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CHEMICAL PROCESSING INSTRUMENTATION AND CONTROL RESEARCH PROGRAM

Harry Schneider

September 18, 1957

AEC RESEARCH AND DEVELOPMENT REPORT

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CHEMICAL PROCESSING INSTRUMENTATION AND CONTROL RESEARCH PROGRAM

By

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September 30, 1957

Chemical Development Section
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Phillips Petroleum Company
Atomic Energy Division

Under Contract
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Idaho Operations Office
U. S. Atomic Energy Commission

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I. Introduction

In the interest of reducing nuclear fuel reprocessing costs at ICPP and providing advanced technology for future plant design, the potentiality of improved process control techniques, and eventual process automation, cannot be overlooked. The benefits obtained through automatic control in other chemical industries is the subject of a multitude of reports in the open literature. There is no apparent reason why similar benefits should not be realized in the nuclear fuel reprocessing field.

Although it is difficult to assign true economic value to all the benefits attainable through improved control techniques some are fairly obvious. At CPP such factors as increased capacity, reduced operating manpower, reduced recycle time and decreased inventories should all lead to reduced costs. For future plants, even greater potential exists through reduced investment cost as well.

The most notable advances in process control technology in recent years have been accomplished through the application of continuous automatic analytical instruments. The knowledge of instantaneous stream composition provided by these instruments is by itself beneficial to the operation of the process. More important, however, is the potential created for automatic control of the process on the basis of measurement of direct process criteria. This potential makes the automatic plant appear more feasible and has created the incentive for these studies. The long range objective of most of these studies is process automation.

A program for the development of analytical instrumentation and improved control techniques using these instruments as applied to nuclear fuel reprocessing is currently being undertaken by the Chemical Development Section of the CPP Technical Branch. The program philosophy and objectives, a survey of the problems involved, general approach, lines of endeavor and specific applications are presented in this report.

II. General Program Objectives and Justification

It is a well recognized fact that the success of a continuous chemical process hinges directly upon the ability to control the process. As a rule, the necessary control is accomplished directly or indirectly through two major channels; instrumentation and chemical analysis.

In the usual sense, instrumentation refers to the application of automatic devices to the control of a process or unit operation by continuous measurement of properties such as temperature, pressure, flow rates, liquid level, etc. Chemical control, on the other hand, refers to the semi-continuous determination of stream compositions by measurement of properties such as color, chemical or electrical equivalence, density, viscosity, refractive index, radioactive characteristics, etc. As a rule, instrumentation must be coupled with chemical analysis to maintain complete process operability and versatility. When chemical analysis is applied to the end product of the unit operation or process, it also affords product quality control.

In recent years much attention has been focused upon the development of specialized instruments to perform the same function as the chemical control. Since flowing stream properties are measured, this type of instrumentation is commonly referred to as "in-line" instrumentation.

Much confusion has arisen concerning the function of in-line instruments and the objectives of related development programs. In most cases, the in-line instrument has been preceded by a chemical control method; it would seem, then that the function of the instruments is to reduce or eliminate the personnel, equipment, and time associated with the chemical control method. It should also follow that the primary objective of an in-line instrument development program is reduction in process investment and/or operating costs through this channel. The primary objective is not always achieved even though the instrument developed successfully performs automatically the chemical control method which it has succeeded. Instead, all that has been accomplished is that process control is now more spectacular. Unfortunately, this is quite a common result, particularly in cases of old plant "streamlining". In these cases, no saving in plant investment is possible because the investment has already been made. Instead, the instrument itself, with associated plant tie-ins, represents new investment.

If reduction in operating costs is to be achieved only through the elimination of the personnel and time involved in performing the chemical analysis, then the chances are that it, too, will be overshadowed by an increase in instrument maintenance time and personnel. It would thus be obvious that development of the automatic analyzer alone is not sufficient to achieve the primary objectives of process cost reduction. The function of the in-line instrument should then be reconsidered. The only way in which the in-line instrument can be made to serve its ultimate purpose is to make it function as a control device primarily designed for the enhancement of the continuous process. It is thus simply a tool by which the continuous process can be optimized and eventually made an automatic process. There is an enormous difference between a process which merely makes use of automatic controls and a process which is truly automatic. The truly automatic process provides the only real potential for process cost reduction.

In summary, then, it may be said that the objective of the development program is really process automation through in-line instruments rather than just providing in-line instruments to replace the chemical analysis.

