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# ON-LINE MONITORING OF PLUTONIUM IN MIXED URANIUM-PLUTONIUM SOLUTIONS

K. J. Hofstetter, T. V. Rebagay, and G. A. Huff*

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## ABSTRACT

The measurement of the total and isotopic plutonium concentrations in mixed uranium-plutonium solutions blended with highly radioactive fission product nuclides and other radionuclides (e.g., Cs-137 and Co-60) has been investigated at the Barnwell Nuclear Fuel Plant (BNFP). An on-line total and isotopic plutonium monitoring system is being tested for its ability to assay the plutonium abundances in solutions as might be found in the process streams of a light water reactor (LWR) spent fuel processing plant. The monitoring system is fully automated and designed to be maintained remotely. It is capable of near real-time inventory of plutonium in process streams and provides the basis for on-line computerized accounting of special nuclear materials.

## INTRODUCTION

Recent concerns over the proliferation of nuclear weapons have stimulated interest in the identification of fuel recycling technologies that offer less proliferation risk than the traditional (conventional) Purex process in which plutonium is separated from uranium and decontaminated from fission products. The coprocessing of plutonium with uranium has been suggested as an alternative. (1, 2, 3) This option is considered to have the advantages of permitting both fuel recycling capabilities and proliferation resistance. A technically feasible coprocessing flowsheet has been proposed which could accomplish these objectives. (4) The incorporation of highly radioactive, gamma-emitting radionuclides into the fuel through incomplete removal of the fission products during reprocessing or by adding spikes to the fuel during fabrication has been suggested as an additional deterrent.

Design concepts (5) for proliferation-resistant fuel alternatives have been developed based on the Barnwell Nuclear Fuel Plant (BNFP) model which was designed for reprocessing light water reactor (LWR) spent fuel using Purex processing technology. Diversion resistance of special

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nuclear materials (uranium and plutonium) can be enhanced by producing a mixed-nitrate coprocessed product that is highly radioactive due to the retention of some fission products. In addition, the coprocessing flowsheet has alternatives for high- and low-fission product decontamination of the mixed nitrates. The low-fission product decontamination alternative is envisioned to increase diversion resistance of the fuel cycle.

This study is focused on the nondestructive assay of plutonium in solutions that might be found in a reprocessing plant using a coprocessing flowsheet. Verification and quantification of plutonium are essential to protect against covert substitution and operating errors as well as for inventory and process control. The nondestructive assay of plutonium is vital to the implementation of a dynamic materials control system, frequently being the only available means for timely measurement of in-process plutonium. Toward this end, an on-line total and isotopic plutonium concentration monitor has been developed and tested for its performance at the BNFP. The on-line monitoring system, which has been previously evaluated in the BNFP laboratory, used highly purified plutonium nitrate solutions of varying concentrations and its performance as a potential safeguard monitoring device has been considered excellent. (6) The present work now addresses the response and operational characteristics of this system to mixed uranium-plutonium solutions in highly radioactive environments. This study may provide a basis for upgrading and improvement of the monitor to accurately measure plutonium in the presence of high radiation fields caused by the incorporation of fission products. The quantitative extent of the effects of this highly radioactive environment on the sensitivity and accuracy of the monitoring system is assessed.

## EXPERIMENTAL

The on-line total and isotopic plutonium concentration monitoring system used in this study has been described in detail elsewhere. (7) This system is capable of near real-time inventory of plutonium and could provide the basis for on-line computerized accounting of the plutonium in the process streams of a light water reactor spent fuel reprocessing plant. (8) Briefly, the system includes a high-resolution lithium-drifted germanium detector, a multichannel pulse-height analyzer, a dedicated minicomputer, and the sample cell assembly. The cell is equipped with a 0.02-inch brass absorber to reduce the background contribution of low-energy gamma and X-rays (E <60 keV) and a built-in Cd-109 source for an overall check of the system performance and stability and for energy calibration. The system is fully automated and designed to be maintained remotely.

The feed and product solutions were formulated based on the composition of LWR spent fuel. (9, 10, 11) Natural and enriched uranium (3.5% U-235) were utilized for the uranium component of the blends while plutonium of typical LWR grade isotopic composition was used. The uranium/plutonium ratio (based on elemental content) was varied from 99/1 for feed compositions to 75/25 for product solutions. Control solutions consisting of highly purified uranium nitrate and plutonium nitrate were also prepared for comparison.

