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TRIP REPORT - VISIT TO THE ANALYTICAL CHEMISTRY AND [redacted] INSTRUMENT DEVELOPMENT SECTIONS OF THE DuPONT COMPANY'S SAVANNAH RIVER PLANT

D. G. Miller

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TRIP REPORT - VISIT TO THE ANALYTICAL CHEMISTRY AND INSTRUMENT DEVELOPMENT SECTIONS OF THE DuPONT COMPANY'S SAVANNAH RIVER PLANT

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At the Savannah River Plant the writer contacted Dr. C. H. Ice of the Analytical Chemistry Section, Dr. R. G. Axtman of the Instrument Development Division and many of their associates.

The present development program of the Analytical Chemistry Section, headed by Dr. H. M. Kelly, as outlined by Dr. Ice and Dr. A. L. Marston is as follows:

LABORATORY METHODS

Solvent Quality. The "F" test for the performance of the tributyl phosphate solvent in the Purex process has been modified. The new procedure, developed by R. M. Wallace and E. R. Russell, will be known as the Zirconium Index or "Z" Test. "Z" is the number of moles of zirconium retained per billion liters of solvent after the test is completed. An aliquot of solvent is washed and equilibrated with Zr-Nb tracer solution of known specific activity and standardized total zirconium concentration. It is then scrubbed with 3 M HNO_3. The activity retained by the solvent is then a measure of the "Z" number. This method has been in use for a short period of time at the Savannah River Plant and has shown good correlation between the "Z" number and plant performance of a given solvent sample. The writer has a detailed outline of the "Z" test method.

Coulometric Titration of Plutonium and Uranium. Work on this analytical method is continuing and essentially parallels the work done or currently in progress at Hanford. The Savannah River Laboratory has not yet found the method satisfactory.

Aniline Point. This method is currently under investigation as a possibility for the determination of the tributyl phosphate concentration in solvents. The dialectric constant and acid saturation methods are presently in use.

PLANT ANALYSES

Gamma Monitor. A gamma monitor utilizing the pulse counting technique is currently under development. No plant installations have been completed. An attempt has been made to use a count rate meter with a cold-cathode circuit. Because of the large input threshold of this instrument, the effect of temperature on phototube gain has been a limiting factor.

Alpha Monitor. Various methods are currently under consideration for the detection of alpha activity by the use of ZnS phosphors emersed in the solution to be analyzed. The use of mylar film to prevent contamination of the phosphor has shown some promise. Some work has been done using a detector consisting of a phototube on which ZnS has been deposited directly utilizing Krylon or other plastic materials as the bonding agent. This work closely parallels the work currently being performed in the Chemical Instrumentation Unit here.

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O/A Analyzers. Five in-line turbidimeters have been constructed and two of these are currently installed in process vessels. The performance of these instruments is now being followed by the Instrument Development Section and will be discussed in a later section of this report.

COLORIMETER - CONDUCTIMETER

An instrument is under development for the measurement of uranium by the light absorption technique (photometer) and acid by conductimetry. The photometric method for uranium is similar to the method used here at the Purex Semi-Works. The conductimetric acid method is described in DP-76.

Gamma Photometer. The gamma absorptometer developed here at Hanford, using either Tm-170 or Am-241 as the photon source, is to be used at the Savannah River Plant for the determination of uranium and plutonium.

Polarograph. The Savannah River Laboratory has been looking at the polarographic method for the in-line determination of low-level uranium concentrations. Again, this work parallels the work at Hanford with the same problems, e.g., iron interference, encountered and not yet surmounted.

Miscellaneous Analytical Problems. Other analytical problems under consideration at this time include: x-ray diffraction studies for the characterization of the plutonium fluorides and their hydrates, plutonium oxides, chemical analyses for oxygen and fluorine, fission counting, alpha pulse height analysis using the gridded ion chamber technique, alpha-gamma coincidence counting, and improved design of the gamma scintillation counter to reduce the sensitivity of this instrument to source position.

EMISSION SPECTROSCOPY (Dr. A. L. Marston)

Micro Electrolysis. Work is currently being performed toward the development of a method for the determination of hydrogen/tritium ratios by exciting and analyzing the emission spectrum of the off-gas produced by the electrolysis of .05 milliliter water samples.