Before proceeding, it is worthwhile to include at this point a few words on what is meant by an automatic plant and what are its inherent advantages. The simplest definition which satisfies our needs is a plant which eliminates the need for routine human decisions and judgment. This is accomplished by providing the knowledge and basis for judgment to a device which in turn makes the decisions and performs accordingly. The device is made up of many parts as follows: (see Figure 1)

1. The sensing element which determines some criterion of the operation.
2. The decoder which converts the signal to useable information.
3. The comparison and decision unit which compares the information received with information on what is desired and decides what to do.
4. The data storage unit which supplies information on what is desired.
5. The encoder which converts the decision to useable form.
6. The controller which performs the necessary action consistent with the decision.
7. The feed back system which completes the information loop.

The human element has been removed from all but one phase; namely, that of supplying the basic information required for judgment. In order for the device to function, it must have a priori knowledge. However, once given this knowledge, it can be made to perform accordingly on a routine basis.

This leads to the fundamental inherent advantage of the automatic plant; the one advantage from which all others can be derived. Simply, this is that the devices can with proper technological development be made to perform with negligible or reproducible error. This is not possible with a human being. The derived advantages from eliminating the human error are too numerous to mention in detail here and should be obvious. A few will be mentioned later on. In general, they lead to maximum sustained throughput and thereby, minimum plant investment and operating cost.

From this definition alone, it should be obvious that the automation objective is long range, both in terms of CPP and future plants. Furthermore, it is probable that the full value of automation may never be realized at CPP without extensive modifications. However, it must be realized from the outset that the automatic chemical reprocessing plant is the ultimate objective and that research and development should always be headed in this direction. Whether or not CPP is automated can be established on either of two bases; namely, (1) that there is sufficient justification based on the enhancement of CPP as an operating plant or (2) that it is feasible to use CPP as a research facility to demonstrate the principle. As the objective is pursued on either basis, the proposed application of each instrument must be thoroughly reviewed.

In some instances short range applications must be considered. Since these do not in most cases apply to the automation objective they must be justified on the basis of enhancement of CPP operations only. In this case, again critical review is essential perhaps even more critical than for the long range aspects.

With these facts in mind, the general objectives of the program can be outlined as follows:

1. To provide greater operating flexibility and reliability.
2. To provide more stringent safeguards relative to criticality, product loss, radiation hazard, and other safety considerations.
3. To provide product quality control.
4. To provide potential for simplified process or plant design and thus for reduced investment and operating costs.
5. To make advanced technology feasible.
6. Ultimately to provide process automation.

The term long range should not be misconstrued. It is not intended to mean that these problems should be held in abeyance until short range investigations are complete but rather that the accomplishment of the objectives should not be expected for some time to come. It should be obvious though that postponement of the starting date merely postpones the completion date. Sufficient background is available on most of these projects to start immediately.

III. Survey of Instrument Problems

The accomplishment of objectives 1-3 should result in direct benefit to CPP on a short or medium range basis. The development of instruments which satisfy these objectives is a primary obligation and will receive top priority. Certain general problems are immediately obvious. These are:

1. Input measurement for SS Accountability.
2. Product monitoring of raffinates.
3. Monitoring of vessel contents for fissionable material where criticality is a potential hazard.
4. Specific fission product monitoring.
5. Monitoring of feed streams for certain key components which represent process driving forces.
6. Monitoring of by-product streams (e.g. off-gas) for potential safety hazards.

The remainder of the previously outlined objectives can be thought of as longer range problems which may or may not have direct application at CPP. Problems are more difficult to define in this category since they must be closely allied to the overall process development program. However, it is certain that more fundamental knowledge of the process and associated unit operations will be required before any significant advances can be made.

Many additional factors must be considered before specific lines of endeavor can be outlined. These factors and some of the necessary background information are presented in the ensuing paragraphs.

A. Instruments

The leader in the field of in-line instrumentation at AEC installations appears definitely to be HAPO, at least at present. After surveying their program and philosophies both in the literature and by personal communication with the leaders of their program, it has become obvious that their program has been divided into two phases. The first phase had as a basis the demonstration of the feasibility of using in-line instrumentation in a radioactive processing plant. This basis led to the adoption of certain philosophies concerning the design of instruments; that is, the features of simplicity, reliability, ruggedness and ease of maintenance or replacement were stressed with the result that in many cases accuracy and functional features were sacrificed. It was also doubtful in many cases whether the payoff would be significant. Instruments developed at HAPO during this program phase therefore have inherent disadvantages. In general, the accuracy obtained is seldom better than ± 10 percent. Also, cyclic sampling is used through automatic programming such that anywhere from 15 to 55 minutes dead time exists between stream analyses. Thus, the instruments are strictly monitors which have reduced the dead time between stream analyses from 4-6 hours (as performed by the control laboratory) to 15-55 minutes. As will be shown in a later discussion, such instruments are unsuitable for process control in most cases.

