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A History of the United States Primary Uranium Reference Material – Origin,  
Production, Certification of Uranium Metal SRM 960/CRM 112-A

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# A History of the United States Primary Uranium Reference Material – Origin, Production, Certification of Uranium Metal SRM 960/CRM 112-A

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## ABSTRACT

The New Brunswick Laboratory (NBL) was founded in 1949 by the U.S. Atomic Energy Commission to assist in the development of uranium (and in 1959 plutonium) analytical measurement methods. Over the years, the laboratory developed expertise and analytical methods relating to many aspects of the nuclear fuel cycle and weapons materials. The National Bureau of Standards (currently named the National Institute for Standards and Technology, or NIST) served then and now as the nation's source for Standard Reference Materials (SRMs) and is the certifying authority and the national metrology institute for the United States. Beginning in the early 1950's, the need for a primary uranium reference material became apparent. NBS, NBL and various AEC laboratories collaborated on identifying desired reference materials properties, procuring base materials, developing the necessary chemical and handling techniques, studying chemical forms for suitability and storage as reference materials, and certifying the chemical and physical properties of the produced SRMs. The first uranium standard reference material was a  $U_3O_8$  material. In 1959, the Advisory Committee for Standard Reference Materials and Measurements decided that due to stoichiometry problems with uranium oxides, a pure uranium metal standard was needed. A cropped dingot was purchased from Mallinckrodt Chemical Works and sent to NBL to hold for future use since the company was stopping preparation of pure dingot uranium metal and switching to a process that would yield lower-purity metal. During the early 1960's the material was processed, and in the late 1960's and early 1970's certification measurements were performed. This report details the production and processing of the material and the measurements performed over the years prior to and during the original certification, and the subsequent verification measurements.

## BACKGROUND<sup>1</sup>

**May, 1956** Dr. Clement J. Rodden, Director, New Brunswick Laboratory (NBL) finds that a 276-lb. dingot metal, identified as NBL C-8583, is quite pure. This substantiates his long held idea that "...owing to the problem of the stoichiometry of uranium oxide it would be preferable to have a pure uranium metal to be used as a standard." The use of Dingot C-8583 as a standard was suggested at the Gatlinburg Meeting in 1958. The determination of uranium assay on Dingot C-8583 extended over several years during which time the atomic weight of uranium changed. It was also analyzed spectrographically and chemically for impurities. Dingot C-8583 had a limited distribution with chief users being foreign countries. There were 10 receivers from May 1958 to February 1966, including National Bureau of Standards (NBS), Los Alamos Scientific Laboratory (LASL), United Kingdom, Euratom, and Oak Ridge National Laboratory.

**July 20, 1959** At a meeting of the Advisory Committee for Standard Reference Materials and Methods of Measurement it was suggested that uranium metal replace uranium oxide ( $U_3O_8$ ) as a chemical standard for uranium. It was decided to obtain a cropped dingot of about 1500 lbs. from Mallinckrodt Chemical Works (MCW), St. Charles, MO, to hold for future use since the company was stopping the production of pure dingot uranium metal. They would soon be adding impurities to their dingots.

**RECEIPT AND INITIAL CUTTING OF DINGOT E-8427**

**July 22, 1959** Mallinckrodt Chemical Works, St. Charles, MO, shipped a scalped dingot, number 2111, weighing 1605 lbs. (728 Kg) to NBL, New Brunswick, NJ.<sup>2</sup> The dingot was identified at NBL as E-8427.

**September 11, 1959** A letter from Mallinckrodt Chemical Works, St. Charles, MO, to Clement J. Rodden at NBL lists spectrographic and chemical impurity analyses results for 27 elements and gives the density of Dingot 2111.<sup>3</sup> Data are in Table I.

