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TRIP REPORT

FIFTH TRIPARTITE CONFERENCE ON INSTRUMENTATION AND VISITS TO SRP AND ORNL

Classification Cancelled and Changed To DECLASSIFIED
By Authority of Per Doc May 1973
By
Verified By P. Sullivan 11-8-99
J. E. Savely 1-20-00

SPECIAL RE-REVIEW
FINAL DETERMINATION
DECLASSIFICATION CONFIRMED
BY J. P. Deroun DATE 7-23-81
BY J. W. Jordan DATE 7-23-81

By

P. E. Brown

Process Control Development
Chemical Research & Development
HANFORD LABORATORIES OPERATION

December 12, 1956

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TRIP REPORT

FIFTH TRIPARTITE CONFERENCE ON INSTRUMENTATION AND VISITS TO SRP AND ORNL

ABSTRACT

The writer's trip to the Fifth Tripartite Conference on Instrumentation at Brookhaven National Laboratory and visits to the Savannah River Plant and Oak Ridge National Laboratory are reported.

Conference topics of particular interest only are discussed. These include the following:

The British delegation reports on infrared gas analysis; gamma, alpha, and neutron monitoring; the use of an ultraviolet absorptometer for uranium.

The Savannah River Plant reports on the swirl cell for beta-gamma counting and for a contact alpha counter, both for use on low-level effluents; the gamma absorptometer as a routine control laboratory instrument; induction methods for determining the thickness of slug coatings.

The general concern for reliability of components and systems which have been treated in many cases by the use of magnetic and transistor circuits.

At Savannah River Plant, the following topics were discussed: alpha and gamma counting in solutions; the spark counter; methods for in-vessel determination of neutralization end-point; plutonium determination by neutron counting in aqueous solutions.

At Oak Ridge National Laboratory, the following topics were discussed: an in-vessel probe for determining conductivity and density; a gamma monitor designed to avoid any surface contamination; the a.c. polarograph; the per cent composition indicator for TBP; neutron counting; count rate meter circuits.

INTRODUCTION

The writer attended the USAEC-sponsored Fifth Tripartite Conference on Instrumentation held at Brookhaven National Laboratory, Upton, Long Island, New York, from October 22 through October 25, 1956. The writer presented a paper, co-authored with D. G. Miller, at the conference entitled "A Continuous In-Line Monitor for Uranium or Plutonium."

The Savannah River Plant was visited on October 29 and 30 where the SRP in-line program was discussed with Messrs. Ice, Propst, Axtmann, Kropp, Cist, Weisner, Watkins, Martens, and Sheldon. Oak Ridge National Laboratory was visited October 31 and November 1 and 2 where the ORNL in-line program was discussed with Messrs. Landry, Kelley, Fisher, Ullmann, Culler, Lindauer, Manning, Glass, Bell, and Stelzner.

FIFTH TRIPARTITE CONFERENCE ON INSTRUMENTATION - BROOKHAVEN NATIONAL LABORATORY

The proceedings of the conference will be published by the USAEC, which sponsored the conference, at sometime during the next year. For this reason, details of all conference papers and discussions will not be covered in this report. Only those matters which are considered to be of immediate interest will be discussed. The agenda appears in HW-47050.(1)

A large majority of the conference papers and discussions was concerned with reactor instrumentation of all types. Heavy emphasis was placed by almost all the speakers on the need for a very high degree of reliability in components and systems.

It is of interest to note that the speakers representing builders of reactors for submarines or aircraft have, for all practical purposes, abandoned the use of vacuum tubes in favor of magnetic amplifiers or transistorized amplifiers. Discussion of component reliability showed that the National Laboratories, perhaps for reasons of flexibility, adhered to the use of vacuum tubes.

The following summarizes the points of interest in the papers concerned with process analytical instrumentation.

1. Mr. K. R. Sandiford of the United Kingdom Atomic Energy Authority discussed non-nuclear instrumentation associated with the operation of the Calder Hall power reactors. These reactors use CO_2 as the principal coolant and heat transfer medium. It is necessary to protect the coolant loop from contamination by steam and, conversely, to protect the steam loop from contamination by CO_2. Both detectors are continuous infrared gas analyzers using standard materials of the desired condition as references. To reduce corrosion, oxygen dissolved in the heat exchanger feed water must be minimized. Oxygen is analyzed for using a modified Hersch cell (a galvanic cell using KOH) for which a time constant of 80 seconds and an accuracy of ±5 per cent are claimed. The detection range is of the order of 0.1 to 0.3 ppm O_2.

