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TITLE

ANALYSIS OF THE TWELVE TWO-TON TEST BATCHES FOR THE REVIEW OF PLUTONIUM FORMATION RATES AS A FUNCTION OF MWD.

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ANALYSIS OF THE TWELVE TWO-TON TEST BATCHES FOR THE REVIEW OF PLUTONIUM FORMATION RATES AS A FUNCTION OF MWD

by

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ANALYSIS OF THE TWELVE TWO-TON TEST BATCHES FOR THE REVIEW OF PLUTONIUM FORMATION RATES AS A FUNCTION OF MWD

INTRODUCTION

HW-64886-RD, "Status of the Twelve Two-Ton Test Batches for the Review of Plutonium Formation Rates as a Function of MWD," Secret, April 29, 1960, D. W. Hoba(1), describes the program for which this document supplies the analytical results. A discussion of analytical methods and precision follows the tabulated results.

The twelve test batches were individually dissolved in preflushed Redox dissolvers. The flush solutions were fluorimetrically analyzed for uranium and in all cases reported to contain less than 1×10^{-3} pounds of U/gal.

Dissolution of each test batch was continued until the control board specific gravity reached a constant value. Duplicate one-ml doorstop samples were taken from the dissolver and analyzed for SpG, HNO_3, and uranium. Digestion of the dissolver solution continued for an additional four hours. Sampling and analyzing were repeated and agreement among the four samples was taken as proof of complete dissolution. At this time, six one-ml doorstop samples and one twenty-ml pig sample were taken to provide analytical samples. These samples were required to agree in specific gravity within ± 0.006 g/cc before they were accepted as representative of the dissolver solutions.

The twenty-ml pig sample provided test solutions from which uranium and plutonium were separated and purified for the determination of isotopic distribution. It was also the source of the Am^{241}, Cm^{242} distribution, where reported. All other reported results were obtained from analysis of the six one-ml doorstop samples.

Each test batch received an identification code at each of the facilities in which it was processed. All of these codes are listed with their associate facility to avoid confusion.

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RESULTS

Sample Identification Codes

	Batch No. 1 H-Reactor	Batch No. 2 H-Reactor	Batch No. 3 H-Reactor
100 Area			
202-S	C-2 run H-20	A-2 run H-15	C-2 run H-1
222-S - Anal. Chem.	6994X 7025X 7028X 7029X 7042X 7043X	6638X 6639X 6651X 6652X 6653X 6654X	7161X 7162X 7163X 7164X 7165X 7166X
222-S - Proc. Chem.	7000X 11AC	6644X 6AC	7152X 12AC
Dissolver Sampling Date	8-1-60	7-22-60	8-5-60
lbs U/gal	3.775 ± 0.010	4.025 ± 0.030	4.260 ± 0.061
g Pu/gal	0.7321	0.7971	0.8334
g Pu/lb U	0.1939 ± 0.0016	0.1980 ± 0.0030	0.1956 ± 0.0009
lb U charged(1)	4035.264	4033.258	4019.267
MWD(1)	882.70	894.49	894.51
g Pu/MWD	0.8864	0.8928	0.8789
c/m/gal alpha total	$(5.636 \pm 0.049) \times 10^{10}$	$(6.135 \pm 0.102) \times 10^{10}$	$(6.429 \pm 0.063) \times 10^{10}$
c/m/gal AmCm	4.30×10^{8}	4.07×10^{8}	4.77×10^{8}
"F" factor (g Pu/c/m)	1.309×10^{-11}	1.308×10^{-11}	1.306×10^{-11}
alpha per cent Pu^{238}	1.1	1.4	1.1
weight per cent Pu^{238}	0.004	0.006	0.004
Pu^{239}	95.66 ± 0.08	95.59 ± 0.15	95.60 ± 0.37
Pu^{240}	4.11 ± 0.06	4.06 ± 0.15	4.21 ± 0.35
Pu^{241}	0.22 ± 0.04	0.35 ± 0.03	0.19 ± 0.06
Pu^{242}	0.01	<0.01	<0.01
weight per cent U^{235}	0.675 ± 0.005	0.6505 ± 0.002	0.662 ± 0.002
g/gal Am^{241}			
g/gal Cm^{242}			
g/gal Np	2.4×10^{-3}	2.3×10^{-3}	3.4×10^{-3}

