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DP-MS--85-127

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ONLINE FIBER-OPTIC PHOTOMETER

by

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A paper proposed for presentation at the
Du Pont Process Analytical Instrumentation Symposium
Wilmington, DE
October 16-17, 1985

This paper was prepared in connection with work done under Contract No. DE-AC09-76SR00001 with the U.S. Department of Energy.

DISCLAIMER

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ABSTRACT

The development and implementation of a fiber-optic photometer for process control at the Savannah River Plant (SRP) will be discussed. The instrument is a modified Du Pont 400 photometric analyzer which incorporates quartz fiber-optic cables, a high-intensity tungsten-halogen lamp source, and a sight glass with sealed optics. Six of these photometers have been installed at SRP to monitor the elution of neptunium or plutonium from anion exchange columns. The fiber optics allow the instrument to be located 50 feet from the sight glass, which is in a highly radioactive area. This ensures easy access to the instrument and minimizes radiation exposure to personnel. The modifications to the analyzer and its application to process control will be presented.

* The information contained in this article was developed during the course of work under Contract No. DE-AC09-76SR00001 with the U.S. Department of Energy.

INTRODUCTION

As part of the production of plutonium-238 heat sources at the Savannah River Plant (SRP), neptunium and plutonium are separated from other elements, precipitated, and converted to an oxide form. To increase the efficiency of the precipitation step, the Np or Pu is loaded onto an anion exchange column, and then eluted as a concentrated plug of solution. This process is shown in Figure 1. Upon elution of Np and Pu, valves must be switched to obtain a heart-cut, maximizing the concentration of collected product. In the past, operators performed this task manually, relying on a visual indication of the product in a sight glass. Recently the processing facility has been upgraded to run under distributed process control, using automatic valve switching. This necessitated an automated indication of product elution from the anion exchange columns.

A photometric monitor based on the Du Pont 400 photometric analyzer was chosen to provide elution information. The Du Pont 400 is a two-wavelength filter photometer which ratios the logarithmic output from two phototubes. The measuring wavelength is chosen on a peak of the species of interest. The reference wavelength is chosen at or near the baseline of the spectrum. By figuring ratios of the log intensity at these wavelengths, the concentration of analyte in the stream can be determined.

Due to the radioactive nature of the elements being separated, the anion exchange columns and sight glasses must be contained in a glovebox. This precluded the direct application of the analyzer,

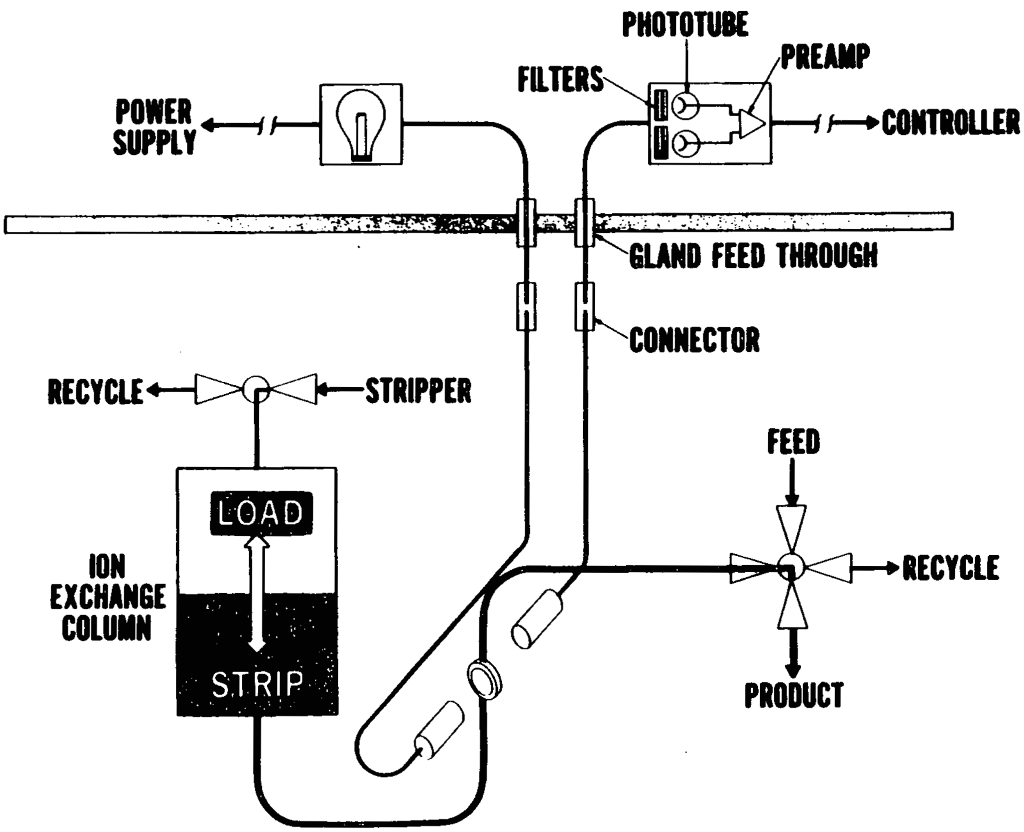

FIGURE 1. HB-Line Photometer Process Schematic

since this environment would be too harsh for the instrument. The solution was to modify the analyzer for use with fiber optics.