2. Dr. Denis Taylor of the United Kingdom Atomic Energy Research Establishment at Harwell discussed the instrumentation of their separations plants, their gaseous diffusion plants, and their ore refinery. They have been plagued by the problem of sampling in their chemical separations plant. The attempted solution involved bringing "side streams" through their shielding and monitoring these for gamma activity. The buildup of background on these stainless steel pipes has effectively restricted the use of this method to streams of extremely high activity such as dissolver solutions. They have monitored the progress of their metal dissolution by means of a gamma monitor located close to the vessel. Apparently, they make, or attempt to make, a complete dissolution since the stated level of activity varies from 3×10^3 r to 1 r. Details of this application were refused on the basis of British security regulations.

A neutron monitoring system is used as a nuclear safety measure in the UF_6 plant, depending upon the alpha neutron reaction produced by U-235 in the fluorine. A BF_3 proportional counter is used with suitable moderation and shielding.

An ultraviolet absorptometer is used on the effluents from their ion exchange columns in the ore refinery. The system is automatic, in-line, and semi-continuous in operation. The material to be tested is drawn into the sample cell where ammonium thiocyanate and stannous chloride are added in predetermined amounts. After the reaction, the solution is measured using ultraviolet light. The system is used to determine U content down to 0.05 g./l. It operates on a 20-minute cycle during which it checks the streams from each of four ion exchange columns. An alarm is provided to indicate when a stream exceeds a predetermined value.

An alpha counter using ZnS scintillator protected by rubber hydrochloride film is provided to measure Pu in aqueous solutions. The solution flows into a weir box above which the phosphor is suspended. The details of this installation are covered in AERE EC/M90.

An absorptometer using thulium-170 is used for plutonium solutions of higher concentration. Further details of this installation were not released.

3. Messrs. Wilson and Cathey of Savannah River Plant discussed a number of instruments used for various monitoring and process applications. They have an absorptometer for U and Pu, but it is a laboratory analytical instrument not an in-line analyzer. It uses 10 mg. of Am-241, a multiplier phototube, and an NaI crystal. A shutter permits the source to be shut off so that a correction can be made for the gamma content of the sample. The samples are dropped into the apparatus in plastic sample vials. The readings are standardized against known U or Pu solutions. They claim an accuracy of better than ±0.5 per cent in the 50 g./l. range.

For beta and gamma monitoring, they use an adaptation of the ORNL Hurst swirl cell, with the solution entering at the bottom of the cylinder tangentially at 20 psig. This builds up to the top of the cell and thickens to form a wall whose thickness depends on the location of the outlet orifice at the bottom. The detector is an NaI(Tl) crystal extending down into the cell but at no time contacting the sampled material. It is coupled to the multiplier phototube by a lucite light pipe. There has been no trouble from splashing or diffusion. They have been bothered by the buildup of contamination on the uncoated stainless steel cell walls. Teflon would probably retain less activity but apparently will not stand the erosion caused by the high velocity of the entering stream. The method is used on effluent streams where the level of activity is quite low - about 7×10^{-7} curies of Sr-90.

The alpha monitor is a contact type used on effluent water to measure the amount of contamination due to U but will, of course, detect any alpha emitter present. They claim a sensitivity of 1×10^{-8} curies of natural U per liter. A General Electric Company plastic phosphor, GE 661A568GR1, 5-in.-diameter, uncoated ZnS, is used with a 5-in.-diameter multiplier phototube. The detector is coupled to the phosphor by a 1/4-in. air gap. A uranium source is located in the sample cup to give a source of alphas for indication that the unit is operating.

In cladding of slugs, two problems are involved: measurement of the Ni electroplated on the U and thickness of the bonding material for the jacket. The thickness of the Ni plate is measured by a transformer with an air gap which the slug bridges. The cladding thickness between 0.1 and 0.6 mils can be

measured. For the bonding coat, a similar method is used with the transformer operated at 5 Kc. The coating thickness can be measured between 8 and 20 mils.

To determine separation of the jacket from the bond, an ultrasonic technique is used. A barium titanate crystal generates an ultrasonic beam which is transmitted through the slug to a receiver. Depending upon the time taken to scan the slug, the total unbonded area can be determined. The readout is novel in that the display is on chemically treated, or teledeltos, paper. This gives a profile of black on white corresponding to the unbonded areas. The slug is submerged in water and is rotated on its axis as it passes through the beam.

