MONTHLY PROGRESS REPORT

MARCH 15, 1966

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BUILDING ANALYSIS GROUP

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SUMMARY

The second gamma scanning installation was put into operation in addition to provisions for the gamma scanning of high level trash. The instrumentation was calibrated and analyses are now being performed on a routine basis.

Electronic difficulties on the heavy element mass spectrometer were corrected and the instrument is back in operation. The major portion of the backlog of samples, including the first quarter samples of the Plutonium Isotopic Sample Exchange Program, have been completed. A procedure for the analysis of neptunium-237 was perfected and studies have been completed to determine the optimum conditions (least noise) for the electron multiplier.

Plutonium oxide shipment 10 from Savannah River was calorimetered. A shipper-receiver deviation of less than 0.1% was found.

The study concerning the reduction of volume and dissolution of incinerator ash and the spectrophotometric study on the

reaction mechanisms of plutonium in ascorbic acid and sulfamic acid were completed.

Investigations have been initiated on the surveillance of units and the analysis and determination of the origin of gases in previously fabricated units.

The evaluation of the experimental polyester reinforced fiber glass alpha box is continuing.

The water and carbon analysis apparatus was improved by incorporation of glass parts.

Work is continuing on gas chromatography of plutonium oxide, the determination of fluoride in plutonium oxide, and the construction of an electronically controlled soap bubbler flowmeter for surface area measurements.

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DETAILED REPORT

A. Gamma Scanning

A second gamma scanning instrument was put into operation to double the previous capabilities. Calibration was completed and routine analyses are now being performed on approximately 500 cans of trash per week.

In addition, the range of the one gamma scanning instrument was expanded to include the analysis of high level trash. By incorporating stainless steel shielding between the can of trash and the scintillation detectors, as much as 12 grams of isotope in non-burnable trash and 8 grams in burnable trash can be analyzed.

The gamma scanning data to date follows:

Total Cans	5572	
Burnable	2441	
Non-burnable	3131	
Greater than 1 gram	545	(>880.4)
Burnable	279	(>500.3)
Non-burnable	266	(>380.1)

Intermediate	3222
Burnable	1357
Non-burnable	1865
Less than 33 Milligrams	1805
Burnable	805
Non-burnable	1000
Total Drums (<.165g)	419
Burnable	179
Non-burnable	240

B. Mass Spectrometry

Several malfunctions of the electronic components were corrected. The Sola transformer was replaced with a unit of higher amperage rating. Several cracked solder joints and some broken wires were repaired, in addition to grounding of the leads in the source cable that were found to be "floating". Contacts were cleaned on the various selector switches of the vibrating reed electrometer and the rheostats of the source filament controls.

The major portion of the backlog of samples has been completed, including the samples from the first quarter of the Plutonium

Isotopic Sample Exchange Program.

A method for the mass spectrometric analysis of neptunium-237 was perfected. A sample of plutonium containing neptunium was "spiked" with known quantities of pure neptunium oxide. An accuracy can be attained that is equivalent to plutonium isotopes in the same concentration range.

A comparison was made of the multiplier gain to the noise level for the 20 stage electron multiplier. The noise level was not excessive if the multiplier gain did not exceed 8 x 10^6. This fact will permit more optimum operating conditions in the future.

C. Calorimetry

Calorimetry on shipment #10 was completed. A shipper-receiver deviation of .08% was found; a comparison of Mound and Savannah River wattage values follow:

Container No.	Mound	Savannah River
3	43.515	43.374
1	36.181	36.078
5	45.266	45.180
13	41.842	41.701
18	44.354	44.428

Container No.	Mound	Savannah River
10	41.517	41.524
17	42.438	42.513
7	45.992	46.025
	341.105	340.823
Grams	591.82	591.33

An additional standard cell was incorporated into the calorimeter electronics to provide more accuracy at the higher wattage levels.

D. Volume Reduction and Dissolution of Incinerator Ash

Development work was conducted on various samples from the incinerator process to determine if a more efficient dissolution of the ash was possible.

Various data were attained on fifteen samples, numbers 180 to 194 inclusive. Attempts were unsuccessful to dissolve the ash with nitric acid and hydrofluoric acid on samples 180 to 184 as received directly from the incinerator (<5% dissolution). Treatment of the raw ash with 18 N sulfuric acid and nitric acid was more effective with as much as 33% dissolution (sample

184). The major portion of the remaining pink residue, evidently plutonium sulfate, was soluble in hot water. From 48% (sample 184) to 92% (sample 182) of the original ash was soluble by the combination of these methods.

To determine whether a decrease in volume would be possible by ignition of the ash at higher temperatures, samples 180 to 194 were ignited at 600°C and 1000°C. The weight loss on ignition at 600°C varied from 10.6% (sample 190) to 39.2% (sample 194). An additional loss of weight, from 3.2% (sample 192) to 24.0% (sample 181), was noted after ignition at 1000°C. Total loss of weight at 1000°C varied from 18.1% (sample 190) to 59.0% (sample 194).