EXPERIMENTAL

The Du Pont 400 photometric analyzers were modified for use with fiber optics. Each analyzer consisted of a lamp housing, photometer housing, and control station (Figure 2). In this application, the sample housing was not required. In the lamp housing, the 6-volt transformer and 6.3-volt tungsten lamp were replaced by a 12-volt transformer and a 10.8-volt tungsten-halogen lamp (General Electric Model EPT). The tungsten-halogen lamp provides the higher light intensity required by the fiber optics, and has a rated lifetime of 8000 hours. The secondary from the transformer was stepped down to 9.8 volts to extend the lamp life even further.

An upscale driver-circuit was added to the photometer section. This circuit drives up the output from the reference phototube during low-light/no-light situations so that a high absorbance signal is displayed.

The control stations were repackaged for mounting in a shallow wall cabinet (Figure 3). Each control station was equipped with a 4-20 mA output, a dual setpoint current alarm, and a remote calibration option. The 4-20 mA output drives a strip-chart recorder located in the control room and allows the operators to monitor the elution cycle. The current alarm has two independent setpoints which are used to signal the process controller to switch

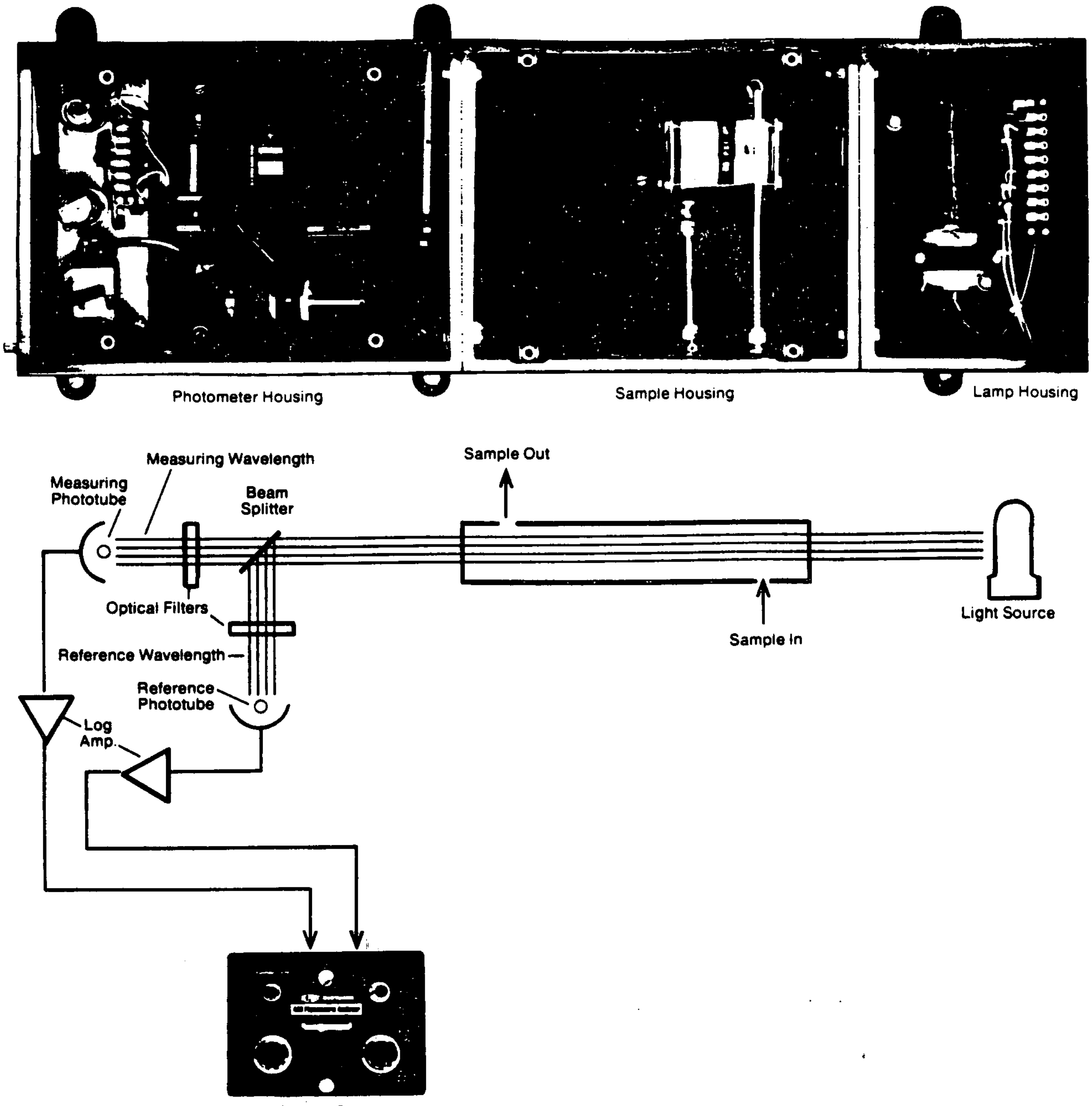

FIGURE 2. Du Pont 400 Photometer Analyzer Components

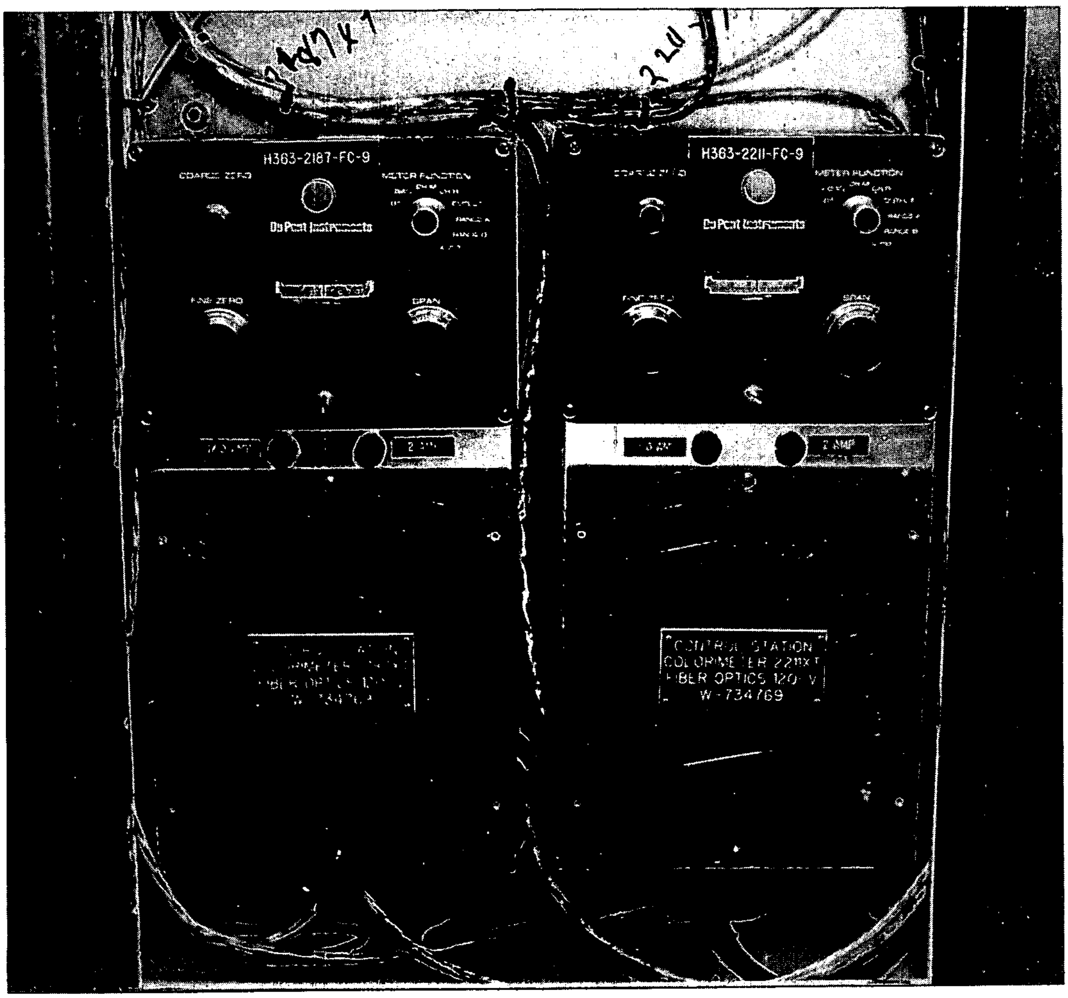

FIGURE 3. Mounting at Control Stations in Shallow-Wall Cabinet

valves. The remote calibration option allows the process controller to move a calibration filter into the light path to verify correct operation of the instrument before the elution cycle is started. The calibration filter is located just inside the photometer housing and is controlled by an air-driven solenoid.

Fused silica fiber-optic cable (Fiberguide Industries, Stirling, NJ) was used to transmit light to and from the photometers. Fused silica was chosen instead of glass or acrylic optical fiber due to its excellent resistance to radiation and high transmission efficiency over long distances. The cable was composed of a bundle of forty-nine 200-micron-diameter fibers encased in a polyethylene sheath. A multiple fiber bundle was chosen instead of a larger diameter single fiber because it carried more light to the phototubes, increasing the dynamic range of the photometer. The ends of the fiber bundles were terminated in 1/4-inch stainless steel tubing, for easier connection to standard Swagelok™ fittings.