Closed Tube Excitation. In order to eliminate the need for extensive gloved box techniques in the excitation of radioactive sources for emission spectrographic analysis, a closed tube excitation method is being investigated.

Infrared Spectrometry. A method for the determination of organic solvent in aqueous solutions has been developed and placed in use. The method involves extraction of the organics into carbon tetrachloride and determination of the infrared absorption spectrum. The method will detect less than 0.5 percent of organic in aqueous solution with a relative precision of ± 10 percent The infrared technique has also been applied to the determination of TBP in ultrasene.

Mass Spectrometry. Methods for the determination of hydrogen deuterium ratios are being established.

Tri-Non Infrared Analyzer. This instrument is to be applied to the continuous determination of deuterium in water samples.

Ion Exchange. The Savannah River Laboratory has been employing an ion exchange method for the determination of plutonium in solutions containing fission products, uranium, and americium-curium. Ion exchange is also used for the separation and determination of zirconium. It has also been shown that this method can be used for Am-Cm in dissolver solution, although the lab is still using the CeF_4 procedure. These methods utilize very small columns and both Dowex-2 and Dowex-50 resins. Details of these procedures will be issued within three months as a DP report authored by E. R. Russell.

A few of the problems currently under investigation by the Instrument Development Division are as follows:

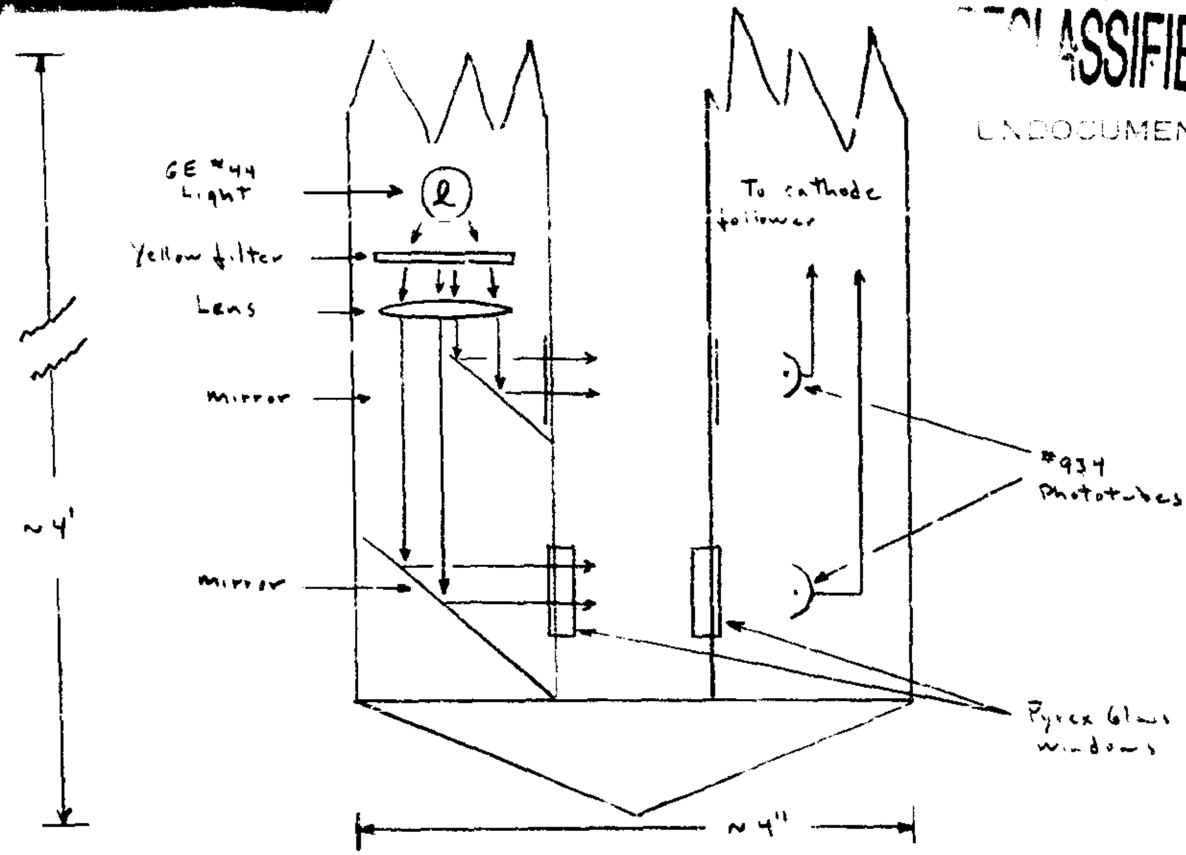
Weir-Type Alpha Counter. A weir-type alpha counter has been developed for the determination of alpha emitters in solution. This instrument utilizes a 10-inch by 14-inch ZnS screen. The screen is of plexiglass with ZnS powder dusted on and bonded using Krylon plastic spray. The screen is held 1/8 inch above the solution surface and is heated to a temperature greater than the solution temperature to prevent condensation of vapors on the screen surface. A 5-inch phototube "looks" at the screen. No light pipe is used but a skirt-like reflector surrounds the phototube and screen. This instrument has a background of 4 c/m and a solution containing an alpha emitter at a concentration of 200 dpm/ml yields a counting rate of 21 c/m.

