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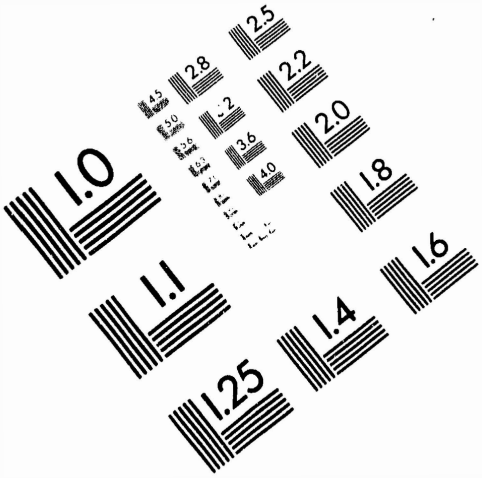

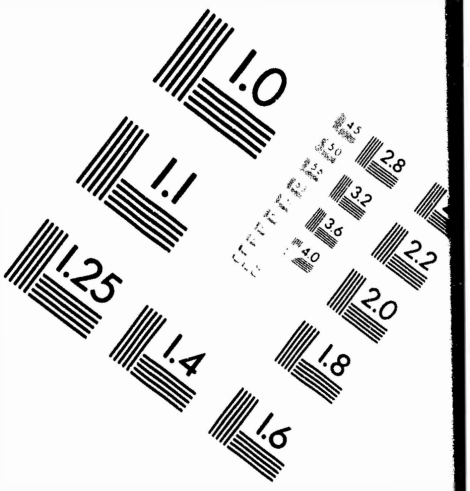

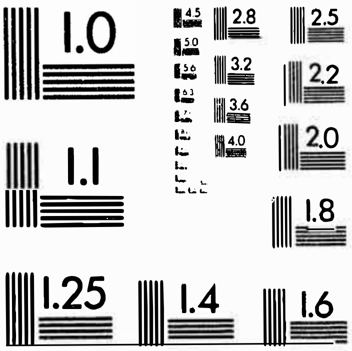

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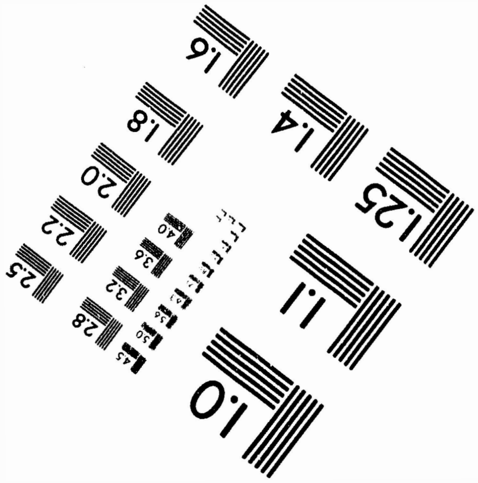

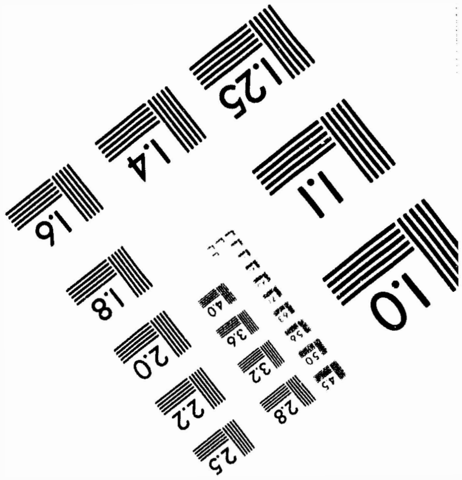

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Explosives Department
Wilmington 98, Delaware

✓DPW-55-217 (SR/H--716)

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Reviewing Official CJ Banick
1 18, 1955
C. J. Banick, AED Class Officer
Date 2/4/89

J. B. Tinker, Manager
Process Section

TRIP REPORT
✓PRODUCTION OF U-233 METAL AT LOS ALAMOS AND OAK RIDGE

It is presently planned that thorium metal, irradiated at SRP, will be shipped to Oak Ridge for separation and the U-233 so derived will be shipped to Los Alamos as a solution for reduction to metal and fabrication of weapons components. Should production exceed the Oak Ridge capacity (about 13,000 lb. Th per month) it is probable that responsibility for separations and U-233 fabrication will shift to SRP. It is the purpose of this letter to present an outline of the processes as developed at Oak Ridge (Y-12) and Los Alamos for the reduction to metal and fabrication of components.