Connecting fittings shown in Figure 4 were made out of 304L stainless steel and mounted directly to the lamp and photometer housings of the Du Pont 400 analyzers. The fittings were tapped to accept 1/4-inch pipe to 1/4-inch tube Swagelok™ fittings. The ends of the fiber bundles were secured in the fittings with Teflon® (Registered trademark of Du Pont) ferrules. Hand-tightening was adequate to secure the fibers; no hand tools had to be introduced into the glove box to make connections.

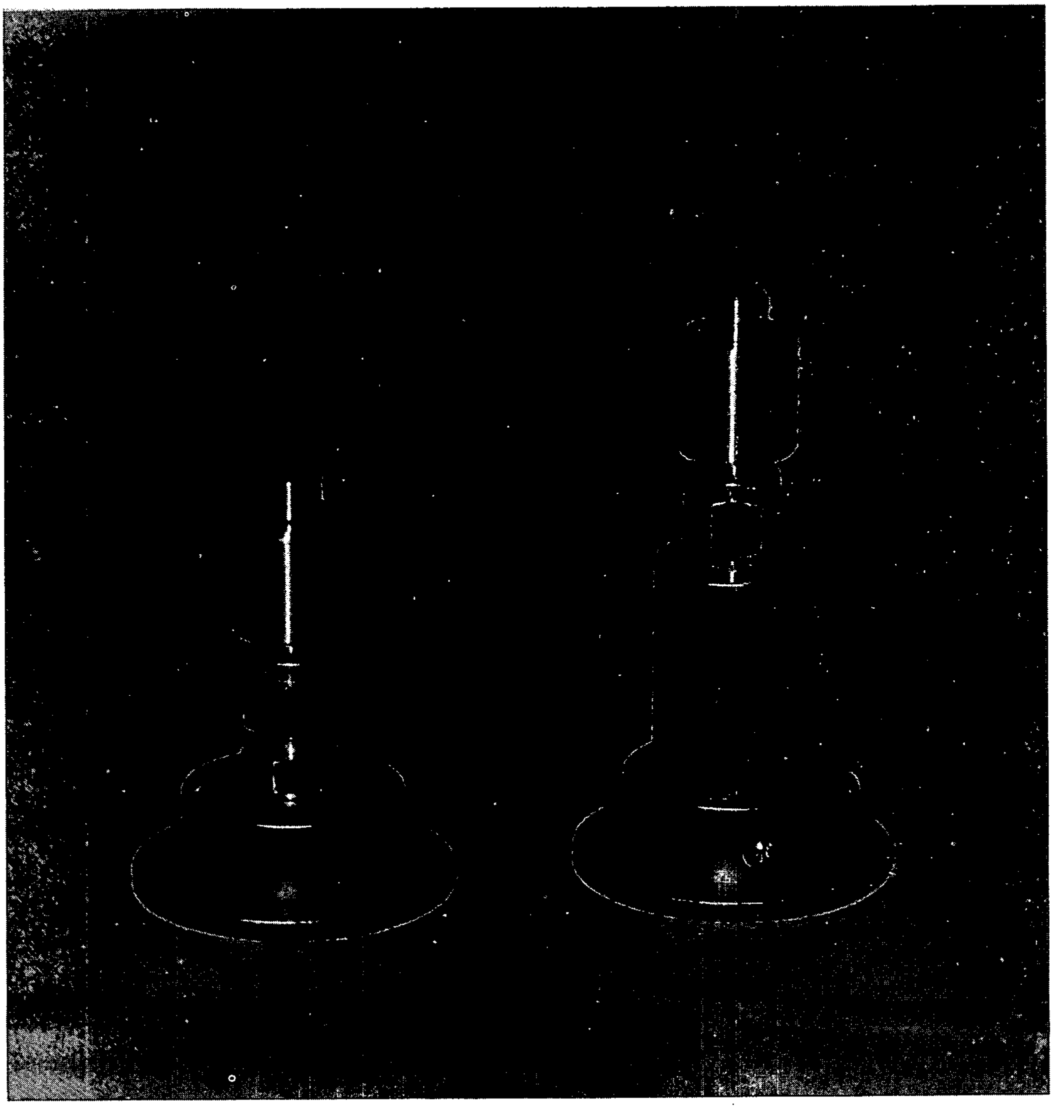

FIGURE 4. Stainless Steel Connecting Fittings

Lens assemblies shown in Figure 5 were fabricated from a 1-inch O.D. stainless steel tube and a 1/4-inch Swagelok™ fitting. A 1-inch diameter, 1 inch focal-length glass plano-convex collimating lens (Melles Griot, Irvine, CA) was edged slightly to reduce the diameter. The lens was seated in the end of the tubing and the fiber-optic cable was connected to the Swagelok™ fitting.

A sealed sight glass, designed for use with fiber optics, is shown in Figure 6. It was machined from 304L stainless steel, has a 2 cm pathlength, and used 1-inch Swagelok™ fittings for connection to the fiber-optic lens assemblies.

