SECRET COVER SHEET

UNCLASSIFIED

X-426 (Rev. 8-55)

OAK RIDGE NATIONAL LABORATORY
Operated By
UNION CARBIDE NUCLEAR COMPANY

UCC

POST OFFICE BOX P
OAK RIDGE, TENNESSEE

ORNL
CENTRAL FILES NUMBER
56-4-104

"External Transmittal Authorized"

Cy 35A

DATE: April 11, 1956

SUBJECT: Proposed Inline Instrumentation Program at Oak Ridge National Laboratory for FY 57

TO: F. L. Culler

FROM: M. J. Kelly, J. W. Landry, R. W. Stelzner

DISCLAIMER

CLASSIFICATION CANCELLED
DATE 11/14/61
For The Atomic Energy Commission
H. R. Carroll
Chief, Declassification Branch

CLASSIFICATION CHANGED TO
CONFIDENTIAL
DATE FEB 15 1957
H. R. Carroll
Chief, Declassification Branch

CLASSIFICATION CANCELLED
DATE FEB 15 1957
For The Atomic Energy Commission
H. R. Carroll
Chief, Declassification Branch

CONTENTS

TABLES

1.0 INTRODUCTION AND SUMMARY

The proposed inline instrumentation program at Oak Ridge National Laboratory for FY 57 follows the basic two year program proposal for FY 56-57 as reported in ORNL Memo CF-55-12-145.

The proposed program for FY 57 calls for a budget of $100,000 under AEC account 2724, and comprises 36 man-months of ORNL effort. The proposed program for FY 57 will be reviewed and adjusted pending future developments in the field of inline instrumentation.

The basic proposal was established at the 30 September 1955 meeting between representatives of ORNL and of Savannah River Laboratory. This proposed program consisted of five points; the ORNL inline instrumentation program in FY 56 has proceeded along these five points:

1. <u>Review of Process Control at ORNL</u>

   Make a brief over-all study of process control so as to integrate into the SRL program the experience ORNL has gained in continuous operation of their pilot plants.

2. <u>Development of Sampling Facilities</u>

   Study sampling facilities so as to provide means for introducing a continuous sample into any instrument. Include provisions for handling the contamination and dirt problems in instrument cells with and without optical windows.

3. <u>Development of a Specific Gravity-Conductivity Instrument</u>

   Develop plant type methods and/or instruments for the practical application of the SRL Analytical Chemistry Division's findings on the dual use of specific gravity and conductivity to measure uranium and acid in high concentrations.

4. Testing of Inline Instruments in ORNL Hot Pilot Plants

Test SRL instrument candidates in "hot" facilities at the ORNL pilot plants. The first items which will be ready for testing are the Savannah River Analytical Chemistry Division's close-coupled alpha and gamma monitor prototypes.

5. Laboratory Study of Basic Parameters

Select one basic analytical parameter for study from among viscosity, refractive index, electro-potentiometric methods, or others to be defined. This study will be chosen so as not to conflict with the Savannah River Analytical Chemistry Division's program.

The review of process control at ORNL has been essentially completed. Future techniques in process control will be reported in FY 57 as they develop. The development of sampling facilities is scheduled for completion 30 June 1956. The development of a specific gravity-conductivity instrument and the laboratory study of basic parameters both are partially completed and the proposed completion date is 30 June 1957. The testing of inline instruments in ORNL hot pilot plants can commence when the development of certain inline instruments reaches the hot testing stage.

Thirty-six man-months and a total of $100,000 will be applied on the ORNL inline instrumentation program in FY 57. The proposed program will require the full-time services of M. J. Kelly of the Instrumentation and Controls Division, J. W. Landry of the Chemical Technology Division, and R. W. Stelzner of the Analytical Chemistry Division. A breakdown of manpower and costs is given in Table 1.