The phase II program (currently being initiated) will have a two-fold objective; namely, to develop instruments suitable for control rather than just monitoring and to study further process variables and their control. Because of the aforementioned disadvantages, the monitor-type instrument must be improved upon considerably with stress being placed on greater accuracy and truly continuous analysis.

From the experience gained at HAP0, it seems inevitable that, if the automation objective is to be achieved, the continuous control type instruments must be developed. Since automation is the primary long range objective of our in-line instrument development program, it seems advisable that the simple, inaccurate monitor type of instrument be bypassed and instead an instrument suitable for control should be stressed immediately. This is the approach which has been adopted except in obvious cases where the monitor type instrument may benefit CPP operations.

The accuracy and response time required for instruments of this type can be more precisely defined only by further process studies. The need for the continuous instrument can be demonstrated by a simple example as follows:

If a process variable is to be measured as a function of time, some finite range of values can be expected: i.e., a minimal and maximal value will exist between which the value of the variable will always be found. The variable may have an infinite number of values in this range. From the theoretical standpoint, then, an infinite number of measurements must be made to define the variable at all times. From a practical standpoint, this is not the case because the measuring device cannot have infinite resolution. Since the resolution is finite, there is some minimum change in the value of the variable which can be detected. It follows, therefore, that there are a finite number of discrete increments in the range of values of the variable to be detected depending upon the resolution of the instrument. This number can be expressed as,

$$n = \frac{(\text{range of the variable})}{(\text{min. increment detectable})} + 1$$

For example, if a uranium concentration were to be detected which varied in the range from, say, 1-3 gms per liter and the resolution of the instrument were 0.1 grams per liter, then the maximum number of discrete readings in this range with this instrument is,

$$n = \frac{(3-1)}{0.1} + 1 = 21$$

This says that, as far as the instrument is concerned, it can see only 21 possible values of the variable in this range. This resolution would correspond to an accuracy of 2.5%. If it is assumed that all values of the variable have equal probability, then all discrete values of the variable which can be detected, n , may occur in the time required to traverse the full range. If for the example chosen, a concentration change of 2 gm/liter, the full range of the instrument could be expected to occur in one minute, a sampling rate of about once every 3 seconds would be required. This is obtained by simply dividing the time of

traverse by n. In summary, then, if it were desired to measure a change in uranium concentration of 2 gms/liter/minute in the range of 1-3 gms/liter with an accuracy of 2.5 percent, an instrument having a resolution of 0.1 gms/liter and a maximum full range response time of 1 minute would be required. Also, the stream would have to be sampled every 3 seconds.

This review shows that in order to determine the specifications for the instrument to be developed, it is necessary to have the following process information:

1. Maximum range of the variable to be detected.
2. The accuracy required of the measurement (this may depend on flowsheet criteria).
3. The maximum rate of change of the variable to be detected.
4. The probability of the variable having specific values within the range.

Once this information is available, instrument resolution and response time plus sampling rate and frequency can be determined. Again, referring to the uranium detector example for the values chosen, a sampling rate of once every 3 seconds was required. For the most part, the chosen values are realistic. The rate of change of concentration might be questioned but with the status of knowledge being what it is now, a definite answer is not possible at this time. The only conclusions which can be reached are (1) that it is desirable to determine what rates of change of variables are to be dealt with and (2) that the only feasible method of determining these values is through continuous instrumentation. Thus, the first instrument to be developed must have maximum resolution, fast response, and must approach continuous measurement, at least as far as it is economically feasible, in order to evaluate future instrumentation needs. The information gained from the first instrument should make it possible to more precisely evaluate the design of those to follow. This approach will be taken on the basis that the additional cost, if any, incurred over that for a simpler design, can be written off as a research cost.

The specification of instrument requirements is further complexed by the uncertainties in both fuel composition and processing load associated with CPP or any other plant of this type. To be consistent with the stated objectives, it is necessary that the process with which the instruments are associated have certain properties. These properties are in turn defined on the basis of the fuels being processed. Fuel classifications have been defined elsewhere in the Chemical Development Program and it will suffice to say that the classifications are based on firmness of composition, expected steady state load and duration of load. These bases then define the process requirements and the extent of research and development which can be justified.