Different levels of fission products and spikes were mixed with the simulated uranium/plutonium coprocessing solutions consistent with the levels suggested for enhanced proliferation resistance without affecting the characteristics of the blends when refabricated as nuclear fuel. The fission products studied were Ru-106/Rh-106, Ce-144/Pr-144, Zr-95/Nb-95, and Ru-103 while the spikes were Co-60 and Cs-137. While Cs-137 is invariably present as a fission product, it is also considered a spike in this study due to its long half-life and availability when compared to the other shorter lived fission products. Table 1 shows the compositions of the blends studied.

Sampling was done by drawing the test solutions into the counting cell of the monitoring system. Since the system was designed for remote control and operation, the radiological safety of the analyst in making the plutonium determinations is assured. Two 1-hour countings of each sample were made and the resulting plutonium values were averaged.

The total and isotopic compositions of plutonium in the different blends were determined by measuring the absolute intensities of the gamma rays characteristics of the Pu-238, Pu-239, and Pu-241 by direct gamma-ray spectroscopy and computer analysis of the spectral data. (12) Plutonium gamma rays in the energy range of 120 to 250 keV were analyzed (129.3 keV peak for Pu-239, 152.7 keV peak for Pu-238, and 148.6 keV peak for Pu-241; the 208.0 keV peak due to U-237 daughter of Pu-241 was also analyzed in the case of "aged" plutonium) while the 185.6 keV gamma line due to U-235 was used for the detection of uranium. To determine the extent of spectral interference of fission products and spikes on the determination of plutonium, the control solutions were analyzed under parallel conditions as the test solutions. Figures 1 through 3 show some representative gamma-ray spectra of the test solutions and control solutions obtained in the energy region of interest.

## RESULTS AND DISCUSSION

Coprocessing is technically feasible for any mixture of uranium and plutonium and plays an important role in the development of a proliferation-resistant fuel cycle. The laboratory protocol in this investigation was designed to demonstrate the sensitivity and accuracy of the on-line isotopic concentration monitoring system in measuring plutonium in the presence of high quantities of fission products and other strong gamma-ray emitters. The results of the laboratory experiments were then evaluated to determine its utility as a safeguards and accountability device for a reprocessing plant with a coprocessing flowsheet.

A summary of the data for the response of the monitor to plutonium is presented in Table 2. Included in the table are the plutonium concentrations measured by isotope dilution mass spectrometry and controlled potential coulometry. Two different isotopic batches were studied. The gamma-ray spectra of the test solutions and the control solutions consisting of LWR-grade, highly purified plutonium nitrate and/or uranium nitrate are depicted in Figures 1 through 3.

As can be seen in Figure 1b, the control solution containing plutonium nitrate only (2.08 grams plutonium/liter) has a barely discernible photopeak at 152.7 keV indicating that, at this plutonium concentration, the calculated Pu-238 value would be suspect. It can be concluded that the abundance of Pu-238 in the plutonium nitrate used to formulate the simulated product stream is extremely low. All the other photopeaks (129 keV, 149.6 keV, and 208 keV gamma lines) are well-defined and sufficiently delineated from the background radiation. Similarly, the 185.6 keV gamma line due to U-235 does not have sufficient intensity (Figure 1) for the quantitative assay of uranium although the uranium nitrate used had been enriched with U-235 (3.5% enrichment). The uranium concentration is in the range calculated for complete fuel dissolution. (11) It is interesting to note that the background radiation of the uranium isotopes appears to have no effect on the general shape of the plutonium spectra at the energy region of interest (120 to 250 keV).

The question posed by denaturing plutonium with fission products or other strong gamma-emitting radionuclides is whether these denaturants improve or hinder the accuracy of the nondestructive assay measurement. Thus, our analyses of the various coprocessing solutions denatured by blending with these selected denaturants are focused on the reliability and accuracy of the monitoring system to measure plutonium only. The extent of spectral interference or enhancement, if any, was evaluated for each denaturant to resolve the problem.

All the fission products (Ru-106/Rh-106, Ru-103, Ce-144/Pr-144, and Zr-95/Nb-95) employed as denaturants appear in every step of the nuclear fuel cycle. Each fission product was of known activity and concentration.* Aliquot portions of these radionuclides were measured quantitatively such that their concentrations lie in the range from 0.1 to 5.0 μCi per gram of fissile material required for proliferation deterrence. (13) The use of nominal amounts of ruthenium and cobalt as denaturants is not expected to adversely affect fuel performance. (14) Ruthenium-106 and Ce-144 are not present in sufficient quantities in spent nuclear fuels to produce the maximum radiation dose rate level required for proliferation deterrence, but they have shown very little effect on the refabrication of the coprocessed solution and its fuel

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*Radiochemical Center, Amersham, England.

properties. They also exhibit amenability to reprocessing. (13) Although Zr-95/Nb-95 and Ru-103 are not recommended as denaturants due to their short half-lives, they were included to determine if they may cause difficulty in the nondestructive assay of plutonium if present in coprocessing solutions.