The two rectangular windows were made from fused silica and were secured by 1/16-inch Viton B® (Trademark of Du Pont) gaskets. The large windows provided two viewing regions in the sight glass, one for the fiber optics and one for operator viewing to confirm proper operation of the photometers. Past experience showed that optical surfaces inside the glove boxes invariably became coated with a film that reduced the optical transmittance. The present sight glass uses Swagelok™ fittings to seal off the fiber optics from vapors in the glove box to prevent this coating.

DISCUSSION

Absorbance spectra for plutonium and neptunium are shown in Figure 7. Both species are in the +4 oxidation state and form relatively stable nitrate complexes. It is these negatively charged nitrate complexes which load onto the anion exchange column. To get near quantitative loading, the nitric acid concentration is adjusted to 7-8M. To elute the neptunium or plutonium

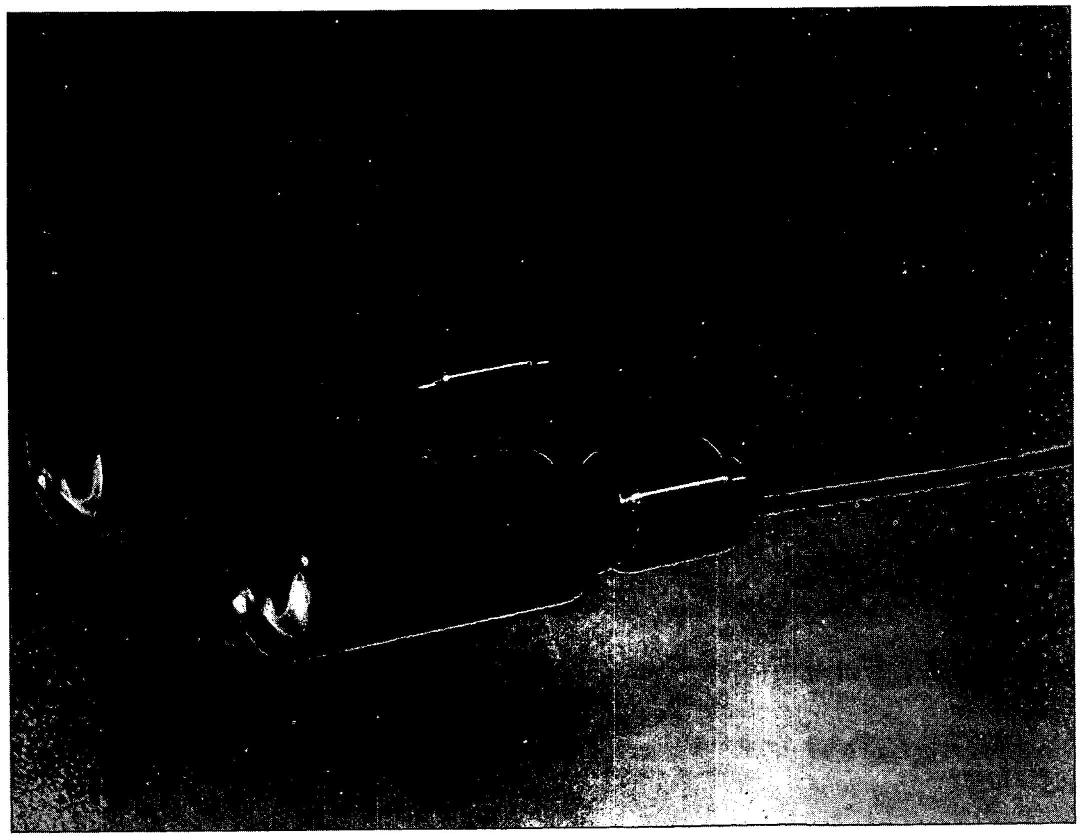

FIGURE 5. Lens Assemblies

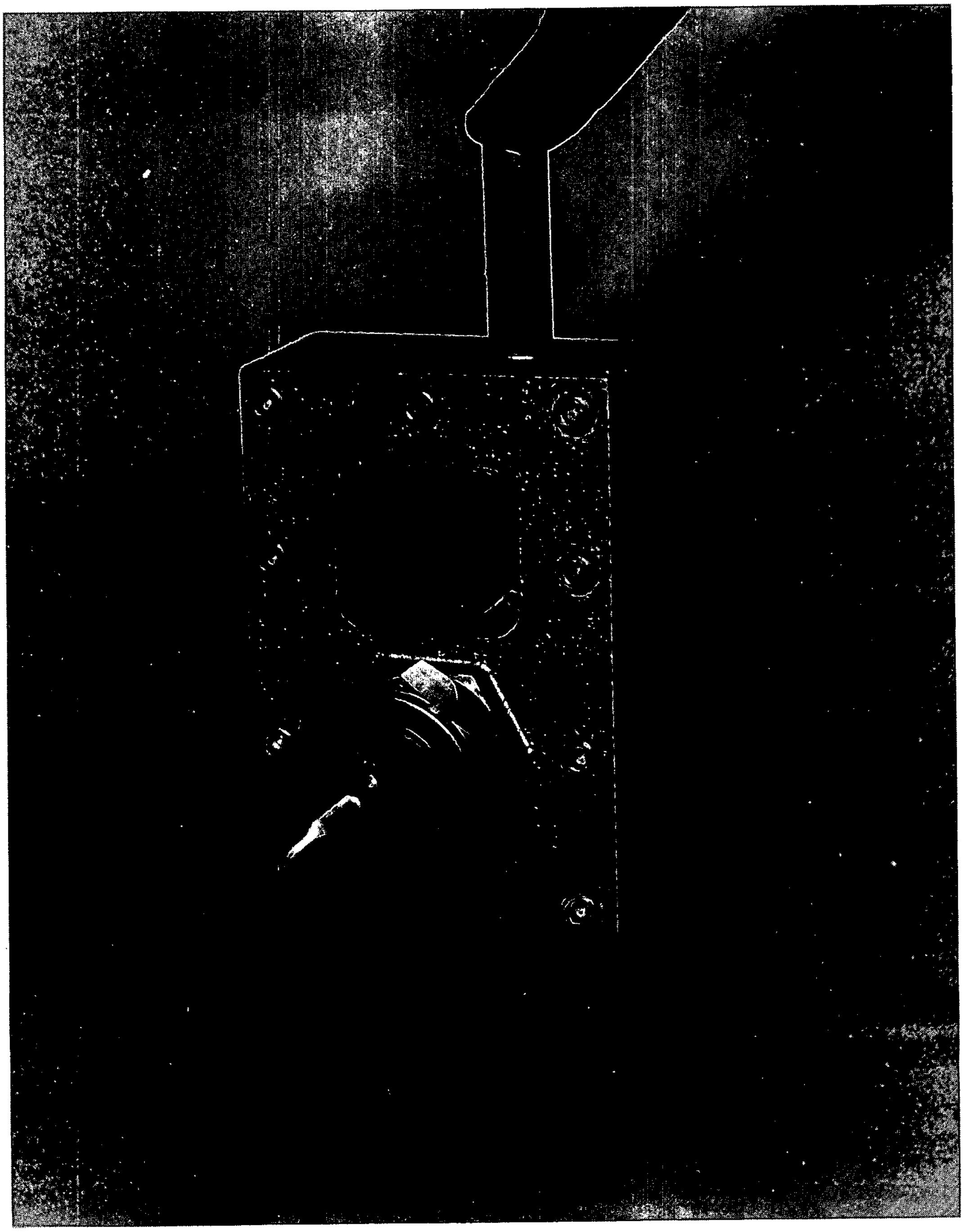

FIGURE 6. Sealed Sight-Glass