In summary, a complete survey of future instrument needs is not possible at this time and the present agenda should include only those problems which are consistent with (1) the program objectives, (2) the present knowledge of

the trends in fuel composition and processing load, (3) the present knowledge of the associated processes and processing needs, (4) the probability of new processing technology and (5) the scope of the overall research program.

As a guide to evaluating specific problems, a system of classification has been devised which uses as a basis the instrument application and which is consistent with the objectives and philosophies previously outlined. Three classes can be listed as follows:

CLASSIFICATION OF INSTRUMENT APPLICATIONS

Class I Application

The application of high precision instruments suitable for continuous measurement and control of a Class I fuel process for the purpose of improved control or as research tools for process evaluation. Such instruments do not solely benefit CPP operations and for the most part involve new approaches, require the greatest development effort and must be coordinated with control studies to accomplish the primary objective of automation.

Class II Application

The direct application of instruments to CPP for the improvement of operating techniques associated with any fuel process. Instruments of this type are not necessarily suitable for continuous measurement and control and are not high precision instruments. Most of these have undergone preliminary development at other sites and therefore involve application and improvement study only.

Class III Application

Only general application is of interest in this category. The instruments are developed primarily for the purpose of advancement in instrument technology by the demonstration of new or improved methods of measurement and/or control.

B. Automation

In general, the major problem in making any process automatic is one of converting art to science. This is just another way of saying that the human decisions required for control of the process must be based on reproducible scientific fact rather than "spur of the moment" technical estimates. The key to the conversion, of course, is knowledge. Having the knowledge, the tools required for application can be developed.

As an aid in understanding what specific information is needed, why it is needed, and how it can be applied after it is obtained, it is convenient to take as a typical case the operation of a single solvent extraction column and analyze the automation problem relative to it. See Figure 2.

To begin with, the primary function of the unit for our purpose is to transfer uranium from an aqueous to an organic phase with a maximum separation from fission products. Therefore, the product of the operation can be defined as an organic solution of uranium containing a minimum of fission products. This, however, must be qualified by at least the additional final result of maximum recovery, although many other qualifications can be imposed. Thus, the ultimate criteria of the unit operation can be specified as maximum recovery and minimum contamination (i.e. maximum product quality). These being the criteria of the process, methods of insuring the function of the operation can be examined relative to them. In general, the control of the function can be either open loop or closed loop. Most methods of control presently employed at CPP are of the open loop type.

The open loop method of control attempts to insure fulfillment of the process criteria by maintaining controlled settings of the variables associated with the operation. For the solvent extraction operation these variables are stream flow rate, column interface level, pulse amplitude and frequency, and, indirectly, entering stream composition. Stream flow rates and pulse conditions are presumably maintained by setting stroke amplitude and frequency on piston pumps. Cold stream compositions such as scrub and solvent are maintained by making up large batches under laboratory control suitable for many hours and even days of feeding. In the case of hot feed stream composition, control is effected through control of the variables associated with the preceding operation since this stream represents the product of the prior operation. Ultimately the products of the operation are sampled to determine the success of the operation but the time lag involved is so large that the application of corrective measures to prevent accumulation of off standard products is impossible. Thus, the only recourse for correction is through recycle of the accumulated product. Although the desired product quality is eventually achieved, the inefficiency of this type of control makes it extremely unattractive. In addition to this major disadvantage, it is a well known fact that in open loop systems any errors in control result in a direct proportional effect on the output of the system. Processwise, this means that either considerable versatility must be inherent in the operation to accommodate these errors without malfunction or if this is not possible, then extremely accurate controls must be used. Either solution to the problem is costly and troublesome in most cases.

The closed loop method of control, on the other hand, does not depend on maintenance of control settings of the variables but rather samples the process criteria continuously and applies continuously corrective action consistent with adherence to the criteria. In essence, the controls "float"

about the desired settings always assuring proper values of the criteria; namely, maximum recovery and minimum contamination. Contrary to the open loop system, the control elements in the closed loop system are active and errors inherent in them are continuously corrected as well.

If the operation is to be made automatic; that is, self-correcting, then the closed loop system of control is essential. Since operational control at CPP is open loop, the first problem of automation becomes self evident; namely, the conversion to closed loop control.