2.0 OVER-ALL PROGRAM OBJECTIVES

The primary objective of the ORNL inline instrumentation program is to integrate certain development and testing facilities of ORNL into the SRL inline instrumentation program and co-operatively secure advanced methods of process control which will be mutually valuable to the Savannah River separations plants and the ORNL pilot plants. The ORNL group will serve also as a liaison group for the exchange of process techniques applicable to continuous processing.

3.0 PROPOSED PROGRAM FOR FY 57

3.1 Review of Process Control at ORNL

It is proposed that 0.5 man-month and a total of $1000 be applied in FY 57 to report developments in process control and continuous processing techniques at the ORNL pilot plants during FY 57. New instrumentation exclusive of inline instrumentation would be included.

"A Review of Process Control at Oak Ridge National Laboratory's Radiochemical Pilot Plants" will report the techniques and specifies the equipment which are employed for the following items of process control in the Metal Recovery (Purex) Plant and the Thorex Pilot Plant:

a. Liquid level
b. Density
c. Temperature
d. Pressure
e. Flow rate
f. Radiation level
g. Continuous evaporator operation
h. Plutonium resin elution

Future techniques in the above and other items of process control will be reported in FY 57 as they develop. An example would be reports on the results of experiments presently in progress on the use of ultrasonics for the measurement and control of liquid level and interface level. New techniques with respect to chemistry and equipment that will be useful in continuous processing will be indicated in the reports. Examples of the latter, which are being evaluated in ORNL pilot plants, are liquid-solid separators (hydroclones and canned-rotor centrifuges), continuous dissolvers, slug chargers, volumetric airlifts, and extraction contactors.

3.2 Development of Sampling Facilities

This program proposal does not provide for the development of sampling facilities after 30 June 1956. In the event that the development of sampling facilities is extended into FY 57 it will be included with the testing of inline instruments in ORNL hot pilot plants.

Development of Sampling Facilities consists of two phases: (a) the development of instrument cells for contacting instrument sensing elements with continuous samples of process solutions, and (b) the development of samplers for delivering the process solutions to the instrument cells. Development of sampling facilities was reviewed in the 31 January 1956 meeting between representatives of SRL and of ORNL. At this meeting it was decided that future ORNL development of sampling facilities will be applied primarily on the development of instrument cells.

3.21 Instrument Cells

In addition to its containing the sensing elements and the contacting solution the instrument cells that must be developed also embody in particular instances the following provisions:

(a) The cell will retain a minor background of radioactivity from radioactive process streams which are transmitted through the cell. The background will be minor with respect to radiation readings which may be made on the transmitted stream.

(b) The cell will provide direct viewing of the process stream where desirable.

(c) The cell will not accumulate dirt.

(d) The cell will present a constant volume of process stream to the sensing element where desirable.

The development of instrument cells is progressing at a number of sites but cells having certain of the above provisions remain to be developed. Three months remain in FY 56 to develop these cells. The development of instrument cells at ORNL may be extended into the FY 57 program until the instrument cells are developed with the necessary provisions. Until the time when inline instruments are available for hot testing, the manpower and budget which presently are allocated to hot testing in FY 57 can be applied for continuing the development of sampling facilities in FY 57.

Several possibilities for instrument cells are being considered. Materials for construction, geometries, and surface finishes for instrument cells are being examined for their resistance to radioactive contamination. The flow characteristics of the cell are being arranged to minimize the dirt problem. Electrolytic methods are being evaluated for immunization of cells to radioactive contamination. A falling stream type of instrument cell is being investigated for minimization of background activity. The use of high-density glass windows in a falling stream type of cell is being evaluated for direct viewing of the process stream. In addition to the falling stream type of instrument cell, the following specific types of instrument cells at ORNL have been evaluated, are being evaluated, and are scheduled to be evaluated:

(a) Instrument cells with the following surfaces in contact with the sample stream:

1. Stainless steel, type 304 L, machined smooth and electro-polished

2. Gold, pinhole-free, 3-mil plate of a dense, high finish
3. Chromium, bright, 3-mil plate
4. Teflon, fluorothene, and polythene liners
5. Titanium
6. Tantalum liner
7. Aluminum, electrolytic (99.95% pure aluminum)

(b) Instrument cells of the electrolytic type:

1. An anodic inhibited cell with an external electrode. - Insulating connectors would be used and the sampler piping would serve as the external electrode. A simple current regulator device would maintain current at about 2 μamps and thereby provide a counter emf which would inhibit the plating of certain contaminants in the cell.

