PROGRESS REPORT

July 15, 1965

SM Building Analysis Group

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

A. WATER ANALYSIS

To obtain more efficient and more accurate water results the CEC Moisture Analyzer has been adapted for radioactive use. For assurance that the instrument is operating properly sodium tungstate and sodium tartrate, which contain known quantities of water, are being analyzed. Also the CEC results are being compared to the conventional gravimetric results on plutonium oxide. The data follows:

Sample	Water Present	Water Found	% Recovery
Sodium Tungstate	8.4 mg.	8.2 mg.	97.6%
Sodium Tungstate	7.9 mg.	7.8 mg.	98.7%
Sodium Tartrate	9.0 mg.	9.0 mg.	100%
NO-30	.18% (gravimetric)	.20% (CEC)	

The investigation is continuing with lesser quantities of water and other plutonium oxide samples.

B. TRASH ANALYSIS

Work is continuing on the development of a method for determining

the quantity of plutonium^{238} by gamma counting drums of waste material. An accuracy of 10% is hoped to be obtained with results independent of high neutron flux and also independent of the density of the trash. Several modifications of the previous procedure have been made:

1) Polypropylene and cadmium now shield the crystals. This eliminates the neutron counting spectrum but gives rise to a prompt photo emission with a maximum energy at 0.140 mev.

2) The top five channels are being used rather than peak area analysis. The gamma peak height is done at 0.100 mev. and reduces interference from the prompt photo emission to a negligible level. This enables gamma counting to be performed with neutron fluxes of 1 roentgen per hour.

3) The density correction has been modified. The new factor is

$$\left[1 + \left(\frac{I_{os} - I_s}{I_s}\right) 0.693\right] \; Id$$

where I_{os} = Counts/minute of standards (empty drum) minus background

I_s = Counts/minute of standards thru the trash drum

I_d = Counts/minute of trash drum minus background

This factor is now linear whereas the old factor was not valid below a density correction of .05.

4) The line voltage to the analyzer is now filtered and stabilized.

5) Nine new standards have been prepared (and five more are planned) including high and low level neutron samples containing high and medium density materials.

6) The drum scanning area has been decreased to encompass only that area filled with cans, which provides a more accurate density correction.

A total of 25 standard runs were conducted including 7 high level neutron samples and 11 high density samples (6 surrounded by glass and metal and 5 encapsulated in stainless steel cans). Variations were made concerning positioning of the standards and the number of standards per run. No systematic errors were apparent but errors were as high as 38.5%.

Uncertainties that still exist include periodic changes in the calibration factor and standards encapsulated in aluminum giving results 20% higher than those in stainless steel. Studies are continuing.

C. GAS CHROMATOGRAPHY

The gas chromatography system is being redesigned to provide a more compact unit for the SM-61 alpha boxes.

D. SURFACE COATINGS

A PVC surface coating and six polyester resins were applied to both stainless steel surfaces and aluminum surfaces and are currently being evaluated inside an alpha box. After seven weeks of exposure to acid fumes and radioactivity only the polyvinyl chloride sample applied to the aluminum surface has failed.

E. MASS SPECTROMETRY

The magnet was realigned and the ion beam focused on the mass spectrometer.

The vacuum difficulties have been corrected with vacuums maintained in the 10^{-7} torr region. However a problem still exists with lengthy (2 hour) pump down and outgassing periods after sample introduction into the system.

Several minor electrical and electronic problems have been resolved. Good resolution is now attained and the mass discrimination factor determinations have been completed for the new electron multiplier.

Mass scans have been completed on shipment samples 60, 61 and 62.

F. PARTICLE SIZE

The previous difficulties in the Sartorius sedimentation balance were corrected by replacement of the pan and sedimentation jar and wetting the pan rod with sodium hydroxide solution prior to analysis.

G. CONTROLLED POTENTIAL COULOMETRY

Analysis of 6 - 12 milligrams of uranium by controlled potential coulometry is being investigated according to Method 1.302 of the Selected Measurements Manual. With a mercury cell, titrations can be completed in approximately 12 minutes. With standard uranium solutions the results had a negative bias of 0.4 - 0.8%. This was believed to be caused by spattering of the solution either during stirring or sparging with an inert gas. The situation will be remedied in hopes of attaining 0.1% accuracy.

H. CARBON ANALYSIS

The equipment for the gravimetric determination of carbon is being modified to include a small 4 inch combustion furnace and an all glass system with ball joints.

I. POLAROGRAPHY

Malfunction of both the derivative polarograph and recorder has temporarily ceased the development of a method for

neptunium. The recorder has been repaired but electronic difficulties still exist with the polarograph.

J. URANIUM COLORIMETRIC METHOD

Difficulties have been experienced in attempts to duplicate the data described in the literature for the spectrophotometric determination of small quantities of uranium in plutonium. The use of isopropyl alcohol instead of ethyl alcohol and decreasing the PAN concentration from 0.1 to .035% appear to alleviate the situation with standard uranium solutions. Similar work in the presence of plutonium is now being investigated.

K. COAT MICROHARDNESS

The data from 10 randomly selected batches of NC-4 and NC-5 coat microhardness results were analyzed to determine if it would be possible to legitimately reduce the number of determinations per batch from 50 to 25. The mean was obtained for the first 25 results and for all 50 results and the 95% confidence limit was determined for a single mean. The results follow:

Sample	Range 1-25	Range 1-50	Mean 1-25	Mean 1-50	95% Conf. Limits For a Single Mean 1-25	95% Conf. Limits For a Single Mean 1-50
4 - 1	281-440	175-440	353	332	±21	±16
4 - 15	257-404	214-404	330	311	±18	±16
4 - 16	206-344	206-344	285	283	±12	± 8
4 - 17	358-458	229-458	354	328	±23	±15
4 - 18	247-523	247-523	357	352	±23	±19
4 - 19	247-574	247-574	355	351	±20	±12
5 - 1	295-415	295-429	349	350	±12	±10
5 - 2	285-415	285-422	360	357	±15	±10
5 - 3	219-401	219-401	293	297	±20	±12
5 - 12	218-358	218-364	287	291	±14	± 9

Because 7 of 10 of the 1-25 results agree within 1.4% of the 1-50 results the data will be submitted to the Design Agency for approval of the shorter procedure.

Wayne R. Amos

WRA: ch

DISTRIBUTION:

Copy 1A - W. R. Amos