Sample Identification Codes

100 Area	Batch No. 4 H - Reactor	Batch No. 5 H - Reactor	Batch No. 6 H - Reactor
202-S	C-2 run H-12	C-2 run H-11	A-2 run H-10
222-S Anal. Chem.	6517X 6518X 6519X 6520X 6521X 6526X	6421X 6422X 6423X 6424X 6425X 6426X	6462X 6463X 6464X 6465X 6566X 6467X
222-S - Proc. Chem.	6530X 3AC	H-11 2AC	6479X 1AC
Dissolver Sampling Date	7-18-60	7-14-60	7-15-60
lbs U/gal	3.713 ± 0.067	3.895 ± 0.050	3.281 ± 0.018
g Pu/gal	1.224	1.288	1.111
g Pu/lb U	0.3297 ± 0.0058	0.3307 ± 0.0060	0.3386 ± 0.0024
lb U charged(1)	4036.586	4038.707	4003.971
MWD(1)	1608.28	1620.09	1630.74
g Pu/MWD	0.8275	0.8244	0.8314
c/m/gal alpha total	$(1.027 \pm 0.008) \times 10^{11}$	$(1.096 \pm 0.019) \times 10^{11}$	$(9.369 \pm 0.101) \times 10^{10}$
c/m/gal AmCm	2.58×10^{9}	3.07×10^{9}	2.43×10^{9}
"F" factor (g Pu/c/m)	1.223×10^{-11}	1.209×10^{-11}	1.217×10^{-11}
alpha per cent Pu^{238}	2.6	2.8	2.4
weight per cent Pu^{238}	0.011	0.012	0.010
Pu^{239}	92.80 ± 0.20	92.35 ± 0.21	92.50 ± 0.06
Pu^{240}	6.54 ± 0.20	6.97 ± 0.20	6.85 ± 0.06
Pu^{241}	0.66 ± 0.04	0.68 ± 0.06	0.65 ± 0.03
Pu^{242}	<0.01	<0.01	<0.01
weight per cent U^{235}	0.6281 ± 0.001	0.6266 ± 0.001	0.6275 ± 0.001
g/gal Am^{241}	4.2×10^{-4}	5.7×10^{-4}	4.0×10^{-4}
g/gal Cm^{242}	2.9×10^{-7}	2.8×10^{-7}	2.7×10^{-7}
g/gal Np	4.1×10^{-3}	4.7×10^{-3}	4.4×10^{-3}

RESULTS (Cont.)

Sample Identification Codes

100 Area	Batch No. 7 KW-Reactor	Batch No. 8 KW-Reactor	Batch No. 9 KW-Reactor
202-S	A-2 run KW-19	C-2 run KW-18	A-2 run KW-17
222-S - Anal. Chem.	6968X 6970X 6971X 6972X 7015X 7017X	6865X 6867X 6870X 6887X 6889X 6902X	6779X 6780X 6781X 6782X 6793X 6794X
222-S - Proc. Chem.	7021X 10AC	6872X 9AC	6762X 8AC
Dissolver Sampling Date	7-31-60	7-29-60	7-25-60
lbs U/gal	3.893 ± 0.007	3.774 ± 0.037	3.744 ± 0.016
g Pu/gal	0.8324	0.8143	0.8221
g Pu/lb U	0.2138 ± 0.0016	0.2158 ± 0.0025	0.2196 ± 0.0016
lb U charged(1)	4151.219	4150.991	4133.180
MWD(1)	947.62	949.21	948.36
g Pu/MWD	0.9366	0.9437	0.9571
c/m/gal alpha total	$(6.400 \pm 0.048) \times 10^{10}$	$(6.319 \pm 0.104) \times 10^{10}$	$(6.327 \pm 0.027) \times 10^{10}$
c/m/gal AmCm	5.10×10^{8}	5.01×10^{8}	4.67×10^{8}
"F" factor (g Pu/c/m)	1.311×10^{-11}	1.299×10^{-11}	1.309×10^{-11}
alpha per cent Pu^{238}	1.2	1.2	1.3
weight per cent Pu^{238}	0.005	0.005	0.005
Pu^{239}	95.80 ± 0.13	95.12 ± 0.22	95.54 ± 0.35
Pu^{240}	4.00 ± 0.11	4.48 ± 0.21	4.10 ± 0.35
Pu^{241}	0.20 ± 0.02	0.40 ± 0.02	0.36 ± 0.04
Pu^{242}	<0.01	<0.01	<0.01
weight per cent U^{235}	0.677 ± 0.003	0.6556 ± 0.002	0.6564 ± 0.001
g/gal Am^{241}			1.1×10^{-5}
g/gal Cm^{242}			2.4×10^{-8}
g/gal Np	2.8×10^{-3}	2.8×10^{-3}	2.7×10^{-3}

