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SYNOPSIS OF TALK ON BEAD LEACH I ANALYSES

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Page

Slide 1: Status of Bead Leach I Analyses as of 5/17/79.

All samples of the bicarbonate and distilled water have been prepared and counted through day 120. 100 λ of each sample (channels 10 - 28 for each sample day) was evaporated on a Pt disc, fired in a Meeker burner, and α - counted. Since leach rates generally decrease with time with accompanying decreases in actinide content in the leachate, count times must be increased with the longer time samples. We suspect that for samples collected on days 230 and 420, radiochemical purification of 10 mls will be required rather than simple evaporation of 100 λ . Also, twice as many α - countings are required since Np and Pu are separate samples after radiochemical purification. After evaporations, both actinides can be counted on the same Pt disc.

The radiochemical purification procedure for the brine samples (channels 1 - 9) have been completed through day 120. The α - counting for both Np and Pu has been completed through day 68.

No ICP analyses have been done on leachate samples. However, calibration studies have been finished in preparation for leachate analyses.

Slide 2: Composition of 76 - 68 Glass and Detection Limits for ICP Analysis of Leachate.

This slide shows the elements which the ICP- emission spectroscopy equipment at LLL can observe in water samples and the composition of 76 - 68 glass. Those elements in common are denoted with an arrow. Analyses for Zr is currently incorrect but the analyses for the other elements is a routine operation with this equipment.

Slide 3 - 5: Acid Calibration Data for ICP Analysis (Replicate Runs of a Standard Solution in Ultrex HNO_3)

Since the leachate samples had been previously acidified to 6 - 8 N HNO_3 in order to stabilize Np and Pu in solution, it was necessary to calibrate the ICP equipment

for acidified samples. Two independent preparations of a well known standard were done by mixing the standard with Ultrex HNO_3 at different normalities. These samples were then analyzed and the data compared to the known value for the standard. The calibration plots shown present the per cent deviation of the acidified standard from the unacidified standard as a function of acid normality. The "X" and "O" represent data from both preparations. Excellent agreement can be seen for all elements except Cd. The "O" data for Cd does not fall on the line described by the "X" data. Molybdenum is the only element showing a positive deviation from the known standard. All other elements indicate that increasing acid concentration tends to mask the element in the solution.

Since the precision of these two runs is so good, we feel confident that by diluting the leachate samples to 4N and applying a correction factor based on these calibration curves, we can obtain correct values for the concentrations of these elements in the leachate. So far this can only be done for the bicarbonate and distilled water as the high salt content of the brine prohibits analysis. The ICD people (particularly Art Langhorst) are working on this problem.

STATUS OF BEAD LEACH I ANALYSES (5/17/79)

	DAY	1	2	3	6	11	20	37	65	120	230	420
BIGBIRD & DW CHANNELS (10-28)	100 Å EVAP. COMPLETED											
	α-COUNTING COMPLETED (Np & Pu)											

BRINE CHANNELS ONLY (1-9)	CHEMISTRY COMPLETED											
	α-COUNTING COMPLETED (Np)											
	α-COUNTING COMPLETED (Pu)											

ALL CHANNELS	ICP ANAL.											
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COMPOSITION OF 76-68 GLASS AND DETECTION LIMITS FOR ICP ANALYSIS OF LEACHATE

ICP DETECTION LIMITS

Al	.01 ug/ml
As	.21
→ B	.03
→ Cd	.003
Co	.008
Cu	.001
→ Fe	.002
Li	.001
Mn	.001
→ Mo	.01
→ Ni	.01
P	.4
Pb	.03
→ Si	.01
→ Sr	.01
→ Ti	.001
→ U	.04
V	.004
→ Zn	.005
→ Zr	.002
→ Ca	.002
K	.2
Mg	.001
→ Na	.01

Glass Composition Weight Percent

0.13	Rb ₂ O
0.33	SrO →
0.21	Y ₂ O ₃
1.77	ZrO ₂ →
2.28	MoO ₃ →
1.07	RuO ₂
0.17	Rh ₂ O ₃
0.53	PdO
0.03	Ag ₂ O
0.03	CdO →
0.26	TeO ₂
1.03	Cs ₂ O
0.56	BaO
0.53	La ₂ O ₃
1.19	CeO ₂
0.53	Pr ₆ O ₁₁
1.67	Nd ₂ O ₃
0.33	Sm ₂ O ₃
0.07	Eu ₂ O ₃
0.05	Gd ₂ O ₃
4.58	U ₃ O ₈ →
9.77	Fe ₂ O ₃ →
0.41	Cr ₂ O ₃
0.20	NiO →
0.48	P ₂ O ₅
12.51	Na ₂ O →
39.80	SiO ₂ →
9.47	B ₂ O ₃ →
2.00	CaO →
4.97	ZnO →
2.97	TiO ₂ →

Detection limits (based on 4 times the matrix blank)

ACID CALIBRATION DATA FOR ICP ANALYSIS
(REPLICATE RUNS OF STD IN ULTREX HNO_3) - 1

ACID CALIBRATION DATA FOR ICP ANALYSIS
(REPLICATE RUNS OF STD IN ULTRAFIX HNO₃) - 2

ACID CALIBRATION DATA FOR ICP ANALYSIS
(REPLICATE RUNS OF STD IN ULTREX HNO_3) - 3