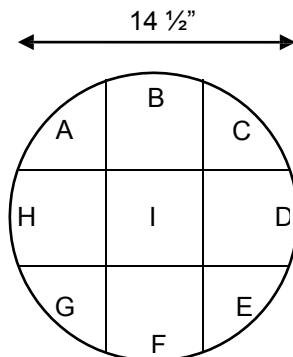
Table I  
Impurity Analyses for MCW Dingot 2111

Spectrographic, ppm							
Ag	<0.1	Cd	0.1	Li	<1	P	<50
Al	<20	Co	<5	Mg	<10	Pb	<5
As	<10	Cr	<5	Mn	<10	Si	18
B	<0.10	Cu	2	Mo	<5	Sn	<5
Be	<0.1	Fe	35	Na	<10	V	<20
Bi	<5	In	<1	Ni	12	Zn	<20

Chemical, ppm	
Nitrogen	11
Hydrogen	2.1
Carbon	11

Density, 19.09 g/cc

**March 5, 1965** NBL shipped the Dingot E-8427, weighing 1605 lbs. (728 Kg), to National Lead Company, Hamilton, OH, to be cut.<sup>4</sup> Instructions for cutting the Dingot were given.<sup>1</sup> Dingot E-8427 was 14½” diameter by 16” long. It was cut in half across the length to obtain two pieces, each 8” long, then each piece was cut into nine pieces and stamped A, B, C, D, E, F, G, H, and I in such a way so that the pieces could be placed together to reconstruct their positions as in the uncut Dingot. The total weight of pieces from one half of the original Dingot was 706 lbs. and for the other half was 744 lbs, for a total weight of 1450 lbs.<sup>1</sup> The terms top, middle and bottom are used in various documents (including later reports of impurities analyses) referring to the location where the pieces are from in the Dingot; however, there is insufficient information to determine to which locations they refer. The diagram below illustrates the cutting instructions and locations of the stamped letters.



**April 21, 1965** National Lead Company, Hamilton, OH, shipped the Dingot E-8427 pieces with a total weight of 1464 lbs. (664 Kg) to NBL, New Brunswick, NJ.<sup>5</sup> Note there is an unexplained 14-lb. discrepancy between the total weight reported in reference 1 (1450 lbs.) and the total weight in the shipping form.

**May, 1965 – February, 1966** Sometime during this time period, NBL performed spectrographic and chemical impurities analyses and uranium analysis on Dingot E-8427.<sup>1</sup> Samples for impurities analyses were taken from pieces A, H, and I, some from Middle and some from Bottom. It is unknown from which of the two halves these pieces were from and to what Middle and Bottom refer. Analysis was done on 14 elements, with n ranging from 2 to 19. Units are not indicated, but ppm is understood as this was typical for these kinds of measurements. The average amount of impurity for each element found on page 10 of the reference is in Table II.

Table II  
Impurities Analysis for Dingot E-8427, pieces A, H, and I

Element	Average ppm	Element	Average ppm
Mg	12	Ca	2
C	19	Cr	5
N	5	Cu	2
Fe	38	Mo	3
Mn	4	Ni	2
Si	47	P	10
Al	20	H	2

Uranium analysis was performed on Dingot E-8427, piece A, Middle and Bottom, by two analysts each using a different method. The results found on page 11 of the reference are in Table III.

Table III  
Uranium Values, in %, for Dingot E-8427, piece A

Sample Location	Analyst A	Analyst B
Bottom Cut	99.97 <sub>7</sub>	
Middle Cut	99.98 <sub>0</sub>	99.966
Middle Cut	99.98 <sub>4</sub>	99.977
Middle Cut	99.96 <sub>9</sub>	99.965
Middle Cut		99.978
Middle Cut		99.962

**PREPARATION INTO NBS STANDARD REFERENCE MATERIAL (SRM) 960**

**August, 1966** In a letter from S. C. T. McDowell, AEC, Washington, DC, to Dr. W. Wayne Meinke, NBS, Washington, DC, Mr. McDowell explains that the AEC Advisory Committee for Standard Reference Materials and Methods of Measurements recommended that NBS obtain NBL dingot metal to be certified and distributed as the primary uranium chemical standard.<sup>6</sup> Mr. McDowell identifies individuals from both organizations to accomplish the substitution of uranium metal for U<sub>3</sub>O<sub>8</sub> as a primary standard. In a letter dated August 23, 1966, Dr. Meinke responds to Mr. McDowell that due to the current heavy workload and the moving of certain laboratories to another location he could not consider further commitments before fiscal year 1968.<sup>7</sup>

