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R. Kalata
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PLUTONIUM ELECTROPOLISHING CELL

K. S. Bergstresser

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CHEMISTRY AND METALLURGY DIVISION

ANALYTICAL GROUP

Charles F. Metz, Group Leader

ABSTRACT

Improvements in apparatus and procedure, used in the preparation of bright-surfaced samples of plutonium metal for analytical purposes, have been described.

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Plutonium Electropolishing Cell

Bright-surfaced samples of plutonium metal can be obtained for analytical purposes by electropolishing. This process involves anodic oxidation and dissolving of a small portion of the metal, thereby causing a loosening of the surface impurities such as oxide which fall off and expose the bright surface of the metal. A description of apparatus and procedure for preparing plutonium samples in this manner has been given⁽¹⁾. Improvements which were found desirable for both apparatus and method of electropolishing are presented in this report.

The plutonium electropolishing cell is illustrated in Figure 1. The cell with a total height of about 14 cm was made from a mercury-seal type of standard taper 29/42 Pyrex outer joint by making a round-bottom closure on the end opposite the standard taper. Two side arms were placed on the cell, midway between the round bottom and the ground joint and separated from each other around the circumference of the cell by about 90°. One side arm was bent upward, parallel to the cell, and ended in a standard taper 10/30 Pyrex outer joint. This joint was fitted during electrolysis with a 10/30 inner joint in which a wad of glass wool had been inserted. This arrangement permitted escape of gases produced during electropolishing, and at the same time collected any plutonium-containing spray carried along with the gas. The other side arm contained the tungsten wire connected to the cathode and, through a tungsten-Pyrex seal, to a metal cap on the outside end of the closed side arm.

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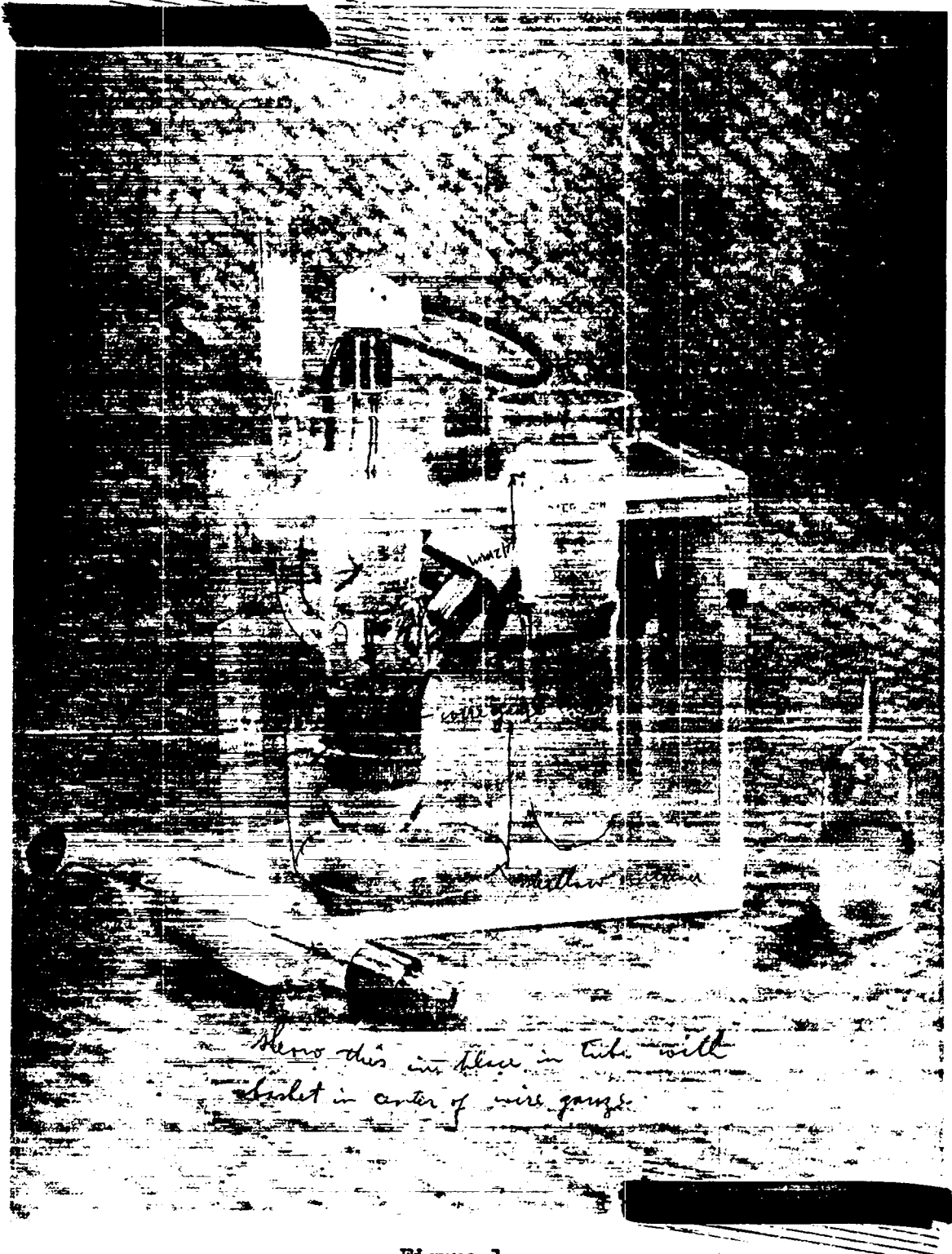


