MONTHLY PROGRESS REPORT

JUNE 9, 1966

PLUTONIUM ANALYSIS GROUP

SUMMARY

Gamma scanning of radioactive wastes continues. An air conditioner has been added to aid in temperature stability. Gamma scan and calorimeter values are being compared to weighed quantities of isotope.

Good shipper-receiver calorimetry agreement is being attained on the plutonium oxide shipments from Savannah River.

Atomic absorption spectroscopy curves were attained for potassium, copper, chromium, nickel, and iron. Development work is continuing on the construction of an yttrium lamp.

Development work is continuing on the establishment of surveillance procedures.

Promising results are being attained in the development of a method for the determination of fluoride in plutonium oxide.

Being investigated are pyrohydrolysis and microdiffusion procedures, followed by spectrophotometry with thorium chloranilate as the color developing agent.

Instability of the mass spectrometer magnet regulator was corrected. Improved electropolishing procedures have been developed.

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DETAILED REPORT

A. GAMMA SCANNING

Radioactive waste continues to be gamma scanned with a total of 9,111 cans (4054 burnable and 5057 non-burnable) analyzed to date.

Calorimetry and gamma scan values are being attained on known quantities of plutonium^{238} in #12 cans. The results to date indicate that gamma scan values are more accurate than calorimetry with less than 0.5 gram of isotope:

Waste Category	Grams Pu^{238}	Avg. Gms. Pu^{238} Found Calorimetry	% Error Calorimetry	Avg. Gms. Pu^{238} Found Gamma Scan	% Error Gamma Scan
Non-Burnable	1.80	1.75	2.78	1.69	6.11
Burnable	0.503	0.494	1.79	0.465	7.55
Non-Burnable	0.207	0.246	18.8	0.197	4.83
Burnable	0.102	0.114	11.76	0.110	7.84

B. CALORIMETRY

Good calorimetry agreement between Savannah River and Mound have been attained on the five most recent shipments:

Shipment No.	SRP Value (Watts)	MLM Value (Watts)	% Difference
15	397.81	398.78	- 0.24
16	424.72	425.18	+ 0.11
17	397.66	397.67	0
18	379.98	380.42	+ 0.12
19	408.51	408.80	+ 0.07
TOTAL	2008.68	2010.85	+ 0.11
	(3485.06 gms)	(3488.82 gms)	

The air conditioning unit in SM-21 was upgraded to provide a more constant temperature for calorimetry operations.

C. ATOMIC ABSORPTION SPECTROSCOPY

Several hollow cathode lamps were received and working curves from 1 ppm to 20 ppm were attained for potassium, copper, chromium, nickel, and iron. To observe whether other elements would interfere with the absorbance readings, analyses were performed in the presence of various ions. Insignificant differences were found when the data with pure solutions were compared to the data with the added elements.

Being investigated is the possibility of constructing a plutonium hollow cathode lamp. Non-radioactive work is being

done with a lead lamp in which the lead hollow cathode has been replaced by a machined piece of yttrium. The quartz window has been reinserted and the lamp is ready for filling with argon and sputtering of the yttrium.

Getting working curves for the refractory elements, boron, beryllium, and uranium, are planned upon receipt of the nitrous oxide and nitrous oxide burner head.

D. SURVEILLANCE

Development work is continuing with the establishment of surveillance procedures. Nickel analysis provided more meaningful data than silica analysis; thus the silica procedure was eliminated. Non-radioactive analyses of known nickel and beryllium solutions proved the surveillance analytical procedures were quantitative. Gamma activation analysis would be as accurate and much simpler than the gravimetric analysis for beryllium, and is therefore being investigated. The capsules to contain the radioactively contaminated beryllium have been designed and constructed and a new source has been ordered.

Two old concept units have been disassembled and are being evaluated. The older unit, manufactured in 1963, contained

more than 50% of the cake material in a dry and loose form. The newer unit, manufactured in 1965, appeared normal in all respects.

E. DETERMINATION OF FLUORIDE IN PLUTONIUM OXIDE

A spectrophotometric analysis with thorium chloranilate as the color developing agent is being investigated for the determination of fluoride in plutonium oxide. A concentration-absorbance curve was linear for 10-200 micrograms of fluoride/milliliter. To reduce interferences from other cations and to convert the fluoride to a form in which analysis is possible without losses during dissolution, microdiffusion and pyrohydrolysis techniques are being investigated.

With microdiffusion, perchloric acid could be readily substituted with either nitric acid or phosphoric acid without loss of accuracy. However, when standard fluoride solutions were carried thru the microdiffusion procedure, only 85% recovery was attained.

With pyrohydrolysis, the equipment is more elaborate but 100% recovery was attained. Pyrohydrolysis work will proceed with

spiked samples of plutonium oxide to observe whether equally good recovery is obtained.

F. MASS SPECTROMETRY

Intermittent instability of the mass spectrometer magnet regulator recently has gotten progressively worse. A detailed study of the circuitry was undertaken to determine the origin of the oscillations. It was found that oscillations were "triggered" by a transient or sag in the line voltage. By by-passing the Sola transformer and increasing the gain on the high gain amplifier, the regulator is now operating within the required limits. Circuitry modification is planned to permanently eliminate any future instability.

The electropolishing technique was refined to obtain a better finish on the source components. Various acid baths and current densities were investigated and the best combination was chosen. An electronic filter was also installed into the rectifier circuit of the DC power supply to provide a more constant voltage.

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