**July 19, 1967** The Office of Safeguards and Materials Management, HQ, accepted a proposal from Union Carbide Corporation, Nuclear Division, Paducah Gaseous Diffusion Plant, Paducah, KY (Paducah) to subdivide and package a portion of Dingot E-8427 to serve as a NBS uranium metal standard.<sup>8</sup> After various revisions to Paducah's proposal were made, the final plan was to carry out the work in two phases. Phase I would consist of a test run on a similar material. Phase II would consist of the actual subdividing of a portion of Dingot E-8427.

**September 11, 1968** A report issued on this date describes the completed Phase I work by Paducah.<sup>9</sup> In this phase Paducah selected their own material, Derby D-484, to use a test material to determine the extent of impurity additions and waste allowance during uranium standard preparation. Derby D-484 was hot rolled and hot sheared to obtain pieces 1/8" x 1/8" x 6", weighing approximately 25 grams. An important aspect of this work was to determine if the hot rolling and shearing process introduced impurities to the material. To determine if this would be the case, a slab of metal was sawed off from the center of the derby to use as a source of 10 impurities analysis samples, "Before Rolling and Shearing". Ten impurities samples, "After Rolling and Shearing", were taken at random during the shearing process. Each of the 20 samples was analyzed in duplicate for 43 elements using emission spectrograph and chemistry methods. Only two elements showed a significant difference before and after rolling and shearing. Oxygen showed a gain of 3.1 ppm and nitrogen showed a loss of 2.0 ppm. Parameters for obtaining pieces of the desired dimensions and weight and accounting for loss to scrap were determined and considered acceptable.

**November 14, 1969** A report issued on this date describes results of additional tests performed after Phase I was completed.<sup>10</sup> A test on a small number of Derby D-484 samples was performed to investigate the effect of nitric acid pickling on oxygen and nitrogen concentrations. Oxygen and nitrogen impurity analysis results after pickling were not significantly different from those for samples with mechanical cleaning only. (Mechanical cleaning by filing was a usual procedure at Paducah at this time.) The conclusion was that a cleaning procedure using mechanical, chemical, or a combination of the two methods was probably adequate providing oxide surfaces were removed down to the shiny uranium metal. Another test performed was a gravimetric uranium measurement of the Derby D-484 pieces. Results indicated an impurity content that was in good agreement with the measurements performed in Phase I. The results of Phase I and these additional tests led to Phase II of the Paducah proposal, subdividing a portion of Dingot E-8427 into pieces to serve as a NBS uranium metal standard.

**June 16, 1970** NBL shipped four pieces of Dingot E-8427 to Paducah for processing according to the Phase II plan.<sup>11,12</sup> Pieces and weights are shown in Table IV.

Table IV  
Dingot E-8427 Shipped to Paducah

Piece	lbs.
B	98.5
D	93.25
F	94.25
H	95.5
Total Weight	381.5 (173 Kg)

**January 27, 1971** Paducah shipped to NBS, Washington, DC, 3100 uranium metal standards, 1/8" x 1/8" x 6", each weighing between 23 and 35 grams with an average weight of 28.1 grams, that were subdivided from Dingot E-8427.<sup>13</sup> The total weight of metal shipped was 87.1 Kg. NBS identified the material as R-297.

**March 10, 1971** A report issued on this date describes the completed Phase II work by Paducah.<sup>14</sup> In this phase NBL Dingot E-8427 was hot rolled and sheared into 3100 uranium standards, 1/8" x 1/8" x 6", each weighing between 23 and 35 grams with an average weight 28.1 grams.