Returning to the solvent extraction example, the question is raised as to how closed loop control is effected. Since the basic criteria for establishing corrective action is product contamination and maximum recovery, then it is necessary to have means available for evaluating these criteria. This establishes the first requirement, chemical instrumentation to detect product in waste streams which constitutes product loss and instruments to detect contaminants in the product stream, namely, fission product radioactivity. Assuming instruments of this type are available as a result of the instrument development program, the next requirement is to determine what corrective action should be taken if required. In other words, if the product quality and recovery at any instant are at the desired values no action is necessary; if they are not, then some adjustment of the operating variables should be made to force them back to the desired values. The most logical procedure for accomplishing the return is to first determine each of the instantaneous values of the operating variables of input stream flow rate, column interface level, and pulse conditions. Secondly, these values must be compared with a standard and evaluated individually and relative to each other in a central control unit. If an off standard condition in one of the variables is detected and can be directly corrected, the problem is straight forward; if it cannot be directly corrected, then compensating action is required. The latter could involve a simple change in one of the other operating variables or a complex readjustment of all of the operating variables. As an example, let us assume that the waste monitor shows excessive uranium losses. All values of operating variables are scanned and it is found that flow rates and interface are at the desired value but the pulse to the column is not optimum because of some partial failure of the transmission system. The first attempt at correction would be to adjust the pulse pump to compensate for the loss in transmission. If the correction is not successful, then other means of compensation must be sought such as increasing the solvent flow rate. This in turn would require adjustment of the scrub flow and perhaps ultimately to reduce the feed rate to the column, etc.

If, on the other hand, when the scan is made all operating variables are found to be standard, the third step in the procedure would be introduced; namely, to determine the instantaneous values of the input stream composition. Here again changes in operating variables can be made to compensate for significant changes in input stream compositions. In the previous example, the excessive uranium loss may have been due to loss of salting strength caused by improper scrub stream composition. If closed loop control of this composition were available, a new scrub composition could be made to correct the off standard condition. If the excessive uranium loss was found to be caused simply by an increase in uranium content of the feed, then any number of corrective actions are possible.

Considerable benefit can be derived from this step in that potentially an optimum set of operating conditions can be used for each feed composition. However, to derive this benefit, a knowledge of feed stream composition is essential. In summary, the control of the extraction operation is now closed loop in that the output of the process, (product recovery and decontamination) is continuously sampled, compared with a desired value (in the central control unit) and changes in the process control system (the operating variables) made in compliance with the error signal (the deviation from the desired values). In some cases, the desired result depends on what is being fed to the process. In any case this information must be incorporated in the control device.

It should be noted also in this example that in addition to assuming the availability of analytical instruments, it was also assumed that (1) when corrective action was required the knowledge of what action to take was available, (2) that after the action was taken the response of system to the action was known so that the action could be applied at a rate consistent with change detection and (3) that the desired settings of the operating variables could also be maintained, or in other words, that closed loop systems of control were available for each operating variable. Just as the instruments for measuring stream compositions are lacking, so are the information and tools for conversion to complete closed loop control.

It is interesting to point out here that a certain amount of closed loop control is effected manually at CPP. The fallacy in developing this further is that a high degree of technical training is required to make the decisions necessary for effecting control. That this is true can easily be concluded from the fact that years of effort by technical personnel are required to develop the processes. Is it then logical to expect that technically untrained operating personnel should be able to control such processes? The answer is no and as a matter of fact, what happens is that a large technical staff is actually used for this purpose in the plant. No process can operate economically under this condition. It is possible, however, to provide all of the technical knowledge fairly easily to a machine, the central control unit in our example, for routine application with maximum dependability.

To satisfy the need for more fundamental knowledge, the following general lines of investigation can be outlined.

1. Continuous monitoring of plant streams solely for the purpose of accumulating key operating data for further analysis of process variables.
2. Development of closed loop systems for control of operating variables (e.g. flow rates, pulse conditions, scrub and solvent compositions).
3. Advancement of existing unit operations theory and development of new theory.

4. Investigation of physical chemical aspects of the systems involved and the kinetics of the processes to support the unit operations studies.
5. Transient and frequency response analysis of flow systems, in particular the cascade flow system.
6. Development of analogue simulation methods for process control studies.
7. Advancement of background in both the mathematical and practical phases of automatic control.
8. Survey of data processing and handling equipment.

Relative to these, instruments may be developed to facilitate the investigation or the investigation will demonstrate both the need for the instrument in the process and the type of instrument which must be developed. The results of such investigations can conceivably benefit CPP directly at any time but are not designed solely for this purpose. In some cases, the sole benefit may be only to provide engineering tools by which improved evaluations of existing plants and processes can be made or by which more advanced designs for new plants and processes can be made with a minimum of uncertainty and/or prototype operation. It should be pointed out, again, that automation of CPP is an objective of these studies but the incentive for the general study is not CPP alone.