2. A cell with an internal electrode. - A reversing potential between the cell and the electrode would be selected to maintain good polishing efficiency but poor plating efficiency so that contamination would be kept in solution.

3. A cell with a gassing needle electrode. - A current density of 10 milliamps/sq cm would result in electropolishing action on the cell but would correspond to a current density of 1-5 amps/sq cm on the needle. The high current density at the needle would result in gas blasting which would keep the needle clean from plating effects.

4. A cell with a fritted surface. - The cell surface would be separated from direct contact with the sample stream by a film of air pressured through the cell surface.

In addition to the above investigations, specially requested tests for instrument cells would continue to be conducted in specific process streams in the hot pilot plants.

3.22 Sampler Devices

Additional sampler installations will be required in the ORNL hot pilot plants for testing inline instruments. Approximately twelve samplers for inline instrumentation will be installed in the expansion of the Metal Recovery Plant. The additional installations will be made according to the best sampler design available. Data are being collected from the development and evaluation of samplers at ORNL and other sites. Recommendations for inline instrument samplers are being kept current for new installations. The Thorex Pilot Plant is preparing a movie of the Thorex type of sampler with which one operator draws up to 30 samples simultaneously without radiation exposure.

The present samplers at the Savannah River Plant sample adequately. Future modifications of the SRP sampler design to facilitate inline instrumentation would also include the following items where possible:

(a) Minimization of irradiation to operating personnel.

(b) A means to immediately check for the presence of a sample in the sample container.

(c) A means to observe the sample appearance and the recirculation rate.

(d) Freedom from jet clogging and from the dilution of samples which sometimes results after the jet steaming operation.

Some of the sampler equipment at SRP in the gallery areas is being modified by a plant engineer group with the objective of increasing freedom of the samplers from maintenance. Outside of the gallery area, changes in the existing SRP samplers are restricted to cases of necessity because of two conditions: (a) much of the sampler equipment is embedded in concrete or is situated in operating areas of limited access; and (b) the deadline is at hand for including sampler modifications in the plant modification plans.

The ORNL inline instrumentation group is building an experimental sampler which duplicates the SRP sampler with respect to hydraulics. The experimental sampler is to be made of glass and it duplicates inside diameters, lengths, slopes, number and kind of turns, jet and airlift arrangement, and instrument cell connections. The experimental sampler is required for conjunctive testing of inline instruments and SRP samplers. Studies are planned, for the experimental sampler, to optimize operation of the SRP sampler for use with inline instrumentation. Improvements for the SRP sampler will be recommended if they can be effected by very simple changes in the gallery equipment. Examples are:

(a) Fixing the air flow rate to the airlift, and

(b) Removing the jet from the liquid circuit and converting the sampler to 100% airlift operation by adding a separator at the jet intake (as in the ORNL sampler).

3.3 Development of a Specific Gravity-Conductivity Instrument

It is proposed that 6.5 man-months and a total of $9000 be applied in FY 57 to complete the development of the specific gravity-conductivity instrument. This includes the proving of a prototype instrument in the Metal Recovery Plant.

The specific gravity-conductivity instrument is being developed to measure high concurrent concentrations of uranyl nitrate and nitric acid. A typical application for which this instrument is being developed is to follow uranium and acid concentrations in feed solution during the dissolving and feed adjustment cycles of the Purex process. The conditions of wide excursions in temperature, of turbulence, high levels of radiation, inaccessability, and mechanical shock which exist in the dissolving and feed adjustment vessels are adverse to direct immersion of instruments in these vessels. A side-stream specific gravity-conductivity instrument therefore is being developed. The specific gravity and conductivity basic components were developed in FY 56 for a side-stream specific gravity-conductivity instrument. Temperature controlling and compensating components remain to be developed.