In spite of the undesirability of Cs-137 as a spike due to its low retention (low boiling point) during fuel refabrication and corrosive effects, it was also considered because it is invariably present in partially separated uranium-plutonium mixtures. (10) Cobalt-60, a strong gamma-emitter, has been recommended as a suitable denaturant to reduce proliferation risk. Fission products have limited usefulness as denaturants but Ce-144 and Ru-106 together with Co-60 could provide semilethal dose rates for limited diversion. The effect of adding Np-237 to the coprocessing solution was also evaluated for its use as a heat spike in another proposed alternative fuel cycle. (15)

The analytical evalutions of the sensitivity of the detection system to plutonium isotopes in the presence of the selected fission products and/or denaturants are listed in Table 2 and the gamma-ray spectra corresponding to representative cases of the various blends are shown in Figures 1 through 3. All the spectral analyses of Pu-238, Pu-239, and Pu-241 were conducted in the intermediate energy region (120 to 250 keV) of the plutonium gamma-ray spectra. No similar analysis was done in the higher energy regions for it has been reported that strong gamma-ray emitters used as denaturants could effectively mask the higher energy gamma lines of plutonium (13) rendering the calculated plutonium values meaningless. Further, an earlier investigation, (16) which dealt with the feasibility of determining the isotopic composition of plutonium by gamma-ray spectrometry, revealed that the plutonium photopeaks most seriously affected are those in the 600 to 800 keV region where the intense gamma-rays of Cs-137 and Zr-95 are very close to the Pu-239 photopeaks.

The levels of the background radiations of the test solutions on which the plutonium photopeaks are superimposed are slightly increased, but the general shape of the observed spectra resembles the control spectra (spectra of the solution containing plutonium nitrate only) suggesting that the fission products (except Ce-144) and/or denaturants have no gamma lines or Compton edges (distribution) in the region of analysis. The peak/background ratios differed slightly from the control ratio, but the discrepancies may be attributed in part to counting errors. That the increase in the background radiation did not diminish the sensitivity of the monitor could be deduced from the test peak area/control peak area ratios. The slight enhancements (approximately 10%) of these ratios may be due to other experimental errors in addition to the counting errors. Consequently, no significant loss of accuracy or precision in the plutonium measurements is expected. This is borne by the good agreement of the values obtained by the monitor with those gathered by the chemical methods.

Although it was reported (16) that the 133-keV line of Ce-144 may cause serious interference if present in any quantity, our results indicated that the amount of Ce-144 added to the coprocessing solutions (which is substantial) did not cause any interference in the analysis of the nearby plutonium photopeaks (129.3-keV line of Pu-239 and 148.6-keV line of Pu-241). The closest plutonium photopeak, the 129.3-keV Pu-239 photopeak, is clearly distinct and well separated from the 133-keV Ce-144 photopeak. The integrated absolute intensity of this line was not very different from the absolute intensity of the 129-keV line of the plutonium control solution.

As mentioned earlier, the 152.7-keV photopeak of Pu-238 does not possess sufficient intensity for accurate evaluation in the case of the simulated product solution of the coprocessing technology. However, this photopeak can be resolved without difficulty in the simulated feed solutions (>1.5% plutonium). Hence, the Pu-238 contents of the feed solutions are given.

The investigation showed that at the levels of fission products and/or denaturants added to the uranium-plutonium coprocessing feed and product solutions, no spectral interference could be detected from these proliferation deterrents in the nondestructive direct gamma-ray spectroscopy of plutonium. It clearly demonstrated the usefulness of the on-line isotopic concentration monitor to assay accurately small quantities of plutonium even in highly radioactive environments. This evaluation of the system's performance may help in design concepts aimed at determining the detection pattern in plutonium-bearing streams of a reprocessing plant with a coprocessing flowsheet.