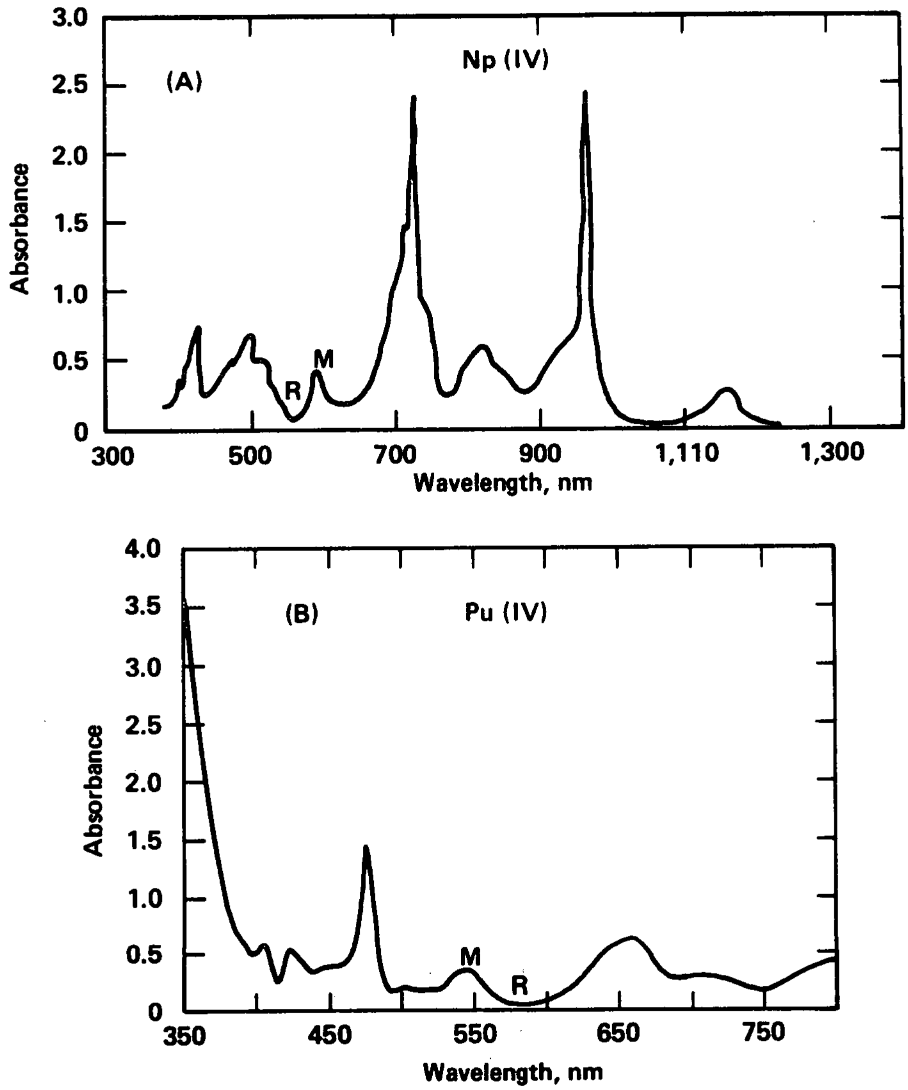

FIGURE 7. Absorbance Spectra for a) Np(IV), b) Pu(IV)

from the column, the nitric acid concentration is decreased to about 0.2M. The lower nitrate concentration shifts the equilibrium away from the anionic nitrate complexes and allows the species to be stripped from the column.

The reference and measuring wavelengths were chosen from the absorbance spectrum for each species of interest. The wavelengths were chosen to show some absorbance at the loading concentration, while maintaining enough dynamic range to monitor the elution profile. For neptunium, the measuring wavelength was set at 595 nm and the reference at 565 nm. For plutonium the measuring wavelength was 546 nm and the reference 585 nm. In both cases a relatively small peak was chosen, allowing the analyzer to measure the higher concentrations of product during the elution cycle.

A schematic of the fiber-optic photometers and the anion exchange process is shown in Figure 1. Feed solution passes through the analyzer sight glass and is loaded onto the anion exchange column. Once the column is loaded, it is backflushed with low-acid solution to strip the product from the column. As the product starts to elute, it is detected by the analyzer and a signal is sent to the process controller to switch the valve from "recycle" to "product". The concentration of the product eluting from the column increases to a maximum and then decreases slowly to zero. When the concentration falls below a preset limit, a second signal will direct the controller to switch the valve back to "recycle".

Two photometers for monitoring neptunium have been installed and are shown in Figure 8. They are located above a suspended ceiling, directly over the glove box containing the neptunium anion-exchange columns. The sight glass and adjacent anion exchange column are shown in Figure 9. The sight glass shown has been replaced with the new sealed optics design.

Four more photometers are currently being constructed to monitor plutonium. All four of these analyzers will be located in wall panels across from the glove box windows for easier access.

