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PROGRESS REPORT

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SM Building Analysis Group

In addition to the normal analytical support, the following work was performed by the SM Building Analysis Group:

A. MASS SPECTROMETRY

One of the gate valves to the vacuum ion pump was repaired and replaced in the system.

An improved source control is being added to the mass spectrometer to permit faster scan rates and more accurate results.

Difficulties are being experienced in maintaining vacuums in the low 10^{-7} torr regions when the sample carriage is inserted into the source chamber. The leak is believed to be caused by a small crack in the ceramic header of the sample carriage.

Another header has been purchased and is presently being installed.

B. TRASH ANALYSIS

The previous difficulties encountered with the gamma counting of radioactive drums of trash was found to be caused by a high neutron flux. In the presence of neutrons a high result was

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attained, with an error estimated to be as much as 500%.

To decrease the effect of neutrons, shielding of the crystals has been attempted with various materials including polypropylene, cadmium, paraffin, borax, and iron. Also tried were several procedures of counting: (1) counting all the channels using the four crystals, (2) counting the three highest channels using the four crystals, (3) counting all the channels using one crystal at a time followed by totaling the counts, and (4) counting the highest three channels using one crystal at a time followed by totaling the counts.

Calorimetered samples are serving as standards. To determine the actual effect the neutron flux contributed, several samples with essentially no neutrons are being compared to samples with a high neutron flux.

Thus far the data has been erratic; generally values are biased on the high side with an accuracy of 20-30%.

Part of the difficulty is contributed to temperature fluctuations in the room. To remedy this, an air conditioner has been purchased and will be installed in the near future.

Also being investigated is a more accurate procedure for determining the amount of material in #12 cans. Gamma coincidence counting appears promising.

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C. POLAROGRAPHY

The polarograph has been set up for radioactive analysis. Initial work will be development of an analytical method for the determination of small quantities of neptunium in plutonium oxide. Pure neptunium oxide has been received; work will commence in the near future.

D. STACK-UP

Investigations are being conducted to determine the cause of continually low stack-up values on plutonium oxide. Because ignition of the material occurs in a quartz combustion tube, the presence of silica was suspected. (This would not be seen in the usual emission spectrographic impurity analysis because the silicon would be volatilized during the nitric-hydrofluoric acid dissolution of the oxide.)

Silicon was determined gravimetrically on NO-28 which was a lot with a stack-up value of 98.0%. A total of 0.49% silica was found. On NO-29 (stack-up value of 98.6%), .05% silica was found.

E. SIZE - SURFACE AREA

Plans have been initiated for the conversion of three non-radioactive alpha boxes for size and surface area measurements in the SM area.

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