As can be seen from the Dingot E-8427 cutting diagram above, the pieces, B, D, F, and H, had curved outer surfaces. These curved surfaces were sawed off to rid the finished metal of potential impurity inclusions. Next a slab was sawed off piece F to serve as a source of impurities analyses samples before rolling and shearing. The weight after all sawing was 156 Kg. The resulting four rectangular blocks were degreased with Freon 113 and pickled in 1:1 nitric acid until shiny (there is no indication of pickling temperature), washed in distilled water and dried with alcohol.

The blocks were heated in an alkaline carbonate salt bath at 1180°F (638°C) and rolled down to 0.125" + 0.031" – 0.000". Sheets were treated with silicone grease to minimize oxidation, and heated to about 500°F (260°C) for shearing into strips. Each strip of the desired size was individually cleaned in the same manner as the blocks were before processing, inspected to preclude laminations, and weighed to the nearest gram. Only strips weighing between 23 and 35 grams were accepted for use as uranium standards.

The uranium standards were assembled in groups of 100 and sealed in heavy plastic. The resulting 31 plastic bags each containing 100 standards each were packaged for shipment in two containers with 1600 standards in one container and 1500 in another, for a total weight of 87.1 Kg, calculated by summing the individual weights of the standards.

Impurities analyses were performed on 10 samples taken from the slab sawed off from piece F before rolling and shearing and 10 samples taken from the strips as they were being sheared. All 20 samples were cleaned in the same manner, equivalent to the way the blocks and standards were cleaning during processing. All 20 samples were analyzed in duplicate for 43 elements. All analyses except for carbon, fluorine, hydrogen, nitrogen, oxygen, and phosphorus were obtained by emission spectrograph.

Total impurity after rolling and shearing was not significantly different from that before rolling and shearing. The only element changing significantly in the rolling and shearing operations was hydrogen, and it showed a slight loss. Only 16 of the 43 elements measured were found in the uranium metal. The sums of detected impurities were 174.55 ppm for metal after rolling and shearing and 174.77 ppm for metal before rolling and shearing. When lower limits of detectability for the other 27 elements are added, the totals are <223.06 ppm after rolling and shearing and <223.28 ppm before rolling and shearing. About two-thirds of the detected impurities are contributed by four elements, Al, Fe, Ni, and Si. Table V shows impurities after rolling and shearing, thus representing the impurities in the final product.

Table V  
Impurities Analyses Results of Uranium Metal Standards After Rolling and Shearing (ug/g)

Ag	<1	Co	<1	Mn	6.7	Sb	<0.5
Al	19.6	Cr	8.0	Mo	2.0	Si	29.5
As	<5	Cu	5.9	N	6.5	Sn	<0.5
Au	<0.3	F	<1	Na	<2	Ta	<0.4
B	0.05	Fe	51.5	Nb	<0.2	Ti	<0.2
Ba	<10	Ge	<0.5	Ni	15.7	Tl	<1
Be	<0.01	H	2.5	O	3.2	V	3.6
Bi	<0.5	In	<1	P	<2	W	<0.3
C	8.6	K	<2	Pb	<0.5	Zn	<10
Ca	<3	Li	<0.2	Rb	<5	Zr	3.1
Cd	<0.1	Mg	8.1	Ru	<0.3	Total	<223.06

**January 4, 1972** A NBS composition analysis report with this date shows results of mass spectrometric analysis on uranium metal R-297 providing an atomic weight value of 238.02894.<sup>15</sup>

**March, 1972** A report of analysis for uranium with a handwritten date of March 1972 for the 3100 rods of uranium metal standard performed at NBS, Washington, DC, provides a value of  $99.9747 \pm 0.0038\%$  uranium.<sup>16</sup> The uncertainty figure represents the 95% confidence interval for the mean, based on 20 degrees of freedom. The analysis method was constant current coulometric titration of uranyl ion [U(VI)] with electrogenerated titanous ion [Ti(III)]. The result of this method was corrected, as is necessary, for the presence of Fe and V as determined at Oak Ridge National Laboratory to obtain this value. Analysis samples were randomly taken from the 31 plastic bags containing the rods.