The residues of samples 182 thru 188, after ignition at 1000°C, were boiled in aqua regia and the solution filtered thru a medium porosity glass frit. The filtrates were evaporated to dryness, and both these residues and the undissolved residues were submitted for emission spectrographic analyses. Major constituents in the filtrate were aluminum, sodium, zinc, silicon, lead, and calcium. The major constituents of the

residues were zinc, nickel, iron, calcium, aluminum, and silicon. A wide variety of other elements were present in both cases in moderate to trace amounts.

Recovery of plutonium was attempted on samples 189 thru 194. Nitric acid and hydrofluoric acid were added to dissolve the residues after the 1000°C ignition. The solutions were then filtered and the non-dissolved residues were dried and weighed to determine the amount of dissolution. The undissolved residue did not contain more than 1% impurity of any one element by emission spectrographic analysis. The filtrate contained major quantities of nickel, lead, aluminum, calcium, iron, and magnesium. Aliquots of the filtrates were alpha mounted to determine the plutonium content.

Sample No.	% Dissolved	Pu Content (mg.)
189	89.5	21
190	61.9	57
191	83.8	28
192	18.7	9
193	39.0	5
194	.5	11

The plutonium recovery amounted to a maximum of 2.7% (sample 190) of the total ash. Other recovery data indicates from 2 to 5% of the total ash is plutonium.

E. Stability Studies of Ascorbic Acid and Sulfamic Acid

Studies were completed on the spectrophotometric stability studies of ascorbic acid and sulfamic acid in various concentrations of nitric acid. By correlating this data to the recovery and production processes, it was possible to determine the optimum conditions for the valence adjustment of plutonium solutions.

F. Surveillance

Procedures have been written for the surveillance of previously fabricated units. Plans are progressing for the shielding of the box in which disassembly will occur. The "disassembly block" has been constructed.

G. Determination of the Origin of Gases in Previously Fabricated Units

Investigations have been initiated to determine the origin of various gases (hydrogen, nitrogen, oxygen, carbon dioxide, argon, and moisture) in previously fabricated units. Mock-ups are being conducted with the various constituents of the unit.

This is then followed by analysis of the emitted gases after bake-out, either by mass spectrometry or gas chromatography.

Considerable carbon dioxide was found to adsorb on the sodium silicate. To date, no other gases have been observed. The investigation is continuing.

H. Polyester Reinforced Fiber Glass Alpha Box

Evaluations are continuing on the experimental polyester reinforced fiber glass alpha box. The zipper gasket is satisfactory for securing the plate glass window. At a vacuum or pressure of approximately 15 inches of water, the gasket seal broke, which is comparable to results attained by the zipper gasket manufacturer. With RTV (either General Electric 102 or Dow Corning Silastic 731) instead of the zipper gasket, approximately twice the vacuum or pressure could be attained before rupture of the seal. With a plate glass window secured with RTV, an increasing internal pressure cracked the window prior to breaking of the RTV seal.

Attachment of the glove ports to the plexiglass hood front

with RTV proved to be satisfactory. However, a cross section of the ports showed considerable inclusions, and the recess for holding the glove during a glove change or trash removal was not deep enough for a safe operation. These problems are being remedied by the manufacturer.

I. Water-Carbon Analyzer

The rubber tubing on the carbon-water analyzer was replaced with glass ball joints and specially designed Teflon stoppers were constructed to seal the combustion tubes. This provides a more efficient operation and permits more accurate results.

J. Gas Chromatography

To remove impurities from the helium carrier gas, the gas chromatograph was equipped with a molecular sieve trap submerged in a liquid nitrogen bath. Flushing the detector with acetone corrected periodic base line difficulties.

Analyses were made on plutonium oxide. Nitrogen (.04%), nitric oxide, and carbon monoxide were found.

The heating tape wrapped around the column does not provide a uniform column temperature. Plans are progressing for the insertion of the entire column in a constant temperature oven. The septums, presently difficult to replace in the alpha boxes, will be replaced by a new flat type septum. This will not only enable easier replacement, but also easier sample injection.

K. Determination of Fluoride in Plutonium Oxide

Difficulties still exist in the dissolution of plutonium oxide without evolution of the hydrogen fluoride. Considerable promise is shown in the fusion of the plutonium oxide with sodium peroxide followed by fusion with sodium hydroxide. A 100 milligram sample of plutonium dioxide was quantitatively dissolved in approximately 30 minutes. Quantitative recovery of fluoride during this dissolution will be investigated.

L. Soap Bubbler Flowmeter

An electronically controlled soap bubbler flowmeter is being constructed to measure gas flow rates on the surface area analyzer. This will eliminate the need for entering the alpha

box gloves during measurement, which has previously produced erratic results.

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