Swirl Type Beta-Gamma Counter. An instrument utilizing the swirl-type sample cell and a terphenyl-in-plastic detector has been developed for beta-gamma counting in the in-line program. This instrument employs the pulse-counting technique.

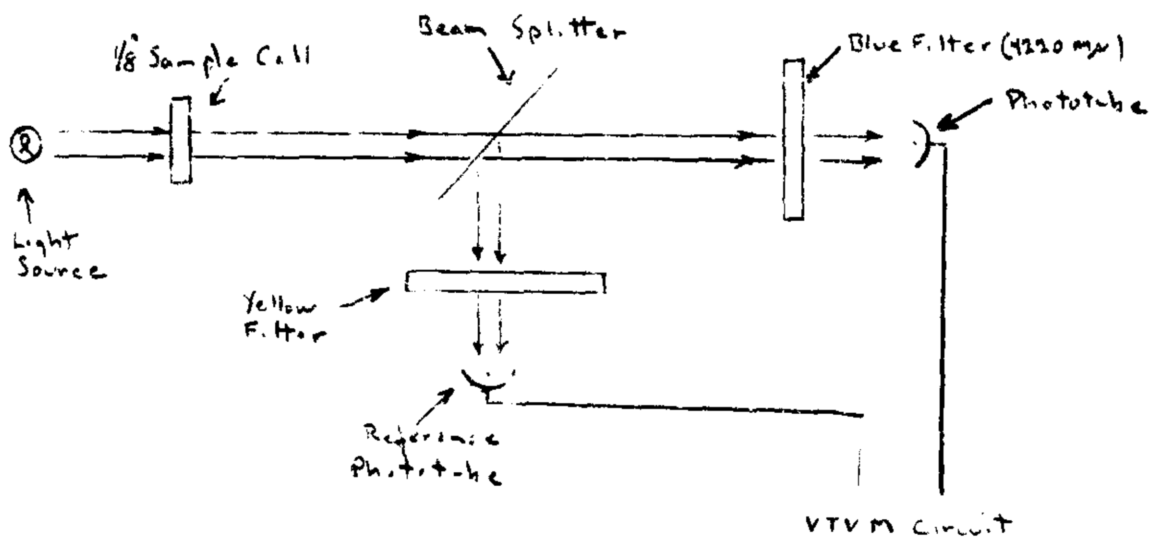
Sub-Critical Pile for Reactivity Measurements

A method developed by Axtman, Dessauer, and Parkinson for the determination of the reactivity of various fuel elements is used at the Savannah River Plant for the determination of the U-235 content of the 5 percent uranium in aluminum slugs. The method yields an absolute precision of ± 0.2 percent (standard deviation?). The instrument costs something less than $50,000. The writer has a report describing this instrument in detail. The time required for reactivity measurements using this instrument is only one minute per slug.