RESULTS (Cont.)

Sample Identification Codes

	Batch No. 10 KW-Reactor	Batch No. 11 KW-Reactor	Batch No. 12 KW-Reactor
100 Area			
202-S	C-2 run KW-14	A-2 run KW-13	C-2 run KW-16
222-S Anal. Chem.	6598X 6615X 6616X 6617X 6635X 6637X	6536X 6537X 6538X 6539X 6540X 6541X	6737X 6738X 6741X 6742X 6745X 6755X
222-S - Proc. Chem.	6604X 5AC	6542X 4AC	6743X 7AC
Dissolver Sampling Date	7-20-60	7-18-60	7-24-60
lbs U/gal	3.615 ± 0.024	3.480 ± 0.022	3.753 ± 0.028
g Pu/gal	1.323	1.263	
g Pu/lb U	0.3660 ± 0.0021	0.3629 ± 0.0057	
lb U charged(1)	4152.439	4142.327	4150.398
MWD(1)	1723.28	1727.19	1734.33
g Pu/MWD	0.8819	0.8703	
c/m/gal alpha total	$(1.134 \pm 0.008) \times 10^{11}$	$(1.075 \pm 0.015) \times 10^{11}$	$(1.189 \pm 0.015) \times 10^{11}$
c/m/gal AmCm	3.54×10^{9}	2.91×10^{9}	3.34×10^{9}
"F" factor (g Pu/c/m)	1.204×10^{-11}	1.208×10^{-11}	
alpha per cent Pu^{238}	2.7	3.0	
weight per cent Pu^{238}	0.012	0.013	
Pu^{239}	92.04 ± 0.20	92.44 ± 0.15	
Pu^{240}	7.20 ± 0.20	6.88 ± 0.15	
Pu^{241}	0.76 ± 0.05	0.68 ± 0.05	
Pu^{242}	<0.01	<0.01	
weight per cent U^{235}	0.6236 ± 0.001	0.6261 ± 0.001	
g/gal Am^{241}	5.9×10^{-4}	4.8×10^{-4}	
g/gal Cm^{242}	3.9×10^{-7}	3.2×10^{-7}	
g/gal Np	5.2×10^{-3}	4.9×10^{-3}	5.3×10^{-3}

DISCUSSION OF RESULTS

Precision

All tabulated precision values are stated at the 95% confidence level.

Lbs U/gal

UX-4a - The uranium content of two aliquots from each of six samplings of the test batch was extracted into 12.5% TBP. The X-ray attenuation of the organic phase is determined in an X-ray photometer that has been calibrated by the criss-cross method. Quality control and referee standards are routinely run with this method. In the calculation of the precision, n equals six.

g/gal Pu - (alpha total c/m/gal - AmCm c/m/gal) x "F" factor

g Pu/lb U - g Pu/gal was determined for each of the six samples of the test batch by using the alpha total c/m/gal of each, minus the average AmCm c/m/gal times the "F" factor. The lbs U/gal used was the average from each of the six samplings in the test batch. Pu to U ratios were calculated individually for each of the six samples in a test batch to minimize the variation due to sampling. Precision was calculated using n equals six.