**May 12, 1972** On this date NBS, Washington, DC, J. Paul Cali, Chief, Office of Standard Reference Materials, issued a Certificate of Analysis for Standard Reference Material 960, Uranium Metal, stating a Uranium Assay of  $99.975 \pm 0.017$  Weight Percent and an Atomic Weight of 238.0289.<sup>17</sup> The uncertainty ascribed to the certified assay value is the 95% confidence interval for a single determination. The new SRM was priced at \$7.25 per unit.<sup>18</sup>

**January 24, 1974** An NBL Report of Analysis with this date for Dingot E-8427 piece E shows the material was analyzed in duplicate spectrographically for impurities.<sup>19</sup> The results are shown in Table VI.

Table VI  
Spectrographic Impurities Analysis Results  
for Dingot E-8427, Piece E

	1	2		1	2		1	2
Ag	0.1	0.1	Cu	5	4	Ni	8	7
Al	18	19	Fe	35	35	Pb	0.2	0.2
B	<0.1	<0.1	Ge	-	-	Sb	<1	<1
Be	<0.5	<0.5	In	-	-	Si	130	75
Bi	<0.1	<0.1	Mg	20	20	Sn	0.2	0.2
Cd	<0.5	<0.5	Mn	9	9	V	-	-
Cr	5	5	Mo	-	-	Zn	<10	<10

**February, 1979** In February, 1979, NBL performed isotopic analysis on Dingot E-8427 using NBS SRM U-005 and NBS SRM U-010 as standards.<sup>20</sup> For  $n = 11$  the isotopic analysis results were:  $\text{Wt}\% \text{ }^{234}\text{U} = 0.00526 \pm 0.00025$ ,  $\text{Wt}\% \text{ }^{235}\text{U} = 0.71142 \pm 0.00028$ ,  $\text{Wt}\% \text{ }^{238}\text{U} = 99.28333 \pm 0.00036$ .

**October 1, 1981** An Interagency Agreement dated September 18, 1981 describes the transfer of storage, accountability and distribution responsibilities of NBS Special Nuclear SRMs from NBS to NBL effective October 1, 1981.<sup>21</sup> A letter to purchasers with this information was issued on August 26, 1981.<sup>22</sup> A NBS Special Nuclear Materials Inventory and Transaction Sheet dated October 1, 1981<sup>23</sup> shows 1800 units of SRM 960 in a column headed "NBS Physical Inv. Aug. 25, '81".

**July 6, 1987** A letter dated August 12, 1986 from Stanley D. Rasberry, NBS, to Carlton D. Bingham, NBL, indicates that NBS is planning to discontinue the Special Nuclear Materials Program and requests that NBL purchase all remaining special nuclear material stock from NBS and become the source of these materials in the future.<sup>24</sup> A letter dated September 24, 1987 from William P. Reed, NBS, to Carleton D. Bingham, NBL, indicates an effective date of July 6, 1987 for the transfer of the Program to NBL and contains an inventory report dated July 2, 1987.<sup>25</sup> This inventory shows a balance of 1151 units of SRM 960.

**October 1, 1987** As of this date, NBL issued new certificates of analysis for the NBS Nuclear SRMs. Some of the materials were given new numbers. NBS SRM 960 became NBL Certified Reference Material (CRM) 112-A. On this date NBL, Argonne, IL, Carleton D. Bingham, Director, issued a Certified Reference Materials Certificate of Analysis for CRM 112-A, Uranium Metal Assay Standard, stating a Uranium Assay of  $99.975 \pm 0.006$  Wt. % and a Relative Atomic Weight of 238.0289.<sup>26</sup> The uncertainty ascribed to the certified assay value is the 95% confidence limit for the mean.

