

**ROUGH DRAFT**

MOUND LABORATORY-MONSANTO  
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**SECRET**  
DATE 7/15/66 INIT S.M.A.

MONTHLY PROGRESS REPORT SYNOPSIS

JULY 15, 1966

THIS DOCUMENT CONSIST OF 4 PAGES  
THIS IS COPY 1 OF 1 SERIES ROUGH DRAFT

PLUTONIUM ANALYSIS GROUP

GAMMA SCANNING

Radioactive trash continues to be gamma scanned with a total of 10,714 cans (4,750 burnable and 5,964 non-burnable) analyzed to date. Gamma scan data were compared to calorimetry values on 16 cans and to weighed quantities of isotope on 12 cans.

Although the results were within experimental error, the calorimeter values were erratic with less than 500 milligrams of plutonium<sup>239</sup>, evidently due to poor packaging and poor equilibrium.

Construction has been completed on the addition to the gamma scan facility.

CALORIMETRY

The calorimeter batteries have been replaced with Evenvolt power supplies. Preliminary data indicates a stable voltage is produced.

ATOMIC ABSORPTION SPECTROSCOPY

Distilled water having a high sodium content was traced to a faulty still. Sodium, copper, and zinc analyses by atomic

GROUP 1

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absorption spectroscopy aided in solving the problem.

Assembly has been completed on the yttrium hollow cathode lamp as preliminary work for the plutonium lamp. The characteristic glow with the argon filler gas was attained without evidence of arcing. The yttrium lamp will be evaluated upon installation of the nitrous oxide burner head.

SURVEILLANCE

Development work has been completed on the surveillance procedures and the procedure manual has been written.

METALLOGRAPHY

Metallographic containers have been constructed for photographing mounts of radioactive materials without contamination of the metallographic instrumentation.

MASS SPECTROMETRY

Preparation work for analyses of the boron exchange samples has been completed. The ion beam transmission has been increased 280 per cent by alignment of the electromagnet and collector slit for optimum peak shape and beam width. The spectrum is free of interferences from mass 85 to 94 and essentially free of interferences from mass 80 to 110. Re-

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agent grade boric acid gave a value of  $18.38 \pm 0.23$  weight per cent boron<sup>10</sup>. The boric acid was converted to sodium tetraborate by fusion with sodium hydroxide directly on the ionizing filament.

Work is presently proceeding on the analyses of two synthetic boron standards prepared by New Brunswick Laboratories.

CONTROLLED POTENTIAL COULOMETRY

A controlled potential coulometer cell was designed and constructed and "cold" work is being performed with iron solutions.

FLUORIDE ANALYSIS

Development work is continuing on the determination of fluoride in plutonium oxide. The effect of the sodium hydroxide concentration and the substitution of nitric or sulfuric acid for the phosphoric acid are being investigated in the microdiffusion method.

The pyrohydrolysis method is essentially complete. Slightly more fluoride was evolved at a higher temperature (900°C), but insignificant fluoride was found upon re-analysis, indicating complete recovery. On five consecutive analyses of

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plutonium oxide, an average of 0.12% fluoride was found, with a deviation of less than  $\pm 0.01\%$ . A sensitivity of 5 ppm or 10 micrograms of fluoride is possible. Data are now being attained on spiked samples to determine the accuracy of the method.

APPARENT DENSITY

Apparent densities are now being determined on SNAP material which has a smaller particle size (2-6 mils) than previous material. Investigations are being conducted to determine whether the mercury is actually wetting the smaller particles under the evacuated conditions.

MISCELLANEOUS

Being investigated is a plunger-type constant volume pipet. If practical, it will be used for alpha box work, which will reduce usage of glass pipets, burets, and graduated cylinders.

The work on the four SM-61 alpha boxes has been completed. This consisted of balance replacement, painting the boxes, installing new windows, replacing the metal electrical outlets with acid resistant units, and laying Teflon sheet to protect the alpha box floors.

Original Signed by  
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