Lbs U charged - as in HW-64886-RD[1]

MWD - as in HW-64886-RD[1]

g Pu/MWD - (g Pu/lbs U x lbs U charged[1]) ÷ MWD[1]

Alpha total c/m/gal

PuA-6b - A total of 24 direct disk mounts were counted in an ASP (Alpha Simpson Proportional Counter) to determine the total alpha counting rate. Two dilutions were prepared from each test batch sampling (6) and two disks were prepared from each dilution. Quality control and referee standards are routinely run with this method. Each alpha total count is corrected to a 50% counting geometry and corrected for alpha count due to uranium. Precision was calculated using n equals six.

AmCm c/m/gal

AmCm-1b - A total of 12 disk mounts (two dilutions and four disks on three doorstop samples) were prepared for the determination of the alpha counting rate due to AmCm. Plutonium and uranium are extracted from the test solution with TTA and TBP, leaving AmCm as the only alpha emitters. Precision is not listed but control standards indicate a standard deviation of ± 20% for the method.

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"F" factor (g Pu/c/m) - $\frac{(100\% - \text{alpha \% } Pu^{238}) \times 2}{(\text{wt.\% } Pu^{239} \times 1.363 + \text{wt.\% } Pu^{240} \times 5.025) \times 10^{11}}$

or $\frac{2 \text{ (d/m/c/m)}}{\text{Specific activity (c/m/g)}}$

$2\ ^{d/m}/_{c/m}$ is included to allow direct application of the "F" factor to the alpha counting rate (corrected to a 50% geometry). Alpha per cent Pu^{238} is determined from alpha energy analysis data. The weight per cent of Pu^{239} and Pu^{240} are determined by thermal emission, mass spectrometry. The contribution of Pu^{241} and Pu^{242} to the total alpha count is not significant. 1.363×10^{11} and 5.025×10^{11} are, respectively, the specific activities of Pu^{239} and Pu^{240} as determined by the equation:

$$\text{S.A.} = \frac{\text{Avagado's No.} \times 0.693}{\text{Atomic Wt.} \times T_{1/2} \text{ (min.)}}$$

Alpha Per Cent Pu^{238}

Plutonium was extracted into TTA from six aliquots in each test batch. One mounted disk from each aliquot was alpha energy analyzed in a Frish grid chamber. Standard deviation for the method is approximately ±0.2 wt.% Pu.

$$\text{Wt. \% } Pu^{238} - \frac{(\text{alpha total c/m/gal} - \text{AMCM c/m/gal}) \times \text{alpha \% } Pu^{238} \times 2}{\text{Specific alpha activity of } Pu^{238} \times \text{g Pu/gal}}$$

Weight Per Cent Pu^{239}, Pu^{240}, and Pu^{242}

Each test batch was processed through Dowex 1 resin to separate plutonium from all other constituents. The eluted plutonium solution was analyzed for isotopic distribution with a thermal emission, mass spectrometer.

Weight Per Cent U^{235}

The effluent material from the resin separation of plutonium was extracted into hexone. Uranium decontamination from F.P's was accomplished by repeatedly washing the hexone phase with saturated ANN and Dibane. The uranium was stripped into aqueous solution, ozonated to remove residual ruthenium and finally fused to from U_3O_8. The isotopic distribution was determined with a mass spectrometer.

g/gal Am^{241} and g/gal Cm^{241}

Two aliquots of the effluent material from the resin separation of plutonium were extracted into 30% TBP in a high nitric acid system. The aqueous phase, now free of Pu and U and partially decontaminated, was alpha energy analyzed.

$$\text{g/gal } Am^{241} = \frac{\text{c/m/gal AmCm} \times \text{alpha \% } Am^{241} \times 2}{100 \times (\text{Specific Activity of } Am^{241})}$$

[redacted]

g/gal Np

Neptunium is separated from plutonium and uranium by extracting into TTA and stripping into HNO_3. Six aliquots from each test batch were treated and alpha counted on an ASP.[3] Half of the mounted disks were alpha energy analyzed to determine the completeness of the separation of neptunium from other alpha emitters. Appropriate corrections were applied to the alpha counting rates.

$$\frac{2 \times \text{corrected c/m/gal Np}}{\text{Specific activity Np}} = \text{g/gal Np}$$

Precision was poor because of the extremely low neptunium concentration.

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