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MOUND LABORATORY (MONSANTO)
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MOUND LABORATORY

OPERATED BY

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MONSANTO CHEMICAL COMPANY
MIAMISBURG, OHIO

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PROGRESS MEMORANDUM

September 16, 1952 - October 15, 1952

CONTROL SECTION

Abstract

Group 20

One hundred ninety-seven purity determinations were made. Fifteen resamples were required. An unusual amount of foil holder repair was necessary.

Group 22

The radium accountability procedure will be incorporated with other classified accountability procedures to form a Classified Accountability Procedures Manual.

Contaminated slides are posing a problem in the D. M. hoods.

Group 24

Operations progressed normally. Evidence of gas generation was noted in the first T-29 counter received from L. A.

Group 33

A report on polonium assay work was submitted.

Preliminary work for gathering the Scioto activation cost figures was completed.

The group made a trip to the Scioto installation on October 14.

GROUP 33
taken from _____
for _____ and
_____ section

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DETAILED REPORT

Group 20 - Electrolytic Purity Assay

During this period 197 products were assayed; 15 required resampling. This is the high peak in Group 20 operations thus far this year. To date 1,279 products have been run; 74 or 5.8 per cent required resampling. This is an average of 32.5 purity determinations per week. Comparable 1951 figures are 1,057 products run; 52 or 4.9 per cent required resampling. This is an average of 26.8 purity determinations per week.

Twenty-one Bismuth analyses were run for R. W. Endebrock.

A qualitative analysis and two pH determinations were made for M. N. Wolfe.

EQUIPMENT AND MAINTENANCE

Eight foils were dropped and recovered during this period.

The loading blocks on the load side of M.L.B. #1 and #2 were cleaned and lubricated.

Forty-two foil holders were cleaned and adjusted. Two of the holders also required new spirals and six holder tips had to be straightened.

The typewriter and Friden Calculator U.S.A. #9743 were given routine service, in the warehouse on September 25.

The Sheffield Shadow Gage was transferred to the machine shop as per agreement with H. J. Shepherd.

The walls and ceiling of rooms T-260, 263, 264, 265 were painted and trimmed the week of September 22, 1952.

DEVELOPMENT

Macro - Assay Method Development

A check on the balance was made to determine the limits of its reproducibility. The method followed was suggested by Dr. A. H. Corwin, Johns Hopkins University.

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In the first test it was found that a variation in the rest point of as much as 0.2 scale division on the pointer scale (equivalent to about 0.02 milligrams) was possible depending on the amplitude of swing. Dr. Corvin listed three possible causes: (1) air leakage in the balance case, (2) a loose adjusting nut on the beam, and (3) knife-edges loosely mounted. No evidence of the first two conditions was found, and nothing could be done to correct the third if it existed.

The next test was reproducibility at zero load. One hundred successive rest points were taken, and they showed a standard deviation of 0.04 scale divisions on the pointer. This meant a probability that: (1) 68 per cent of all rest point readings would fall within ± 0.01 milligrams of their average, (2) 95 per cent of all rest point readings would fall within ± 0.02 milligrams of their average, and (3) 99 per cent of all rest point readings would fall within ± 0.03 milligrams of their average. This seemed to be satisfactory performance.

Any arrestment error would show up as a decrease in reproducibility with increased load, so the next test was reproducibility at 50 gram load. The 50 gram pan weight was counter-balanced against the 20, 10^o, 10^x, 5, 2^o, 2^x, and 1 gram pan weights, and a series of one hundred rest points were taken. They showed a standard deviation of 0.05 scale divisions on the pointer scale. This meant a probability that: (1) 68 per cent of all rest point readings would fall within ± 0.01 milligrams of their average, (2) 95 per cent of all rest point readings would fall within ± 0.03 milligrams of their average, and (3) 99 per cent of all rest point readings would fall within ± 0.04 milligrams of their average.

Because of the strain it would put on the gloves, as well as the time required, the test for error due to placement of the rider was not undertaken. However, the maximum error that could reasonably be attributed to this factor would be 0.01 milligram.

From these results, it was concluded that the balance itself was capable of performing to the degree of reproducibility desired for this development, though there was very little margin for other errors.

The next test was for reproducibility with a control cylinder in balance against the necessary pan and rider weights, and without any handling of the cylinder between rest point readings. One hundred successive rest point readings showed a standard deviation of 0.45 scale divisions on the pointer scale. This meant a probability that (1) 68 per cent of all rest point readings would fall within ± 0.13 milligrams of their average, (2) 95 per cent of all rest point readings would fall within ± 0.25 milligrams of their average, and (3) 99 per cent of all rest point readings would fall within ± 0.38 milligrams of their average.

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This was an undesirable variation. One of every three of the above readings would fall outside of the limits set up for overall variation in the entire purity determination, and no error due to handling the cylinders or the gauges or guns was introduced.

