1912, 1958.

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