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## TABLE 1

## COMPOSITIONS OF FEED AND PRODUCT SOLUTIONS IN URANIUM-PLUTONIUM COPROCESSING OF LWR FUEL

| Materials | Feed | | Product | |
|---|---|---|---|---|
| | A | B | A | B |
| Pu (g/L) | 4.4 | 2.08 | 50.0 | 1.04 |
| U (g/L) | 393.3 | 192.0 | 139.3 | 64.0 |
| Np (g/L) | 0.54 | 0.54 | 0.18 | 0.18 |
| Cs-137 (mCi/L) | 0.829 | 0.37 | 0.64 | 0.64 |
| Co-60 (mCi/L) | 0.176 | 0.146 | 0.878 | 0.878 |
| Ru-106/Rh-106 (mCi/L) | 3.53 | 0.88 | 0.011 | 0.011 |
| Ce-144/Pr-144 (mCi/L) | 4.03 | 1.08 | 2.93 | 2.93 |
| $HNO_3$, M | 3 | 3 | 0.3 | 0.3 |
| Uranium/Plutonium,* Approximate | 99:1 | 99:1 | 75:25 | 98:2 |

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*Based on total metal content.

## TABLE 2

## PLUTONIUM ABUNDANCES IN URANIUM-PLUTONIUM COPROCESSING SOLUTIONS

| | Total Plutonium, g/l | | |
|---|---|---|---|
| | Method of Analysis | | |
| Sample | This Work | Mass Spectrometry | Dilution** |
| 1. Product Control (aged) | 0.99 | 1.401 | 1.50 |
| 2. Product Solution + Ru-106 | 0.94 | 0.951 | 0.87 |
| 3. Product Solution + Co-60 | 1.10 | 0.982 | 0.87 |
| 4. Product Solution + Np-237 | 0.90 | 1.157 | 1.02 |
| 5. Product Solution + Ce-144 | 1.06 | 1.132 | 1.02 |
| 6. Product Solution + Cs-137 | 1.00 | 0.971 | 0.87 |
| 7. Composite (aged) | 1.21 | 1.959 | 1.67 |
| 8. Product Solution | 0.93 | 1.145 | 1.04 |
| 9. Product Solution + Ce-144 | 1.04 | 1.089 | 0.93 |
| 10. Product Control + Ce-144 | 1.14 | 1.178 | 0.98 |
| 11. Feed Solution + Ce-144 | 1.27 | 2.787 | 3.00 |
| 12. Feed Solution + Ru-106 | 1.20 | 1.990 | 1.73 |
| 13. Feed Solution (aged)* | -- | 4.191 | 4.41 |
| 14. Feed Solution (aged)* | -- | 3.940 | 4.28 |
| 15. Feed Solution + Ce-144 | 1.08 | 1.596 | 1.58 |
| 16. Feed Control + Ce-144 | 1.09 | 1.286 | 1.73 |

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*Solutions used for calibration.

**Stock solutions analyzed by controlled potential coulometry.

## TABLE 3

## THE EFFECTS OF DENATURIZATION ON THE RESPONSE OF THE ON-LINE MONITORING SYSTEM TO PLUTONIUM

Plutonium Photopeaks Analyzer*

| Solutions | Pu-239 (129 keV) | | Pu-238 (152 keV) | | Pu-241 (148 keV) | | Pu-241-U-237 (208 keV) | |
|---|---|---|---|---|---|---|---|---|
| | S/B | S/C | S/B | S/C | S/B | S/C | S/B | S/C |
| | Feed Solution (2.08 g/L Pu) | | | | | | | |
| Feed Only | 3.56 | | 2.06 | | 3.80 | | 16.26 | |
| Feed + Np-237 | 2.88 | 1.08 | 1.37 | 0.81 | 3.38 | 1.18 | 14.0 | 1.01 |
| Feed + Co-60 | 2.18 | 1.11 | 1.28 | 1.08 | 2.94 | 1.05 | 12.2 | 0.94 |
| Feed + Co-137 | 2.48 | 0.99 | 1.54 | 0.87 | 3.99 | 0.98 | 17.9 | 1.07 |
| Feed + Ru-106 | 2.76 | 1.08 | 1.62 | 0.87 | 4.47 | 0.98 | 15.4 | 1.03 |
| Feed + Ce-144 | 2.85 | 1.11 | 1.64 | 1.08 | 4.63 | 0.92 | 17.3 | 0.97 |
| Composite | 2.13 | 0.96 | 1.30 | 1.08 | 4.22 | 1.11 | 16.5 | 1.04 |
| | Product Solution (1.04 g/L Pu) | | | | | | | |
| Product Only | 2.95 | | 1.42 | | 3.59 | | 16.9 | |
| Product + Np-237 | 2.83 | 1.05 | 1.78 | 1.44 | 2.90 | 1.02 | 14.4 | 1.04 |
| Product + Co-60 | 2.66 | 1.07 | 1.59 | 1.00 | 3.35 | 1.00 | 13.8 | 1.11 |
| Product + Cs-137 | 2.10 | 1.04 | 1.40 | 0.57 | 2.25 | 1.00 | 4.05 | 0.98 |
| Product + Ru-106 | 2.68 | 1.04 | 1.38 | 0.30 | 3.43 | 1.00 | 15.4 | 1.06 |
| Product + Ce-144 | 2.09 | 1.02 | 1.49 | 0.98 | 2.72 | 1.13 | 8.36 | 1.06 |
| Composite | 2.14 | 1.14 | 1.18 | 1.17 | 3.25 | 1.23 | 14.3 | 1.24 |