The Oak Ridge process was developed for production of U-235 while processes at Los Alamos were developed for U-235 and U-233. Differences in U-235 and U-233 processing arise from the high purity required of U-233 metal. Dr. R. D. Baker of Los Alamos emphasized the inability of the weapons people to specify the purity required for the final U-233 metal and strongly recommended that the production facility be designed to produce maximum purity material. In addition, he recommended that maximum flexibility be incorporated for future changes and that the capacity be adequate to handle a 50% recycle from weapons fabrication.

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In the following discussions, the Los Alamos U-233 process is presented in most detail but major variations from this process at Oak Ridge are discussed at the conclusion of each unit operation.

No. 1 PEROXIDE PRECIPITATION

The feed to the first peroxide precipitation is very similar to that planned for the Savannah River Plant as outlined in DPST-55-131 dated January 24, 1955. The uranium concentration is about 143 g/l with large amounts of ammonium citrate and acetate added during the previous resin column operation.

Step 1 - Add feed solution equivalent to 500 grams of U-233.

2 - Adjust the pH to 1.5 with concentrated HNO_3(70%) (requires 100 - 300 ml).

3 - Precipitate uranium peroxide by the addition of 30% H_2O_2 (approximately 150 ml) while adding 28% NH_4OH (approximately 200 ml) at such a rate that the pH is held at 1.5. The precipitate is allowed to settle for 15 - 30 minutes at room temperature.

4 - The supernate will contain one gram of uranium per liter which must be recovered. Filter the precipitate on a glass filter funnel (6 in. Corning glass Buchner - medium porosity). This step requires approximately one hour.

5 - Flush water through the cake (approximately 600 ml) and aerate the cake for about five minutes.

6 - Place the filter and cake in a furnace (Heavy Duty Electric Company, Model M-506) and heat at 300°C for eight hours. (This time could possibly be reduced to less than three hours).

7 - Cool the assembly and wet the cake with water. Dissolve the cake by the slow addition of HNO_3(70%).

8 - Dilute the solution with water to 2 liters.

No. 2 PRECIPITATION

Step 1 - Add 10 grams of malonic acid and 2 grams of citric acid to the dilute solution of the previous operation. Ten grams of versene acid may be added if decontamination from thorium is required.

2 - Adjust the pH to 1.5 with NH_4OH(28%).

3 - Precipitate uranium peroxide with NH_4OH and 30% H_2O_2 in a manner similar to the No. 1 precipitation.

4 - Repeat steps 4, 5, 6, 7, and 8 of the No. 1 precipitation.

No. 3 PRECIPITATION

Repeat precipitation and filter operations as discussed in No. 2 precipitation.

The requirements for three precipitations to achieve high purity material is not well demonstrated and the people at Los Alamos have been requested to evaluate the purification achieved in the final precipitation.

The necessity for igniting the uranium peroxide to UO_3 prior to redissolution and reprecipitation is in doubt. The Oak Ridge practice is to simply cover the peroxide precipitate with concentrated HNO_3(70%) and heat to boiling. This effects dissolution and is, from an operational standpoint, a much more desirable process.

IGNITION TO U_3O_8

Two batches of precipitate are blended to constitute one batch for the ignition of uranium peroxide to U_3O_8.

Step 1 - Transfer precipitation cakes to a 10% RH - 90% Pt ignition boat. (Cover boat with lid).

2 - Transfer the boat to a Heavy Duty Furnace Model 5610 and raise the temperature to 900°C for twelve hours. (This time can possibly be reduced to less than six hours).

3 - Cool the assembly and transfer the powder to a glass bottle.

At Los Alamos, an accountability weighing of U_3O_8 is made.

REDUCTION AND HYDROFLUORINATION

The U_3O_8 is reduced with hydrogen to UO_2 which is in turn hydrofluorinated to UF_4.

Step 1 - Place one kilogram of uranium as U_3O_8 in a special nickel boat. Position the boat in a special nickel lined cylinder. This cylinder is fitted with soft iron flanges and an annealed copper gasket. The assembly is placed in an electrically heated Heavy Duty Furnace Model M-5024.