3.31 <u>Measurement of Specific Gravity</u>

A side-stream, purge type, high resolution densimeter has been developed for use in the specific gravity-conductivity instrument. This densimeter includes a Taylor 339 RFS differential pressure transmitter. With a sample flow rate of 200 ml/min through the 600 ml densimeter cell a precision of $\pm$ 0.002 gm/ml can be obtained. The densimeter has been found to be suitable for general plant application although with some loss of precision.

Automatic conversion of density to specific gravity by a temperature accessory on the densimeter remains to be investigated in FY 57. The FY 57 objective would be the development of an instrument to record specific gravity at a selected temperature with a precision of $\pm$ 0.002 sp g units independent of changes in the actual sample temperature.

Literature checks and in some cases experimental tests will be made on other density measuring methods. These are:

(a) Cartesian diver devices.- An example is the commercially available "Densitrol".

(b) Sonic and ultrasonic devices.- An example is the commercially available "Electro-Circuit" device.

(c) A viscous dampening device.- An example is the commercially available "Ultra-Viscoson" which measures the density-viscosity product of a solution.

(d) A volumetric-gravimetric device.- An example is an overflow pot linked with a strain gauge.

(e) A range-extending device for the high resolution densimeter.- An example would be an auxiliary density receiver which would reset the suppression to give multiple ranges over any necessary latitude of density measurement.

3.32 Measurement of Conductivity

A side-stream conductivity instrument has been developed for use in the specific gravity-conductivity instrument. It consists of a controlled area type of conductivity cell connected in the bridge circuit of a Foxboro series 9000 recorder. The conductivity cell has been operated continuously for ten weeks. It appears to have excellent stability and it is very insensitive to electrode effects. With a sample flow rate of 200 ml/min through the cell, the apparent over-all precision of the instrument over the necessary concentration range is $\pm$ 0.5%. Automatic temperature compensation for the conductivity measurement remains to be developed in FY 57.

3.33 Control and Compensation of Temperature

It is proposed that 1.0 man-month and a total of $3500 be applied in FY 57 for completing the development of the temperature component of the specific gravity-conductivity instrument. This would include devices for temperature control and automatic temperature compensation.

Sample temperature control to $\pm$ 5°C will be necessary. Temperature compensation within these limits can be accomplished by suitable instrumentation. Temperature measurement to $\pm$ 0.1°C can be obtained in this system by resistance thermometry. A resistance thermometer will be used in the specific gravity-conductivity instrument.

The first device that would be tested for controlling the temperature of the sample stream to the specified tolerance, $\pm$ 5°C, would be a simple heat exchanger. This heat exchanger would immediately precede the sensing elements for density, conductivity, and temperature.

An accessory product resolver is being designed to combine the density and temperature readings so as to provide automatic temperature compensation for the densimeter. Other forms of temperature compensation to be evaluated for the densimeter would include dip tube arrangements which utilize thermal expansion principles. The objective would be to develop an instrument which directly measures specific gravity referred to a selected temperature.

Temperature compensation for the conductivity measurement would be accomplished by positioning the temperature compensating potentiometer in the recorder. The potentiometer would be positioned by means of a servo-mechanism actuated by the signal from the resistance thermometer. The use of thermistors in high-level radiation fields, generally speaking, is considered to be unsatisfactory. Temperature compensation by means of thermistors therefore is considered to be unsatisfactory for an instrument that is being designed to measure the conductivity of feed solutions. The temperature coefficient of conductivity for feed solutions must have the necessary degree of constancy over the range of concentrations concerned in order for temperature compensation to be applied. This is being checked.