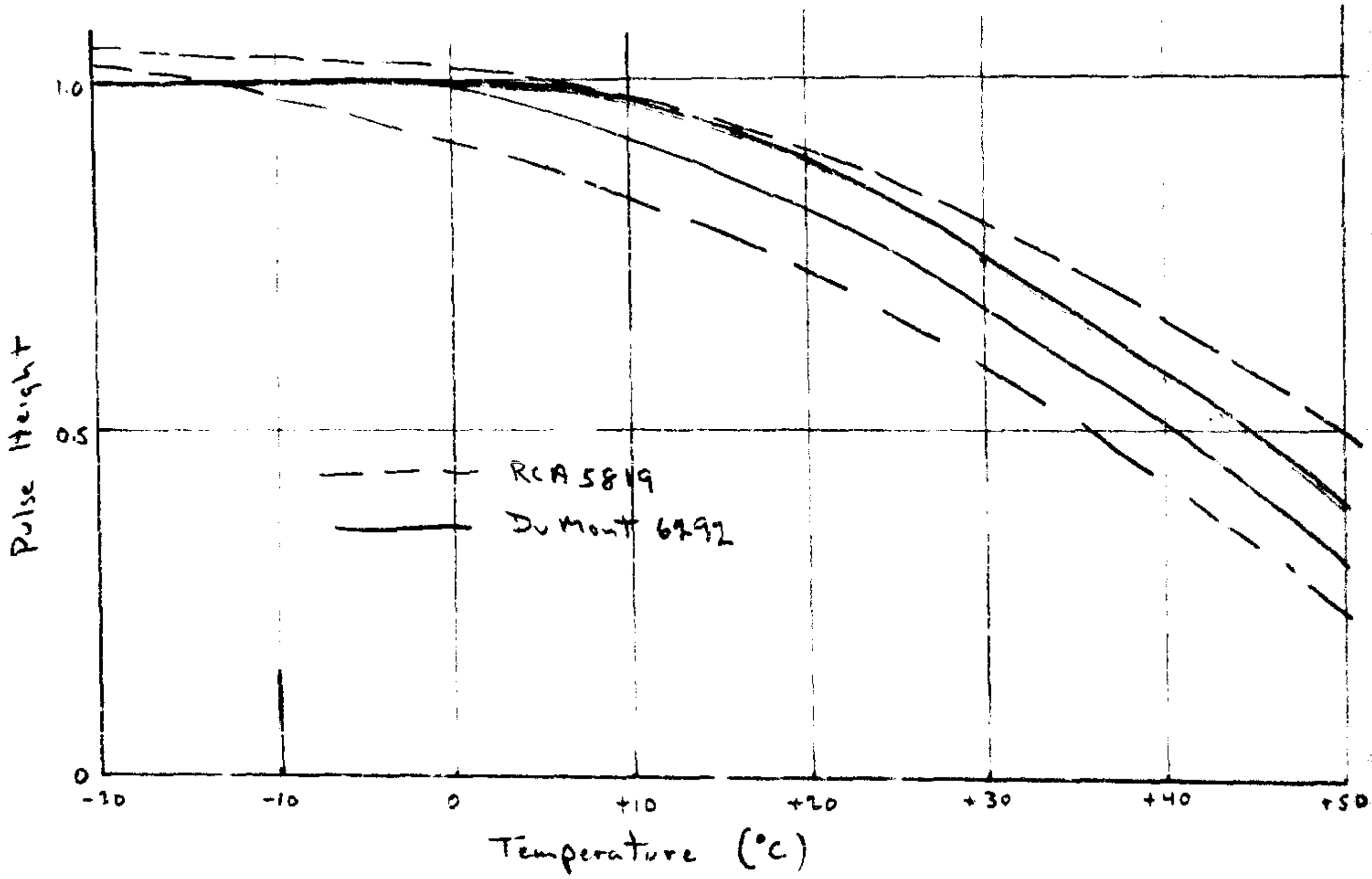
In-Line Turbidity Measurement. As mentioned previously, two turbidimeters are currently in use in plant locations. These are installed in low activity, aqueous waste streams. The instrument is designed for the determination of entrained organic in the range 0 - 1 percent and yields an absolute precision of ± 0.2 percent. The response of the instrument is dependent on particle size. The instrument employs a dual-beam optical system presenting two different absorption path lengths. The ratio of intensities of the two beams is a measure of turbidity. The ratio circuit employed makes the instrument insensitive to fluctuations in light intensity or fouling of cell windows. The turbidity is a function of acid concentration between 0 and 0.05 M but is independent of acid concentration above this range. The application of this instrument to in-line analysis is especially interesting in that it is installed directly in a process tank. The instrument is sketched roughly below.