4. Among other points of interest was the British method for restoring BF_3 counters. They build their tubes of copper and use quartz insulation. When the tube approaches the end of its useful life, they heat it to 300°C. which apparently "gets" the dissociated fluorine and restores the counter completely.

Mr. Bouricious of General Electric Co., ANP, described aluminum to ceramic seals which are useful up to 700°F. with resistance of about 5×10^{14} ohms.

Mr. Taylor of UK-AERE described their pretreatment of polystyrene insulators using gamma from Co-60 to improve the resistance.

VISIT TO SAVANNAH RIVER PLANT

The only "in-line" in prospect at the moment is a gamma monitor now on ICW (organic). Their experience with this has been very similar to that of Hanford - buildup of background and difficulty in sampling. The installation is not accepted by the operating people but is installed on a purely experimental basis. They use a cylindrical stainless steel cell with a Teflon coating. Coating was done by a commercial outfit that coats calendering rolls for papermills. Apparently, a number of formulations were tried but the present one is quite satisfactory. They have been able to rinse off most of the background contamination with dilute HNO_3. Because of the experimental nature of the installation, their sampling has been essentially intermittent, with samples being taken only when the operators have free time. This situation makes it difficult to interpret the data thus far collected.

The contact alpha counter, as originally reported, used multiplier phototubes with ZnS phosphor deposited on the surface; the end was protected by a 1/4-mil-thick Mylar film. The whole assembly was immersed in the test solution so that the phosphor (protected) was covered by solution. Results were excellent in some cases, erratic to bad in others. It soon became apparent that the difficulty lay in the presence of pinholes in the film which permitted the solution to leak onto the phosphor. They had little trouble decontaminating so long as the film was free of holes. They then tested 1/4-mil-thick Teflon films by immersing them in solutions. These films were mounted on Teflon rings and cemented in place. Activity adhering to the film itself was negligible after immersion in solution containing up to 2×10^7 d/m in aqueous. However, there was considerable contamination at the point where the film was cemented to the Teflon ring. This appeared to be a purely physical holdup. Their results indicate that adsorption on the film is negligible. They are not actively working on this instrument because of lack of interest on the part of the Operations people.

Another alpha method scheduled for eventual investigation either at SRP or under the research contract at ORNL is a gas-sweep counter in which a suitable counting gas is swept over a solution and counted. This has the advantage of avoiding almost all problems of background, although its application to the plant is certainly remote at present.

The SRP version of the ORNL Hurst swirl cell was discussed at the conference by Wilson. Additional comments by Axtmann indicate that it cannot be used successfully below 15 psig. inlet and, on anything except low-level effluent streams, it is troubled by background buildup. The top-inlet ORNL original will work with less pressure, but there is considerable splashing and the results are no better on background.

They are using a high resolution specific gravity measurement as one method of determining the degree of elution from the resin beds. (See below for method now favored) Specific gravity is also being used by Wes Lewis at the ORNL Metal Recovery Plant.

Axtmann also commented on the absorptometer (lab model) described in detail at the conference. With standardized vials for sampling, they claim to hold within ±0.2 per cent; with random choice of sampling vial, they hold within 0.5 per cent. The shutter type is still in development and its application to plant use is problematical. It is apparently being designed to reduce the slight amount of work involved with the present laboratory model and will be used in the lab.

Axtmann had some interesting information on use of two "in-vessel" methods of determining neutralization end points in waste tanks. The first involves a temperature measurement. There is a clear and reproducible correlation between the addition of NaOH and temperature rise in the tank - from three pH up to nine pH the temperature slope is steep; at nine pH there is an abrupt change of rate of rise (see also HW-43842(2)). Similarly, conductivity shows a clear decrease to the point at which Fe is removed, at which point it becomes relatively steady; nine pH there is an abrupt break with a sharp increase in slope. Neither method is used by the plants. In both cases, it is imperative that the tanks be kept thoroughly agitated.

The alpha spark counter was touched on briefly at the conference and in HW-43842.(2) It is still very much an experimental device. They have firmed up nothing in the way of a final design; they are not certain that they are really counting alpha. The last design involves the immersion of a grid in the solution which is kept at ground with a screen immediately above the solution, also at ground, and a plate about 0.1 inch above that charged at 3 to 6 x 10^3 volts. They have counted up to 10^3 c/m from solutions containing 10^3 d/m/ml. They have reason to believe that some of this count is spurious, but the indications are that it <u>is</u> proportional to the alpha present. The previous device used a single wire charged above a grounded plate which was held over the solution. With this configuration, the plateau was about 10^3 volts long; with six wires above the plate, the plateau was reduced to about 300 volts. They are continuing this work.