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*S/B represents the ratio of the sample peak counts to the background counts, and S/C is the ratio of the integrated area of the sample photopeak to the integrated area of the control.

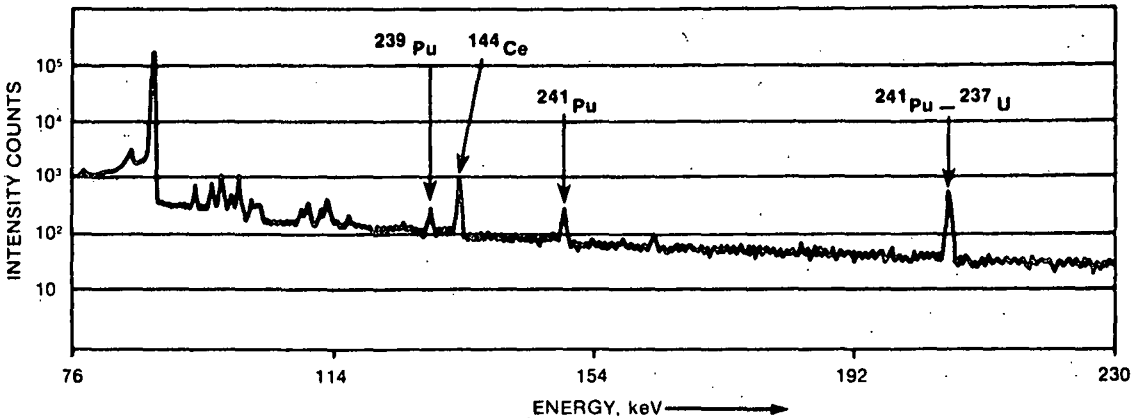

Figure 1A
**Feed Solution**

Solution Composition: Freshly Separated Pu, enriched natural uranium, Np-237, Cs-137, Ru-103, Ru-106/Rh-106, Ce-144/Pr-144, Co-60, and Zr-95/Nb-95 in 3M $HNO_3$