3.34 Specific Gravity-Conductivity Instrument

It is proposed that 4.5 man-months and a total of $5500 be applied in FY 57 on the design, fabrication, and proving of a prototype specific gravity-conductivity instrument.

An instrument which is composed of the previously mentioned densimeter and conductivity meter has been built and operated in a recirculating sampler with salt and salt-acid solutions. The liquid system is made entirely of stainless steel and fluorothene with welded and Swagelok connections. The instrument has a sample volume of 600 ml and accommodates a sample flow rate of 200 ml/min. The precision of this instrument is $\pm$ 0.002 gm/ml for density and $\pm$ 0.5% for conductivity. The instrument presently includes no temperature controlling and compensating components. If satisfactory temperature compensating components are developed for this instrument the data from the primary elements could be presented on an X-Y recorder. The process operator would then be able to determine uranium and acid concentrations by direct reading with no graphical or algebraic manipulation of the data being required. A suitable knowledge of the ratio of metal ions present would be necessary for accurate results. Based on precisions of $\pm$ 0.002 gm/ml, $\pm$ 0.5% conductivity, and $\pm$ 0.1^{o}C the over-all precision of this instrument would be $\pm$ 3 gm/liter for uranium and $\pm$ 0.05 M for nitric acid.

3.4 Testing of Inline Instruments in ORNL Hot Pilot Plants

It is proposed that 19.0 man-months and a total of $45,000 be applied in FY 57 for the testing and proving of inline instruments and components in the ORNL hot pilot plants. The ORNL inline instrumentation budget contains a share of the cost for installing inline instruments in the pilot plants. Inline instruments which are needed mutually by SRL and ORNL, will be tested as they are available at ORNL.

The majority of the pilot plant testing will be done in the Metal Recovery Plant, which employs the Purex process, and constitutes a continuously operating proving ground for inline instrument candidates. Test facilities for the inline instrumentation group and process information requirements of the Metal Recovery Plant were reviewed 1 March 1956. Test situations for inline instruments are available in approximately 19 process streams in the presently operating Metal Recovery Plant and in the modified and expanded Metal Recovery Plants. The structural and piping requirements for a recommended sampling facility are specified in the building plans of the expanded Metal Recovery Plant. Shielded caves and recirculating samplers are being provided in the cell walls. The expansion contains the head-end and the first cycle and is scheduled for operation in 1958. Inline instrumentation facilities are being provided in the expansion for 12 streams, 7 of which were specified March 1 as follows:

a. Feed adjustment tank
b. HAW
c. IAW
d. HCW
e. HPC
f. IBP
g. ICU

Test situations for inline instruments in the ORNL Thorex Pilot Plant also will be available as needed. The Thorex Pilot Plant constitutes a second proving ground for inline instrument candidates. The Thorex Pilot Plant is a semi-continuous plant. This plant operates by runs followed by shutdown periods for maximum experimental flexibility.

3.5 Laboratory Study of Basic Parameters

It is proposed that 10.0 man-months and a total of $23,000 be applied in FY 57 to complete the laboratory study of basic parameters. This includes the proving of a prototype instrument in the Metal Recovery Plant.

3.51 A-C Polarography

The electro-potentiometric method was selected from the list of possible parameters proposed for the study. A-C polarography is being investigated for inline instrumentation application as a monitor for the continuous analysis of uranium concentrations up to 0.01 gm/liter in process waste streams. In the A-c polarographic method, a small a-c voltage is superimposed upon the polarizing d-c voltage that is applied to the mercury drop or microelectrode. The a-c voltage developed by the a-c current flowing through a measuring resistor is amplified, rectified, and presented on a recorder. The recorder automatically displays a derivative wave form whose height is directly proportional to concentration. The measurement of current at the half-wave potential is specific for the discharging ion, although ions with a similar half-wave potential can cause interference by addition to the half-wave height. The extent of any interference in the uranium analysis is being determined in a laboratory evaluation of the method. A method for eliminating the deleterious effect of the condenser current is being investigated. Phase discrimination is the method that will be used to eliminate the out-of-phase condenser current. A breadboard circuit development program is being pursued to evaluate various types of phase detectors for this application. The a-c portion of an A-C polarograph has been designed and construction has commenced with the building of the a-c amplifier section. The d-c portion is being designed and requires the building of a high impedance amplifier. Evaluation tests will be conducted with the constructed instrument. Approximately one month of evaluation testing is believed necessary to determine whether A-C polarography will be feasible for inline instrumentation application.