After development of the required knowledge of the process variables as well as the tools to measure and control these variables, attention would then be turned toward integration into the automatic plant concept. Studies associated with the overall problem of plant automation and process optimization through in-line instrumentation need not be allied to any particular fuel or process and therefore, are dependent only upon manpower, incentive, and status of knowledge.

IV. Lines of Endeavor

A. Instruments

In terms of our immediate obligations to CPP processing (which have been defined as first priorities), Table I lists instruments applicable to aluminum fuel processing which are justifiable on the basis of group one objectives and Class I applications. Any number or all of these instruments are worthy of immediate development. Processes other than the aluminum fuel process (e.g. STR, SIR) are not firmly enough established for Class I considerations but Class II instruments will be developed when the need is established.

In the majority of cases, the streams being analyzed are multicomponent and fairly complex techniques must be used. Three methods of general attack are worthy of note as follows:

The first is to measure combinations of properties such as density, viscosity, refractive index, etc., and determine a suitable set of simultaneous equations from which the concentrations of the major components can be determined by analogue computation. This method has two distinct disadvantages; namely, that if there are n major components, n properties must be measured and that the properties are all different thereby requiring multiple instruments.

The second method is to measure properties such as conductivity, electrolytic potentials, color, etc., using some condition under which the measurements are made as a parameter from which the necessary simultaneous equations can be derived. This method is best illustrated by a few specific examples. In the measurements of electrolytic potential the type of electrode can be varied; in colorimetry, the wave length and in conductivity, the frequency. If the response of each component of the system varies independently as the parameter is varied, then again as many simultaneous equations can be obtained as there are components and the unknown concentrations can be determined by analogue computation. This method although similar to the first, has the potential advantage of a single multichannel instrument rather than multiple single channel units.

The third method involves continuous separation of the component of interest from the bulk of the stream followed by a suitable measurement of a single property. This method has the obvious disadvantage of introducing inherent time lags into the measuring system but has particular advantage where the component of interest is a minor constituent such as product in raffinate streams. To facilitate this method miniature, reliable, automatic separations equipment must be developed as well as the chemical separations flowsheet.

Methods such as polarography, coulometry, potentiometric and amperometric titrations, etc., are inherently cyclic methods and are therefore unattractive as such. Conversion to continuous methods would require either the development of suitable continuous flow metering equipment or the application of pulsed techniques.

Table I
PROPOSED INSTRUMENTATION FOR THE ALUMINUM FUEL PROCESS

<u>Stream</u>	<u>Components to be Measured</u>	<u>Purpose</u>	<u>Tentative Method</u>	<u>Sampler</u>
1 AF and/or Dissolver Effluent	Uranium Total Activity Acid Aluminum Zr, Ru Activity	Accountability Decontamination Control Flowsheet Criterion Flowsheet Criterion Decontamination Control	Colorimeter Gamma Monitor Conductivity Electrode Potential Gamma Spectrometry	G-604 Dissolver G-654 Effluent G-605 Dissolver G-655 Run Tank G-606 1A Feed Tank
1 BU	Total Activity Zr, Ru Activity (Uranium) (Acid)	1A-1B Column Decontamination Factor Process Information	Gamma Monitor Gamma Spectrometry Colorimeter Conductivity	H-602 1B Sample Pot
1 AW	Uranium	Accountability and Process Criterion	Desired	G-613 1AW Sample Pot
1 AX	(%) TBP (Acid) (Specific FP) (Degradation Products)	Recycle Solvent Quality	Oscillometer Desired Gamma Spectrometry Desired	H-618 Solvent Run Tank
1 CW	Uranium	Criticality Safeguard	Colorimeter (Gamma Photometer)	H-603 1C Column
1 DU	Total Activity Zr, Ru Activity Acid Suspended Organic	1st Cycle Overall D. F. Check Point for acid to second cycle Solvent carry-over to 2nd cycle	Gamma Monitor Gamma Spectrometry Conductivity Nephelometry	H-607 LD Sample Pot
1 DW	% TBP	Determine Diluent Life	Oscillometry	H-608 Scrub Collection Tank
2 AW	Uranium	Criticality Control for Raffinate Recycle	Colorimeter	Q-605 2AW Sample Pot

Continued

Table I (Con't.)