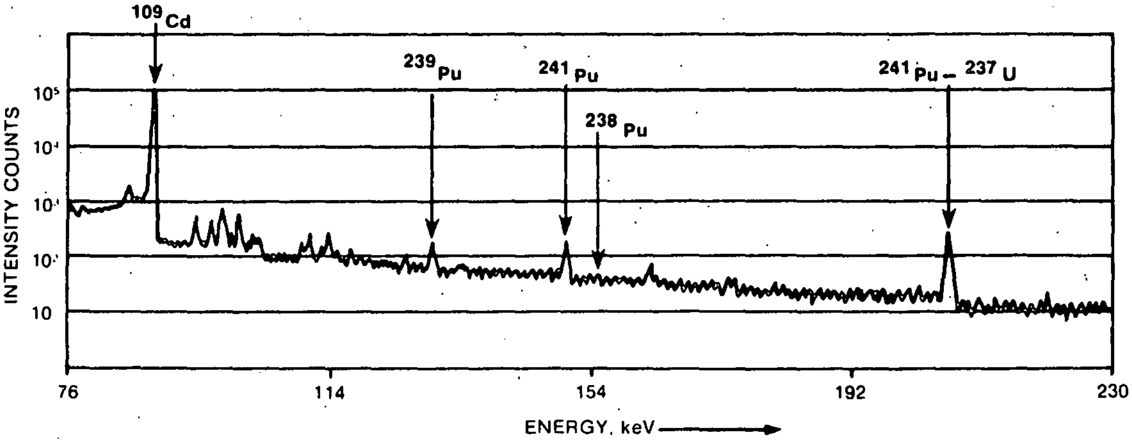

Figure 1B
**Feed Control Solution**

Solution Composition: Freshly Separated Pu (2.08 g/L) in 3M $HNO_3$

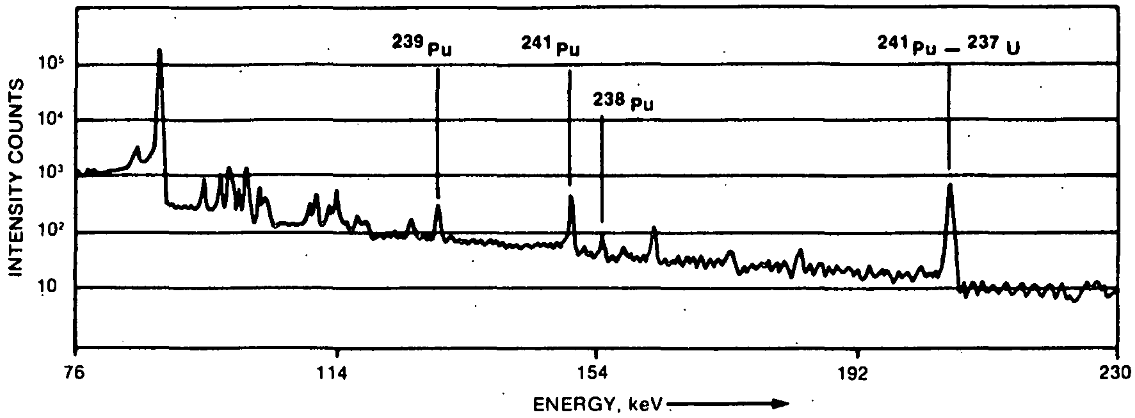

Figure 2A
**Plutonium Control Solution (1% Pu, No U)**

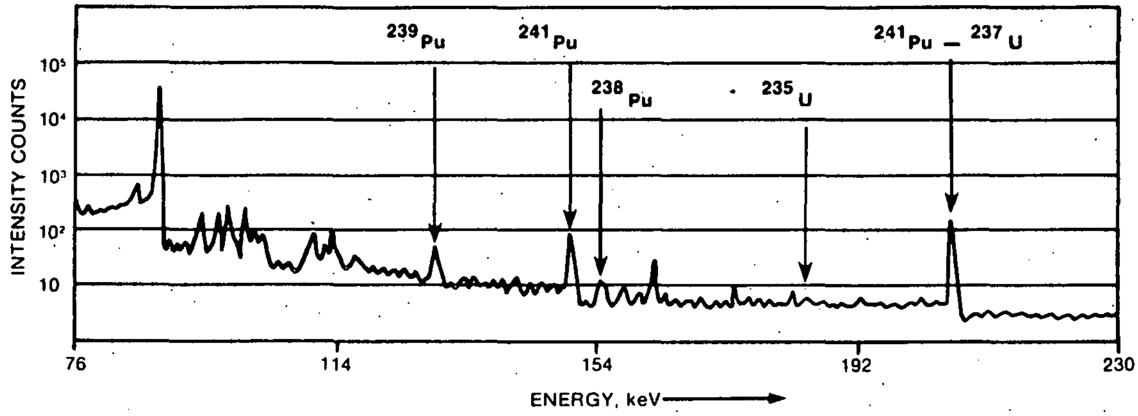

Figure 2B
**Uranium (99%) - Plutonium (1%) Solution**

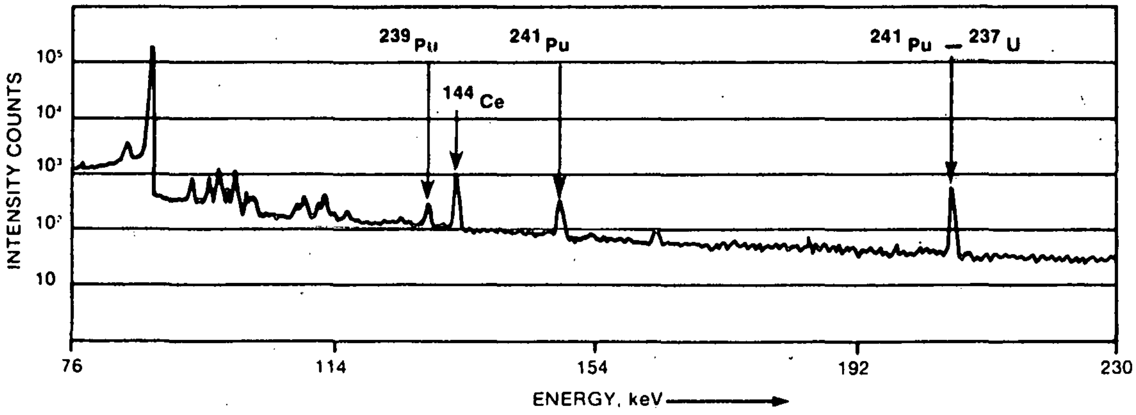

Figure 2C
**Feed Solution**

Solution Composition: "Aged" Pu (3.0 g/L), natural U, Np-237, Co-60, Cs-137, Ce-144/Pr-144, Ru-103, Ru-106/Rh-106, Zr-95/Nb-95