3.52 Other Parameters

If A-C polarography proves to be infeasible for the continuous monitoring of uranium in waste streams, it is proposed to investigate automatic coulometric titration for this purpose. Another possibility to be checked would be an X-ray, or gamma, fluorescence method. It is believed that ultra-violet radiation detection offers too low sensitivity for use in a uranium monitor for waste streams.

It is desired to evaluate a possible nephelometric method for application to continuous monitoring of uranium. It is possible that such a method could be developed into a limit alarm for detecting uranium present in more than a specified concentration in process waste streams. In theory a small side stream from the main waste stream could be continuously withdrawn and split into two sample streams. One of these sample streams would be treated with H_2O_2 and both streams would be passed through matched nephelometer cells. Any precipitate of uranyl peroxide could be detected by system unbalance. The sample streams could be returned to the waste stream where the excess H_2O_2 would decompose. Some of the ions found in process streams might interfere seriously with the described method. Precipitation may occur too slowly or the uranyl peroxide might possibly be too soluble for the method to be feasible. The particular need for monitoring uranium losses may, nevertheless, make an investigation of such a method desirable.

M. J. Kelly

J. W. Landry

R. W. Stelzner

MJK/JWL/RWS:aeh

4.0 APPENDIX

Table 1

Estimated Schedule of Manpower, in Man-Months, and Costs in $\$10^3$, for the Proposed Inline Instrumentation Program at Oak Ridge National Laboratory for FY 57

Proposed Program	Group[a] Man-Months	$\$10^3$	
SPECIFIC PROGRAM, manpower will be supplied by the ORNL Inline Instrumentation Group[a]			
Review of Process Control at ORNL			
Manpower	0.5	0.6	
Special Assistance from ICD[b]		0.4	
			1.0
Development of Sampling Facilities	0		0
Specific Gravity-Conductivity Instrument			
Manpower	6.5	7.8	
Equipment		1.2	
			9.0
Hot Testing			
Manpower	19.0	22.8	
Plant Modification and Equipment		22.2	
			45.0
Basic Parameters			
Manpower	10.0	12.0	
Equipment		11.0	
			23.0
GENERAL PROGRAM			
Consultation Manpower			3.5
Special E & M[c] Assistance Manpower and Supplies			4.0
Circuit Development			3.0
Travel Expense 1 Trip SRL, 1 Trip HAPO			1.5
Miscellaneous, including Models and Publications			1.0
Contingency, approximately 10% for Equipment Including Lead Shielding			9.0
TOTAL GROUP MANPOWER	36.0		
TOTAL COSTS			100.0

a. ORNL Inline Instrumentation Group: M. J. Kelly, J. W. Landry, R. W. Stelzner

b. ORNL Instrumentation and Controls Division

c. ORNL Engineering and Mechanical Division

Table 2

Estimated Schedule of Group Manpower, In Man-months, for the Proposed Inline Instrumentation Program at Oak Ridge National Laboratory in FY 57

Proposed Program	M. J. Kelly	J. W. Landry	R.W.Stelzner	Total
Review of Process Control at ORNL	0.5	0	0	0.5
Development of Sampling Facilities	0	0	0	0
Development of Specific Gravity -Conductivity Instrument	5	1.5	0	6.5
Hot Testing	5.5	8.5	5	19
Basic Parameters	1	2	7	10
Totals	12	12	12	36