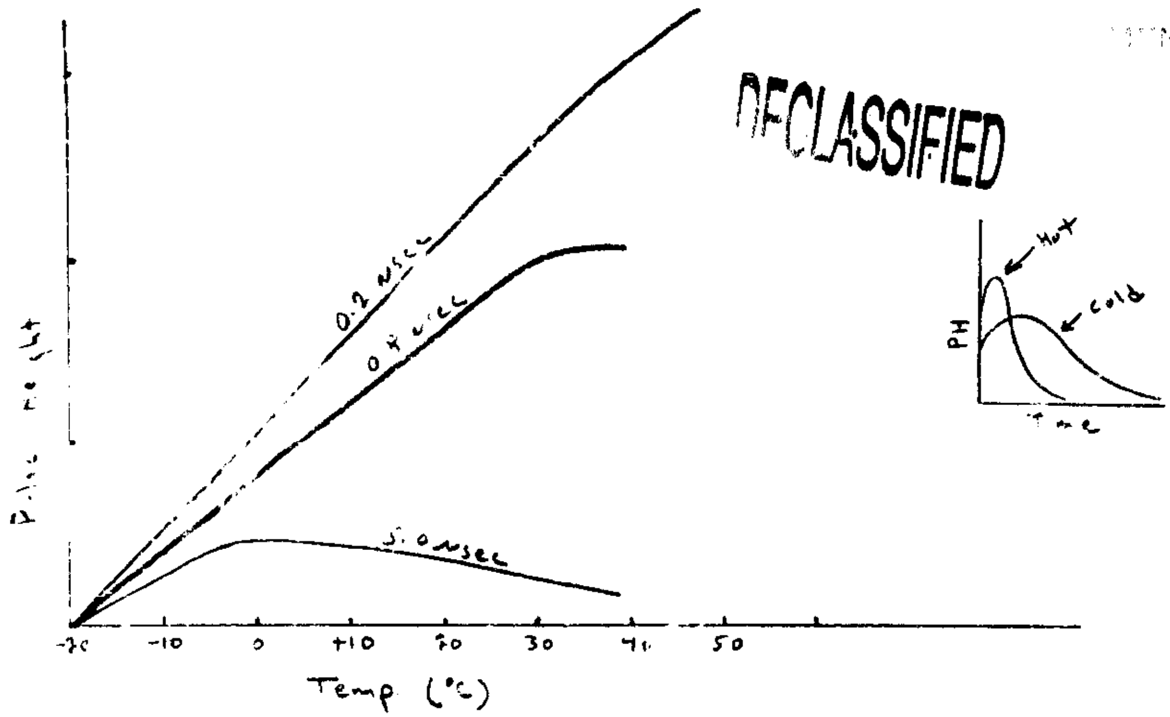
<u>Uranium Photometer</u>. A uranium photometer has been developed for the in-line determination of uranium in the 1.1 to 1.4 <u>M</u> concentration range. This instrument also utilizes a dual-beam system for the elimination of the effects of fluctuation in light intensity. The instrument may be described as follows:



Temperature Coefficient of 5819 to 6292 Phototubes. Dr. F. E. Kinard has carried out a rather extensive investigation into the effect of temperature on the response of the 5819 and 6292 multiplier phototubes. He has also investigated the effect of temperature on the response of NaI(Tl). He has found a rather large temperature coefficient for both tube types and has found a large variation (~ 20 - 30 percent) between tubes of the same type. Roughly, the data for two tubes of each type would look approximately as follows:



Dr. Kinard has also shown that the decay time for NaI(Tl) changes with the temperature resulting in faster pulses at higher temperature. For this reason the observation of the temperature coefficient for NaI(Tl) is dependent on the response time of the pulse analysis system used. Below is a plot of pulse height versus temperature for NaI(Tl) using pulse analysis systems with three different response times:



Dr. Kinard's work will be reported in detail in a forthcoming issue of RSI and will be discussed at the scintillation-counting symposium early next year.