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DPW-55-333

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July 25, 1955

MEMORANDUM

FROM: WILLIAM A. JENKINS

LABORATORY PROJECT FOR ISOLATION OF PLUTONIUM

Reference (1) Memorandum to M. H. Wahl from
F. W. Tober (DPST-55-230) dated
April 14, 1955.

(2) Ltr. to H. Worthington from M. H. Wahl
(DPST-55-271) dated May 31, 1955.

The reference letters describe proposed facilities to be operated under Technical Division supervision for research with plutonium. An attempt is made in this memorandum to visualize the research program for such a facility for the next five years, to aid in determining whether the cost of providing such facilities is justified.

Three mechanical button lines constitute the present metal production facilities. The capacity of these lines will be exceeded sometime between 1956 and 1957 depending on improvement of equipment availability. The reactor power ascension predictions require ultimate capacity increases which will cost about \$1,200,000 for the button line equipment. Two lines are presently located in Building 221-F and a third in Building 221-H. Space for a fourth exists in Building 221-H. Further expansion will be required at additional cost. It will be desirable to base new construction on an improved process as far as possible. Planning must commence immediately to accomplish this aim.

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C. J. Banick, AED Class Officer

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Experience has shown the desirability of maintaining on-site research facilities in support of all phases of plant operation. The present mechanical button line is largely based on off-site experience and no provision is made for this type of research at SRL. Reference (1) proposes a series of glove boxes for experimentation with solution and solid forms of plutonium on 25 gram scale under isolation conditions. It is, in every sense, a research tool and not a small scale production unit. The research interest should focus on increasing plant capacity and on closer control and improvement of product quality. The overall program is distributed under three headings below.

A. PLANT ASSISTANCE

In the past, variations in the mechanical button line process have, of necessity, been handled on a test authorization basis, and as a result only a minimum of effort has been expended on determining the optimum limits of the variable under investigation. It is significant that at the present state of knowledge the only method of evaluating a change in a unit operation is the observation of the behavior of the product in subsequent operations. This reflects the general empirical approach that has been required.

B. NEW PROCESS DEVELOPMENT

New process development is impossible in production equipment. Off-site research is complicated by the lack of representative SRP feed material and the difficulty in coordinating such research with SRP requirements.

C. NUCLEAR CHARACTERISTICS STUDY

If the present trend continues, emphasis on accurate nuclear evaluation of SRP products will become increasingly important. It is wise to encourage the establishment of a nuclear study group to become expert in the accurate evaluation of the products. The effectiveness of this group will be improved by the availability of the equipment proposed in this memorandum.

Possible research programs are outlined below.

A. PLANT ASSISTANCE

Plutonium(IV) Peroxide Precipitation

1) Determine the optimum sulfate concentration prior to precipitation to minimize time of filtration and supernate losses.

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2) Investigate other additives to improve overall performance of peroxide precipitation.

3) Decrease drying times by using washes other than nitric acid and alcohol.

Hydrofluorination

1) Determine the causes and remedy of the pressure surges experienced in plant operation and to determine if present operating conditions border on unsafe practices.

2) Determine the upper temperature to which plutonium(IV) fluoride may be exposed to the air. Cooling of the hydrofluorination furnace requires a considerable portion of the time cycle and removal of the fluoride at temperatures above present practice should materially increase through-put.

Reduction to Metal

Factors that influence reduction yields are not well known. In general, reduction yields have been satisfactory at Rocky Flats, Hanford, and Los Alamos but disappointly low at SRP. Unless this situation improves a systematic study of variables affecting the reduction operation is indicated.

B. NEW PROCESS DEVELOPMENT

1) Experience at Hanford has prompted the substitution of plutonium(IV) oxalate for the peroxide. It is their feeling that the oxalate affords a more manageable and predictable precipitate. The maximum use of Hanford data would be made but comparison analysis at SRL is required.

2) It is suggested in reference (2) that the precipitation of calcium plutonium fluoride might bypass the hydrofluorination step.

3) Recent discussions with Dr. B. B. Cunningham at the University of California have indicated another possibility for bypassing hydrofluorination. It is standard practice in Dr. Cunningham's laboratory to precipitate plutonium(III) fluoride, dry the powder and reduce to metal with elemental barium. Plutonium(III) fluoride, precipitated from aqueous solution, unlike the plutonium(IV) fluoride, may be successfully dried for reduction in the normal manner. Barium is used only because it is available in more pure form than is calcium. This series of reactions has been performed only on a small scale at Berkeley and the ability to scale the

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operation to plant size awaits demonstration. It is possible that the dual advantage of a more manageable precipitate and the elimination of dry hydrofluorination can be achieved by this process modification.

C. NUCLEAR CHARACTERISTICS STUDY1) Neutron Multiplication Under SRP Button Geometry

Some uncertainty has existed as to the exact neutron multiplication obtained in buttons and the influence of dimensional changes. A series of special plutonium samples with known Pu-240 contents in shapes comparable to SRP buttons (Note, larger than 25 grams specified earlier) should be prepared under laboratory conditions and carefully counted for neutron escape. A well planned series of experiments should yield a family of curves that sufficiently bracket button geometry obtained in the plant. At present we rely entirely on Rocky Flats final weapon shape analysis for confirmation of button neutron count. Previous data obtained from Rocky Flats indicate that a one to eight percent uncertainty may be normally expected when predicting final shape count from button count.

2) Evaluation of (α ,n) Contributions

Determination of plutonium quality by neutron counting is complicated by neutrons arising from plutonium alpha particles impinging on light element impurities. Close scrutiny of the present basis for determining (α ,n) contributions does not encourage confidence. This is especially true when the sum of the neutrons calculated from Pu-240 spontaneous fission and from light element impurities is lower than the corrected (for multiplication) measured value. This has been encountered at SRP.

I would propose a program to produce highly pure samples of plutonium metal of known Pu-240 content and to blend this metal with controlled amounts of light element impurity. The (α ,n) content then could be quantitatively measured.

Several other possible research programs warrant only the briefest mention:

- 1) Use of other plutonium halides
- 2) Use of reductants other than calcium
- 3) Electrolytic reduction (carried to the research proposal stage by KAPL)
- 4) Reduction of plutonium oxide
- 5) Precipitation of NH_4PuF_5 , sublimation of NH_4F leaving PuF_4
- 6) Preliminary work on special fabrication techniques. (Deep drawing or casting of metal shells).

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Probably further study by a technical group set up to man the facility would eliminate some of the above suggestions. On the other hand, the existence of such a group thinking of these problems in terms of SRP requirements, with adequate facilities to work, should generate many suggestions that are not visualized at this time.

WAJ:L

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