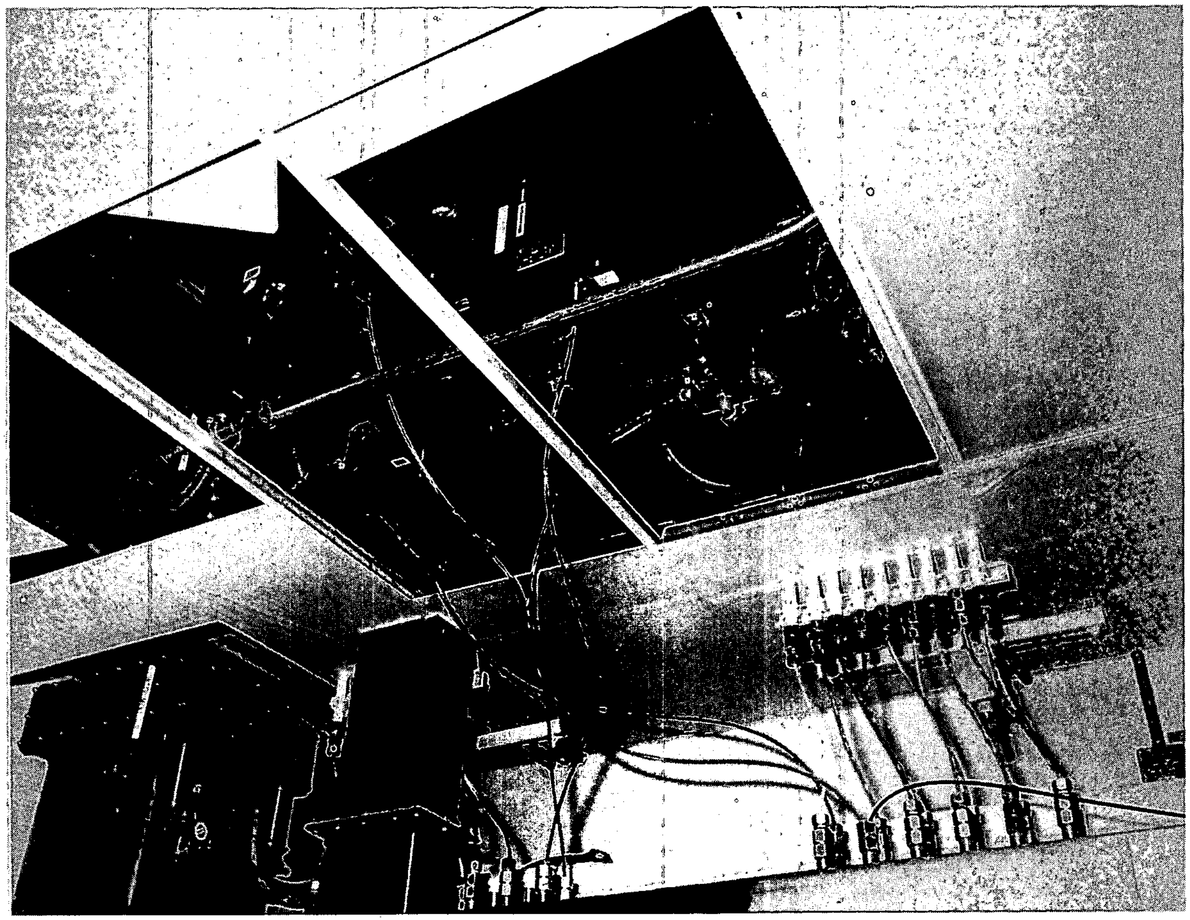

FIGURE 8. Photometers for Monitoring Neptunium

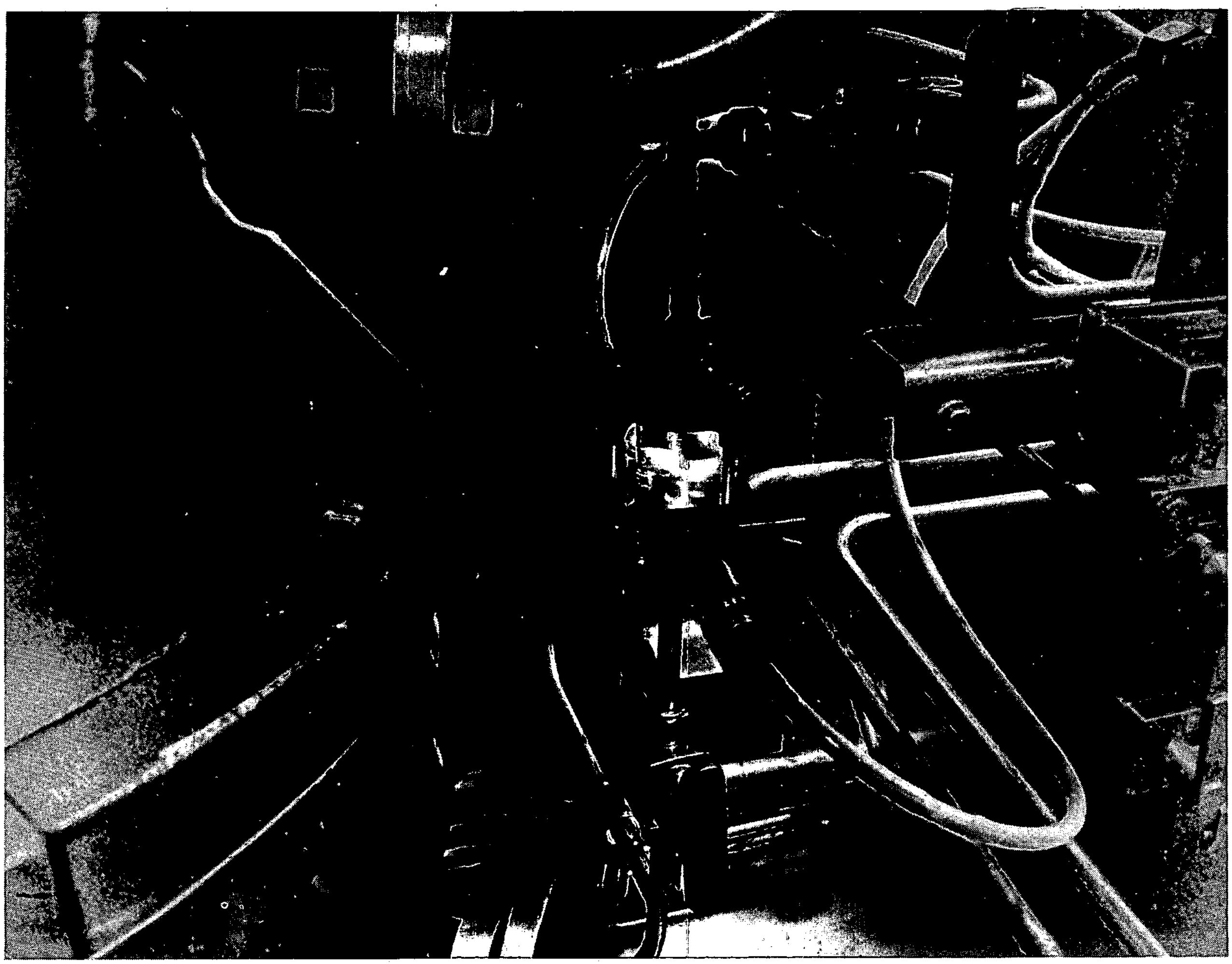

FIGURE 9. Sight-Glass and Anion Exchange Column