PROPOSED INSTRUMENTATION FOR THE ALUMINUM FUEL PROCESS

<u>Stream</u>	<u>Components to be Measured</u>	<u>Purpose</u>	<u>Tentative Method</u>	<u>Sampler</u>
2 BP	Total Activity Zr, Ru Activity	2nd Cycle Overall D.F.	Gamma Monitor Gamma Spectrometry	Q-608 2BP Sample Pot
3 AW	Uranium	Criticality Control for Raffinate Recycle	Colorimeter	S-605 3AW Sample Pot
3 BP	Total Activity Zr, Ru Activity	Product Quality Control and 3rd Cycle Overall D.F.	Gamma Monitor Gamma Spectrometry	S-608 3BP Sample Pot
Off-Gas	I ₂ from RaLa Residual Activity	Radiation and Contamination Safeguard	Gamma Spectrometry Gamma Monitor	To be added To be added
	Hydrogen	Explosion Safeguard	Desired	To be added
PEW	Uranium	Product Loss Safeguard	Colorimeter	To be added prior to WG-100 and 101 WG-100 and 101

Parentheses indicate research constitutes major incentive

It is possible that some components of interest may have specific properties by which they alone can be identified in the presence of many others, but these cases must be considered as the rare exception rather than the general rule. Such an approach does not lend itself to methodical attack at least at present. Several methods involving the interaction of electromagnetic radiation in the RF range with orbital electrons have been suggested but these appear to be beyond the scope of immediate application. Likewise, the search for identifying chemical complexes or organic compounds is beyond the scope of immediate application.

Maximum assistance in developing new methods, expanding existing analytical data and developing basic theory of photometry, coulometry, etc., should be obtained from analytical research groups.

The importance of sampling frequency relative to the successful performance of any instrument has been discussed. The quality of the sample must also be considered; that is, whether the sample is representative of the stream being analyzed, the effects of dissolved gas, temperature effects, etc. Perhaps the optimum location for the sample cell and/or sensing element is in the vessel or stream to be sampled. In the ultimate plant design this may be practical but for CPP installations it would involve locating the element in the processing cells in most cases. Because of the high radiation level in the cells, the element would be too obscure for adjustment and maintenance and radiation damage to the element would occur at an accelerated rate. For these reasons, it is desirable to locate the instrument sampling element in the sample gallery and provide the necessary circulation. The design of the flow sampling apparatus must be studied to insure the necessary sampling rate, minimum system time lags, sample quality, ease of maintenance, and integration with existing facilities.

As previously mentioned, many of the instruments will involve the use of analogue computing devices to solve sets of simultaneous equations. This problem as well as that of proper signal amplification, etc., will necessitate the study of specific electronic components and servo loops.

B. Automation

At present, only a few specific lines of endeavor can be listed as suitable for immediate study. The general approach to the problem is to determine the time constants associated with the various unit operations by development of unit operations theory, experimental investigations using frequency response and transient response techniques, and analogue simulation. Some of this work can be done concurrently with the instrument development program, particularly that associated with unit operations theory. Details of such studies can be defined only as background knowledge and tools are developed. It should be obvious that some of these problems have sufficiently general application that they might be considered elsewhere in the chemical development program. However, it is convenient to think of them in terms of the automation objective and thus have a well integrated program with a single goal. It is also true that such studies are a prerequisite of automation and can be justified on this basis alone.

V. Program Execution

A. Problem Flowsheet

The aforementioned classifications of instrument applications also serve to establish the scope of endeavor justifiable for each instrument. The steps involved in developing any instrument are presented on the problem flowsheet in Figure 3. Also included in the flowsheet are the basic steps in the study of control problems and the preliminary steps in the automation study.

The first step in the procedure prior to the development of the instrument is the survey of the problem. This involves specification of the instrument classification and detailed requirements. The scope of the problem is also determined and if any existing methods can obviously be applied, these are established. It is assumed that an evaluation of the instrument application has been made prior to this point.

Proceeding, a general survey of existing methods applicable to the problem is made and the most attractive method is established. An outline of the uncertainties is made and alternate methods listed. Sufficient information is available at this stage to plan an experimental program for exploitation of the method in the laboratory. Instrument proposals involving new methods (Class III Application) are also introduced at this point for planned exploitation.

Maximum assistance should be anticipated from analytical research groups in choosing alternates, establishing uncertainties, providing basic knowledge on the methods and limited assistance in exploiting the method. For the most part the method is so closely allied to the instrument design that its exploitation is of necessity a function of the instrument development group. The product of the exploitation is an instrument proposal which then undergoes technical evaluation to determine subsequent procedure.