Figure 1

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The electrodes were fabricated from 30 mesh tungsten wire gauze (5 mil diameter wire). The cathode was cylindrical in shape, about 2.5 cm in height, and fitted tightly against the inside wall of the cell. The anode in the form of a 1.4 cm diameter hemisphere was fastened to a 30 mil diameter tungsten wire which was sealed through the lower end of a reduced-tube type of standard taper 29/42 Pyrex inner joint. The upper end of this joint was covered with a metal cap, connected to the 30 mil tungsten wire to make electrical contact with the anode. One of these anode units is shown separately in the foreground in Figure 1.

The electropolishing cell, and another 29/42 standard taper outer joint for holding the cleaned anode unit when not in use, were held in position through 37 mm diameter openings in the top of a plastic box, 10 x 15 x 15 cm. A 70 x 50 mm Pyrex crystallizing dish was placed in the box and underneath the electropolishing cell to collect any plutonium solution in case the cell should be broken accidentally. Necessary electrical connections and a switch were fastened to the sides of the box. For controlling the current during electropolishing a 50-ohm variable resistance and a 0-1 ampere D.C. ammeter were employed. Two storage batteries connected to supply a maximum of 12 volts to the cell were used as a source of direct current.

Electropolishing Procedure

- (1) Place the metal sample in the clean, dry anode basket and insert the anode unit in the electropolishing cell, previously to which has been added a 1:1 mixture of ethylene glycol and 90 % phosphoric acid in sufficient volume to just cover the cathode.

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- (2) When the required electrical connections have been made, electropolish the sample at about 0.1 ampere for 1 to 2 minutes. At times the current may be interrupted because of poor contact between the metal sample and the tungsten wire anode. Electropolishing can be resumed by agitating the anode unit slightly. Unseating the anode unit momentarily from the ground glass joint by lifting it up just a short distance is usually sufficient to renew the electrical contact.
- (3) After turning off the switch and disconnecting the anode unit, remove the latter from the cell, retaining as little of the viscous electrolyte on the tungsten basket as possible. Immediately wash the basket and sample thoroughly with concentrated nitric acid, distilled water, and acetone in this order. When excess acetone has evaporated from the sample and basket, the piece of plutonium metal may be removed and the anode is ready for polishing another sample.

The electropolishing procedure should be performed in a well ventilated hood which is closed as much as possible. It is best to treat only one piece of metal at a time by this process.

Reference

- (1) LA-416, Oct. 1, 1945, "Chemical and Spectrochemical Analysis of Uranium and Plutonium Materials".

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