Kropp offered one comment which may be of considerable value if a corrosion resistant solenoid is ever found. To avoid sticking of the valves due to overheating, they operate them at about 40 volts DC. It appears from their tests that the coils are badly overrated when used on 115 vac, with the resultant heating and eventual failure.

The group under Cist has been responsible for the development of the neutron monitoring equipment now in use in the separations plants. The monitor was originally designed as a nuclear safety device to detect the buildup of plutonium in the 2A mixer-settler bank. The results were excellent. (Discussions with Sheldon and Martens elicited the information that the instrument had been down for less than a week out of the year.) It soon became evident that the results were sufficiently exact to use the monitor for the determination of the quantity of plutonium present in the bank. The neutron counter has since been applied to the resin columns where the counter tubes are mounted in the moderator shield on the outside of the vessel to detect the movement of plutonium onto the bed. It has been applied to the transfer tanks where the counting tube is located in a pipe down the center of the tank and uses the solution as a shield and moderator. It is used also on the precipitators and the recovery tanks. They plan installations to the number of 16 on the new Jumbo B Line and on both of the 1B mixer-settler banks.

They use Nancy Wood BF_3 tubes operated at around 2000 volts. They use an A1A type of linear preamplifier operated very conservatively for the canyon installations - 80-90 volts B+ and a 5-5.5 volts on the heaters. On the safety monitors in the B Line, they use a simplified all-transistor amplifier and count rate meter. The accuracy claimed is better than one per cent, although not for all of the installations where there is some dependence on level.

J. D. Anthony of this group has produced a portable sniffer analyzer for tritium.

VISIT TO OAK RIDGE NATIONAL LABORATORY

Minton Kelley of the Instrument Division has been working on in-vessel measurement of density and conductivity in hot, but not boiling, solutions. This is designed to give an indication of the uranium plus nitric acid content. Tests on the pilot model have so far been encouraging. The probe is installed in the condenser of one of the Metal Recovery concentrators where the temperature is about 95°C. and the ionic content varies as the concentrator boilup is held within limits or exceeded. The entire detecting unit is assembled as a single stainless pipe and consists of the conductivity electrodes plus the bubbler-type density tubes; the whole assembly is put into the tank through a flange and bolted in place. The problem of obtaining a reliable sample is thus not a question.

Work is being pushed on a gamma monitor in which the measured solution will not contact any surface which the scintillator "sees". The method involves the formation of a thin, splashless pencil of liquid falling by gravity in front of the scintillator. The entrance and exit orifices are shielded from the view of the scintillator by lead. The lead shield may be heated to reduce the likelihood of condensation and contamination. They have tested this device "cold" and, at the time, fabrication of parts for a "hot" installation was in progress.

These are the only two in-line monitors presently under development at ORNL. Work is also being done on AC polarography, but its present status is such that its application to plant use is remote.

J. W. Ullmann of the Chemical Technology long-range planning group emphasized that continued advances in in-line analysis would be demanded by the new techniques for chemical processing of the new power and military reactor fuel elements.

R. B. Lindauer, Head - Pilot Plants Section of the Chemical Technology Division, is much interested in the HAPO in-line program. He offered the facilities of both the 3019 Hot Pilot Plant and the Metal Recovery Plant for testing of HAPO devices which for any reason it might be difficult or undesirable to test or prototype in the HAPO plants.

F. W. Manning of the Instrument Division supplied the circuit diagrams and specifications of the percentage composition indicator which was used in the ORNL Purex demonstration for the determination of the per cent TBP in diluent. He noted, however, that the Foxboro Company now has a commercially available device which is somewhat simpler and probably more reliable.

Difficulties encountered with the ORNL CP1511 count rate meter supplied to HAPO by Radiation Counters Laboratory and by Victoreen Instrument Company were discussed with F. M. Glass who designed the circuit.

Methods of neutron counting in solutions were discussed briefly with P. R. Bell of the ORNL Physics Division. He stated that counting with BF_3, B_{10}, or scintillators such as LiI offered no major advantages one over the other.

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1. HW-47050 "Trip Report - Fifth Tripartite Instrumentation Conference at BNL," D. C. Pound.

2. HW-43842 "Trip Report - SRP," R. J. Brouns - A. E. Smith.