Coordination of other sources of information is also required. The ultimate application of the instrument together with the proposed design features will dictate the specifications for the associated sampling installation and the preliminary basis for specific control studies. It is necessary that these studies be headed in the required direction at this point. Many of the instruments will involve electronic detecting units and computing devices, which will be defined in the preliminary instrument proposal. The design of these units for the first laboratory model of the instrument will be done by the instrument development group except in obvious cases where the background of the group may be exceeded. It is anticipated that the designs will not be optimum but rather the least complex functional approach that can be taken. The optimization of designs toward maximum reliability is proposed to be a function of a more specialized instrument development section. This being the case, it will be necessary to provide this section with sufficient definition of the problem to facilitate optimization studies concurrent with development of the laboratory model. Such problems are defined at this point.

Additional assistance with mechanical designs will be required from an engineering design group. It is desirable that the efforts of this section be solicited at this stage of development to familiarize design personnel with the problem. However, as in the case of electronic designs, it will be necessary for the basic instrument development group to provide conceptual designs for at least the first model.

All classes of instruments proceed through the exploitation and evaluation steps. The main function of the evaluation is to establish subsequent procedures on the basis of existing knowledge.

In general, the procedure will follow either of two paths. Class I and Class III applications involve new technology and therefore will require development of the instrument in the laboratory first. Class II applications, on the other hand, represent, for the most part, known technology and first plant models can be designed without preliminary laboratory study in most cases.

To facilitate plant installation, it is necessary to develop a sampling system consistent with CPP plant practice. The conceptual design of a general system for instruments at CPP as well as an optimum system for new plants, will be studied concurrent with the initial stages of instrument study. Components for the CPP system will be tested during this initial period for the purpose of establishing a conceptual design for all CPP instrument needs. It is not anticipated that this work will be repeated except where special problems arise. Development of the optimum sampling system will, however, continue. Having established the general concept, major attention is turned toward the detail design of systems for specific instrument problems. These studies are integrated with design studies of the first plant instrument model.

For Class III application; i.e. demonstration of new methods, the plant model will in most cases be a conversion of the lab model for demonstration purposes only and therefore goes directly to the plant. After demonstration tests the instrument will be returned to the lab for re-evaluation and possible reclassification. Plant models for Class I and II applications on the other hand undergo further lab testing, that is, shakedown operation, followed by plant testing. If design modifications are dictated by the plant test, these are made and the instruments are installed in the plant. For Class II applications, these will be final models. For Class I application, these instruments will be used primarily for process evaluation; that is, to establish the basic information needed for control studies and automation.

As previously discussed, the pursuit of the automation objective requires fairly intensive study of the process, its unit operations and its control. As background in unit operations theory, control theory and automation is developed, a general effort will be made to define control problems, survey existing control techniques, determine process phases in need of improvement, and more specifically outline information and data needed. When evaluation shows the need for a specific control problem investigation and establishes that both sufficient background data and

satisfactory instrumentation are available to proceed, two lines of endeavor are put into operation simultaneously. The first is an empirical study consisting of plant or lab experiments and empirical analysis. The second is a theoretical study using both experimental data and analogue simulation. The empirical and theoretical approaches are continuously correlated and the results evaluated. The problem is recycled through the study loop until the desired effects or technology are developed to a point suitable for conversion to practice.

No direct attempt at automation of the complete plant is anticipated during the period for which the problem flowsheet should be valid; namely, through fiscal year 1960.

VI. General Comments

Much time has been spent in surveying the status of the chemical processing industries and in particular the nuclear fuel reprocessing industry relative to in-line instrumentation and automation. Significant advances in the field have been made to the extent that there now appears to be little if any doubt remaining as to the tremendous benefit to be gained from automation. In view of the urgent need for cheaper nuclear fuel reprocessing, it is difficult to see how the potential advantages of automation can be treated lightly. It is true that the problems associated with automation of radioactive plants are more difficult but the incentive is greater because of the inherent remoteness of the equipment and potential hazard to operating personnel.

Because of the exploratory nature of much of the work to be included in this program, it is possible that some lines of endeavor will prove to be unfruitful. This is a calculated risk involved in any research involving so many unknowns. However, fear of the unknown or complexity of the problems are not valid reasons for abandoning research.

Figure 1

Control Information Loop

Figure 2

SCHEMATIC OF EXTRACTION COLUMN CONTROL

Figure 3
INSTRUMENTATION AND
CONTROL PROBLEM FLOWSHEET