2 - The furnace temperature control is set at 660°C and hydrogen is passed through the cylinder at 4 l/min. It is estimated that one hour is required to reach the temperature after which it is maintained for two hours.

3 - Cool the furnace to 550°C with an air blast.

4 - Reduce the hydrogen flow to 3 l/min and pass in preheated HF at 350 g/hr. Hold these conditions five and one-half hours.

5 - Increase the temperature to 750°C and decrease the hydrogen flow to 2 l/min and the HF flow to 200 g/hr. Hold these conditions for two hours.

6 - Cool the furnace with an air blast but maintain the hydrogen and HF flow until the temperature is less than 300°C.

The practice at Oak Ridge has been the consolidation of ignition, reduction, and hydrofluorination into one operation. The desirability of the accountability point at the U_3O_8 stage prompted the people at Los Alamos to reject the consolidation.

REDUCTION TO METAL

Step 1 - Two batches of hydrofluorination product are combined to form one reduction batch. Blend the UF_4 with calcium and iodine in a shielded pressure chamber similar to the one used for reduction. The sturdy construction of the blender is a safety measure should a pre-ignition similar to the one that occurred at Fernald be encountered. The calcium and iodine are added to the extent of 0.1 mole I_2 per mole UF_4 and 25% excess calcium above the stoichiometric amount.

2 - Pour the blended materials into a 4-1/2 in. ϕ x 14 in. Norton specially purified MgO crucible which has been positioned in a pressure chamber not unlike the one in current use at SRP for plutonium reduction.

3 - Fit an annealed copper gasket in position and bolt the pressure chamber lid in place.

4 - Heat the pressure chamber in an Ajax Induction furnace at 10-12 KW. The charge fires in about fifteen minutes. The furnace should be isolated in a heavy iron box to protect operators should the reduction bomb vent itself.

5 - Remove the pressure chamber from the induction coil and allow it to cool to room temperature.

BUTTON HANDLING

Step 1 - Break the crucible away from the pressure chamber walls with a special hydraulic ram. Dump the slag, crucible and button into a placite coated hood and separate the button by hand. Placite affords special protection from I_2 fumes.

2 - Pickle the button in dilute acetic acid.

3 - Submerge the button in water and bore an analytical sample.

SHAPE METALLURGY

The decision as to shape of the final U-233 component cannot be made as yet and it is impossible to outline a definite plan of operation. Discussion with the Los Alamos people, however, indicated that the fabrication installation would be very similar to the present Bldg. 235 "C" line.

The uranium buttons are melted (1325°C) under vacuum in a high purity MgO crucible in an induction furnace. The melt is poured into a graphite mold which has been coated with very fine MgO dust. The initial temperature of the mold is 800 - 1000°C. If pressing is required, the gamma phase of uranium (about 1000°C) is best at about 1000 psi. Alpha phase pressing is feasible at 5-600°C but a pressure of 50-60,000 psi is required. Final shape handling and coating is very similar to that for plutonium.

MISCELLANEOUS

Waste Recovery

The primary waste streams are:

a) Supernates and wash solution from the first precipitation

b) Supernates and wash solution from the second and third precipitation

c) Reduction residues

d) Fabrication and crucibles residues

e) Metal turnings

The handling of each of these waste streams is discussed below:

a) The large amounts of citrate and acetate ions from the first precipitation complicates the recovery. The recovery of uranium from this stream with TBP extraction is currently being investigated.

[redacted]

b) Flow sheets for TBP extraction of these streams are in current use.

c) The fluoride ion in 2250 grams of these residues and is complexted by 5100 grams of $Al(NO_3)_3 9H_2O$ and dissolution is effected by the gradual addition of HNO_3 (70%) and water. When the reaction has subsided the mixture is refluxed for several hours, settled and transferred to TBP recovery.

d) About 2700 grams of these residues are added to 750 grams of $Al(NO_3)_3 9H_2O$ and is dissolved by the careful addition of 9100 ml of HNO_3 (70%). The solution is refluxed for eight hours, filtered and transferred to TBP extraction recovery.

e) Metal turnings from fabrication machining must be dissolved in HNO_3, diluted and sent to the second or third precipitation step.

It is anticipated that only minor changes in the presently installed waste recovery system would be required to convert from plutonium to U-233.

ATOMIC ENERGY DIVISION

William A. Jenkins

William A. Jenkins
Technical Division

WAJ:L

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END

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7/19/94