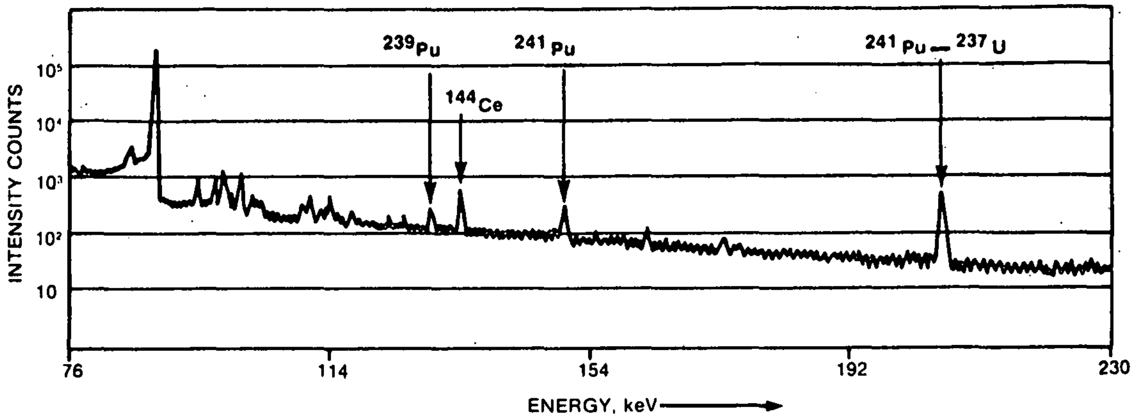

Figure 3A
**Control Solution with Denaturants**

Solution Composition: Pu (1.0 g/L), Np-237, Co-60, Cs-137, Ru-103, Ru-106/Rh-106, Zr-95/Nb-95, in 3M $HNO_3$ - no uranium added.

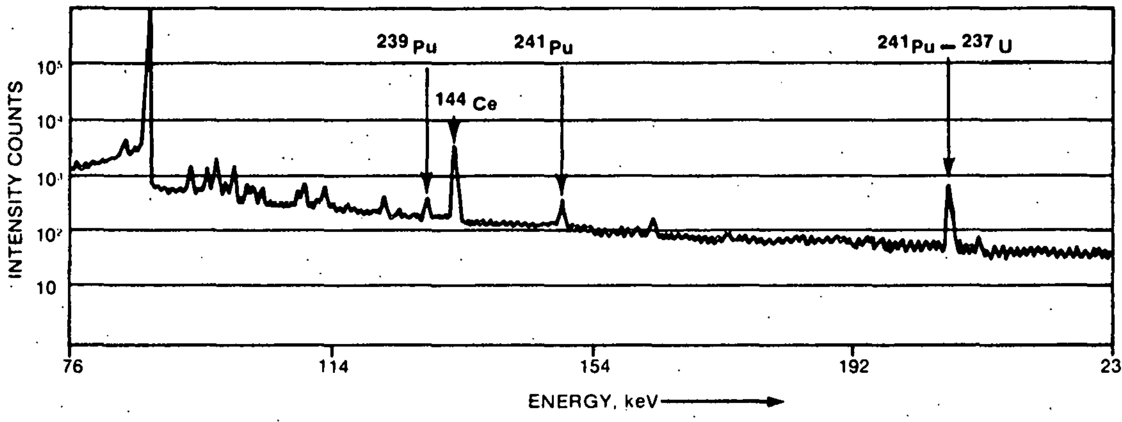

Figure 3B
**Product Solution**

Solution Composition: Pu (1.0 g/L), U(64 g/L), Np-237, Co-60, Cs-137, Ru-103, Ru-106/Rh-106, Zr-95/Nb-95, in 3M $HNO_3$