Group 22 - Inventory

Operations

Events connected with the issuance of a procedure for the control of Radium at Mound Laboratory have caused a reexamination of the Standard Operating Procedure Manual. It has been decided that it would be preferable to compile a separate manual containing those procedures which must carry security classification, and cross-reference the classified manual into the unclassified manual. Classified accountability procedures dealing with the control of radioactive material, now existing as separate documents, will be edited into the form of Standard Operating Procedures and incorporated into the new manual.

Since the introduction of the stannous chloride process of concentration, the concentration of Bismuth per 100 ml. of superjuice solution has been lowered from an average of 200 mg. to 10-15 mg. The reduced Bismuth content has eliminated the precipitation of Bismuth from these solutions upon standing, and apparently obviated the need for introducing additional nitric acid into the DM samples to protect them against precipitation.

During the past several weeks, the Inventory Group has run duplicate samples, diluted with acid and undiluted, and the data as expected show closer agreement with the gamma count in the case of the undiluted solutions. The dilution step has therefore been abandoned, and current DM samples are direct aliquots from the solution to be assayed.

The group has recently had trouble with "hot" bottoms on DM slides as delivered to the Counting Room. The reason for this has not yet been found; it is thought possible that the higher level of activity in the hoods resulting from the change-over to undiluted samples may have increased background to a point where this will be a recurring trouble. The solution may lie in more frequent wash-downs of the hoods in this line.

The problem of the disposition of spent initiators remains unsolved at this writing pending the examination of data being collected by the Electrolysis Group relative to the influence of the presence of Beryllium in the lines on the final neutron count of plated material.

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On October 13, the Inventory Group released ten of these items for shipment to Los Alamos at their request.

The Inventory Group has in its possession sufficient scrap metal to justify the organization of a sale, and the necessary collection, weighing, and classification is now under way. The items consist of platinum both clean and contaminated, clean gold, contaminated silver, and clean beryllium. The material should be ready for delivery to the Warehouse during the current month.

Development

Work is being resumed on the problem of evaluating the discrepancy known to exist between calorimeter value and "Alpha" assay of the same solution. An experiment has been designed which it is hoped will lead to the determination of the magnitude of the discrepancy, and at the same time furnish data for statistical analysis of dilution precision and mounting precision. While the use of statistical methods will reduce the amount of data to be collected to a minimum, it is not yet known how extensive a program will be necessary. Examination of the data from at least two experiments are needed to determine the extent of the program. We plan to use the services of Mr. K. Busch in the program to the extent that he is available.

Group 24 - Calorimetry and Counting

Operations

The assay work load was slightly heavier than normal during this period due in large part to the extra activity required by the Y Section.

The problem of high neutron products, while still in evidence, has been lessened by the recent production of guns with a much lower proportion of high neutron items. More of these items will probably have to be reworked in the near future to bring the stockpile back to a condition approximating normal.

A second T-29 neutron counting geometry, together with the scaler and other electronic gear to complete the setup recommended by Site Y representatives, has been received and installed. This installation was completed near the end of the period and the data taken so far are

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not sufficient for a reliable evaluation of the counters performance.

The completely unexpected and quite unusual difficulty was experienced with the original T-29 geometry, the first one received from Site Y. After a period of about four months of satisfactory service it was discovered that a pressure of considerable magnitude had developed inside the drum containing the water moderator. There was only a small air space above the water level, the drum having been almost completely filled with distilled water. The internal pressure caused a bulging at both the top and bottom ends of the drum forming the moderator cavity, the distortion of the top being the more serious since it prevented proper assembly of the top panel to the moderator drum.

The distortion was corrected satisfactorily by the Engineering Department. Also the filling hole plug was fitted with a pressure gauge so that the internal pressure could be readily checked in the future. After rinsing, the drum was filled with distilled water at room temperature. The reassembled counter was returned to service on September 26. To date no pressure buildup has been noted.

In addition to the regular production work, the following assays were made for other groups during this period:

<u>No.</u>	<u>Assays</u>	<u>Sample</u>	<u>Group</u>
1		Prod. Gauge	Neutron Source
5		Prod. Gun	" "
8		Neutron Source	" "

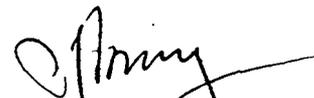
Group 33 - Special Problems

A report on Polonium Assay work was completed during the first part of the period.

Most of the period was spent studying the various aspects of the Scioto Activation cost estimates. The preliminary discussion was completed and procedure for compiling the information required from the operating groups was developed.

An inspection trip was made to Scioto Laboratory on October 14, 1952.

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P. J. Lowry

Copy 1 - Mr. E. C. McCarthy
2 - Mr. P. J. Lowry
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