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ANALYSIS OF E-N LOADINGS

AUTHOR

W. H. ZIMMER

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ANALYSIS OF E-N LOADINGS

INTRODUCTION

Three E-N loaded tubes were dissolved, sampled and analyzed, starting November 9, 1960. The results of these analyses and an explanation of the methods used are the subject of this report.

RESULTS

Each tube loading received an identification code in each facility in which it was processed. All of these codes are listed for future reference.

DISCUSSION

Each batch of slugs was dissolved in a preflushed dissolver. When complete solution was indicated by a leveling off of the specific gravity, two 1 ml samples were taken and analyzed for uranium, specific gravity, and excess nitric acid. The dissolver charge was digested an additional four hours.

At the end of the digestion period, two 1 ml samples and one 20 ml pig sample were taken. The three samples were analyzed for U, SpG, and HNO₃. Agreement between these samples and the previous samples was taken as confirmation of complete dissolution and representative sampling. If agreement was not obtained, sampling was continued.

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After verification of the pig sample, six 1 ml aliquots were taken for analysis by the Analytical Control group. The remainder was aliquoted to provide material for mass analysis and for analysis by the Process Chemistry group.

The data recorded was obtain in the following manner:

ANALYTICAL CONTROL GROUP

lbs. U/gal - X-ray Photometer - six duplicate determinations

g Pu/gal - Direct mount, counted on ASP - six determinations of two dilutions and four disks. Uranium contribution calculated from X-ray data.

AmCm contribution determined by method AMCM-1b (extraction and ASP counting method). Specific alpha activity of the Pu was determined from the formula:

$$S.A = \frac{(wt.\% Pu^{239} \times 1.363 + wt.\% Pu^{240} \times 5.025)}{100\% - \alpha \% Pu^{238}} \times 10^{11} = d/m/g Pu$$

Pu²³⁹ and Pu²⁴⁰ weight per cents were obtained by thermal emission, mass analysis of purified Pu. Pu²³⁸ alpha per cent was determined by extracting Pu with TTA and alpha energy analyzing.

g Am²⁴¹/gal - TBP-TTA extraction and ASP count. Three determinations of three disks. Uranium is separated by TBP extraction and Pu by TTA extraction. Ratio of Am²⁴¹ to Cm²⁴² was determined by alpha energy analysis of Process Chemistry group's separation.

g Cm²⁴² - See above

Alpha per cent Pu²³⁸ - TTA extraction of Pu, ASP counting and alpha energy analysis - Three determinations of two dilutions and four disks.

PROCESS CHEMISTRY GROUP

lbs U/gal - Constant potential, coulometric titration - two determinations.

g Pu/gal - As in analytical Control Group with two exceptions. a) Uranium contribution calculated from coulometric data and b) Pu²³⁸ contribution determined from resin separated Pu that had been alpha energy analyzed.

Coulemetric analysis for Pu in the resin elution was attempted but results were inconclusive.

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* Process Chemistry Analysis omitted because of small sample available.

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$\text{g Am}^{241}/\text{gal}$ Resin column effluent from two aliquots was salted with concentrated HNO_3 and extracted into 30% TBP in Shell Spray Base to remove uranium. Pu remained on the resin. The AmCm - F.P. mixture was counted on an ASP and alpha energy analyzed.

$\text{g Cm}^{242}/\text{gal}$ See above

Pu and U
purification
for mass
analysis Four 1 ml aliquots of dissolver solution were treated with hydroxylamine and sodium nitrite to adjust the valence of the plutonium to Pu(IV). The solutions were run through Dowex 1 - 8 X resin (100-200 mesh) at 50 C. The plutonium retained on the resin is eluted with 0.5 M HNO_3 and 0.02 M NH_2OH and concentrated.

The column effluent is concentrated. The uranium is extracted in a Hexone system using aluminum nitrate as a salting agent. Decontamination is achieved by repeatedly scrubbing the Hexone phase with saturated ANN and Dibane. The uranium is stripped into water, ozonated and fused to U_3O_8 .

H. H. Zimmer

Process Chemistry
CHEMICAL PROCESSING DEPARTMENT

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