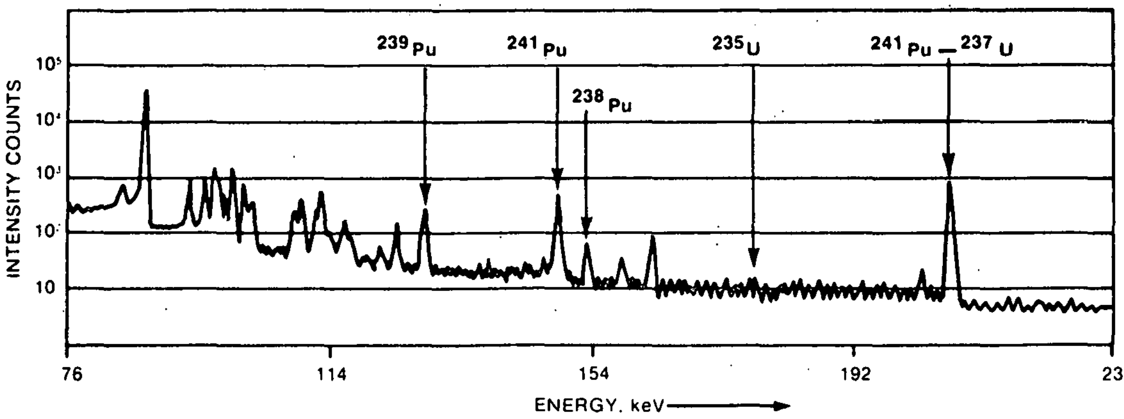

Figure 3C
**Product Solution: Uranium (75%) - Plutonium (25%)**

# TABLE 3

## THE EFFECTS OF DENATURIZATION ON THE RESPONSE OF THE ON-LINE MONITORING SYSTEM TO PLUTONIUM

Plutonium Photopeaks Analyzer*

| Solutions | Pu-239 (129 keV) | | Pu-238 (152 keV) | | Pu-241 (148 keV) | | Pu-241-U-237 (208 keV) | |
|---|---|---|---|---|---|---|---|---|
| | S/B | S/C | S/B | S/C | S/B | S/C | S/B | S/C |
| | Feed Solution (2.08 g/L Pu) | | | | | | | |
| Feed Only | 3.56 | | 2.06 | | 3.80 | | 16.26 | |
| Feed + Np-237 | 2.88 | 1.08 | 1.37 | 0.81 | 3.38 | 1.18 | 14.0 | 1.01 |
| Feed + Co-60 | 2.18 | 1.11 | 1.28 | 1.08 | 2.94 | 1.05 | 12.2 | 0.94 |
| Feed + Co-137 | 2.48 | 0.99 | 1.54 | 0.87 | 3.99 | 0.98 | 17.9 | 1.07 |
| Feed + Ru-106 | 2.76 | 1.08 | 1.62 | 0.87 | 4.47 | 0.98 | 15.4 | 1.03 |
| Feed + Ce-144 | 2.85 | 1.11 | 1.64 | 1.08 | 4.63 | 0.92 | 17.3 | 0.97 |
| Composite | 2.13 | 0.96 | 1.30 | 1.08 | 4.22 | 1.11 | 16.5 | 1.04 |
| | Product Solution (1.04 g/L Pu) | | | | | | | |
| Product Only | 2.95 | | 1.42 | | 3.59 | | 16.9 | |
| Product + Np-237 | 2.83 | 1.05 | 1.78 | 1.44 | 2.90 | 1.02 | 14.4 | 1.04 |
| Product + Co-60 | 2.66 | 1.07 | 1.59 | 1.00 | 3.35 | 1.00 | 13.8 | 1.11 |
| Product + Cs-137 | 2.10 | 1.04 | 1.40 | 0.57 | 2.25 | 1.00 | 4.05 | 0.98 |
| Product + Ru-106 | 2.68 | 1.04 | 1.38 | 0.30 | 3.43 | 1.00 | 15.4 | 1.06 |
| Product + Ce-144 | 2.09 | 1.02 | 1.49 | 0.98 | 2.72 | 1.13 | 8.36 | 1.06 |
| Composite | 2.14 | 1.14 | 1.18 | 1.17 | 3.25 | 1.23 | 14.3 | 1.24 |

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*S/B represents the ratio of the sample peak counts to the background counts, and S/C is the ratio of the integrated area of the sample photopeak to the integrated area of the control.

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