**Late 1996** The inventory of packaged CRM 112-A units was dwindling and the decision was made to reduce the unit size to a nominal 4 grams. In recent years, customers had been requesting a smaller unit size in an effort to minimize waste to reduce waste handling costs. In an effort to provide customers CRM 112-A in the desired quantity, 100 bulk units were cut and packaged to obtain 481 nominally 4-gram units (actual weight approximately 4.5 grams) and 8 nominally 1-gram units. Assay measurements performed by high precision titration on the old 26-gram units and the new 4-gram units yielded results that did not differ significantly from each other. Isotopic measurements performed by thermal ionization mass spectrometry resulted in a relative atomic weight value that reproduced the existing certificate value. Therefore, the certificate for the new unit size CRM would have the same certified values for assay and relative atomic weight as the old.<sup>28</sup>

**September 30, 1998** NBL, Argonne, IL, Margaret E. M. Tolbert, Laboratory Director, issued a Certified Reference Material Certificate of Analysis for CRM 112-A, Uranium Metal Assay Standard, stating a Uranium Assay of  $99.975 \pm 0.006$  Wt. % and a Relative Atomic Mass of 238.0289.<sup>29</sup> The reference material is described as containing approximately 4 grams of uranium in the form of a rod. The CRM was priced at \$568.00 per unit.<sup>30</sup>

**July 10, 2002** A NBL report on this date, Density Measurements of CRM 112-A and Dingot, describes density measurements performed on samples of CRM 112-A and Dingot E-8427.<sup>31</sup> CRM 112-A samples were taken from packaged units of the certified reference material and Dingot E-



8427 samples were taken from the remaining NBL Dingot E-8427 pieces that had not been processed into certified reference material units. The mean value for the density and its 95% confidence interval is reported as  $19.010 \pm 0.089$  g/mL, with seven degrees of freedom.

### DENSITY AND ELEMENTAL ANALYSES COMPARISON

Density measurements were made on this material on three occasions. Table VII contains all the data for comparison purposes.

Table VII  
Comparison of Density Measurements

Analysis place/date	Density, g/cm <sup>3</sup>
1959 MCW Dingot	19.09
2001 NBL Dingot	18.92
2002 NBL Dingot	$19.01 \pm 0.089$ (95% C.I.)

Elemental analyses were performed on this material on four different occasions. Table VIII contains all the data for comparison purposes.

Table VIII  
Comparison of Elemental Analysis

Element	1959 MCW Dingot	1965 NBL Dingot Pieces	1971 Paducah After Cutting	1974 NBL Dingot Piece E	
				1	2
Ag	<0.1		<1	0.1	0.1
Al	<20	20	19.6	18	19
As	<10		<5		
Au			<0.3		
B	<0.10		0.05	<0.1	<0.1
Ba			<10		
Be	<0.1		<0.01	<0.5	<0.5
Bi	<5		<0.5	<0.1	<0.1
C	11	19	8.6		
Ca		2	<3		
Cd	0.1		<0.1	<0.5	<0.5
Co	<5		<1		
Cr	<5	5	8.0	5	5
Cu	2	2	5.9	5	4
F			<1		
Fe	35	38	51.5	35	35
Ge			<0.5		
H	2.1	2	2.5		
In	<1		<1		
K			<2		
Li	<1		<0.2		
Mg	<10	12	8.1	20	20
Mn	<10	4	6.7	9	9
Mo	<5	3	2.0		
N	11	5	6.5		
Na	<10		<2		

Element	1959 MCW Dingot	1965 NBL Dingot Pieces	1971 Paducah After Cutting	1974 NBL Dingot Piece E	
				1	2
Nb			<0.2		
Ni	12	2	15.7	8	7
O			3.2		
P	<50	10	<2		
Pb	<5		<0.5	0.2	0.2
Rb			<5		
Ru			<0.3		
Sb			<0.5	<1	<1
Si	18	47	29.5	130	75
Sn	<5		<0.5	0.2	0.2
Ta			<0.4		
Ti			<0.2		
Tl			<1		
V	<20		3.6		
W			<0.3		
Zn	<20		<10	<10	<10
Zr			3.1		

**September 30, 2010** In 2010, the material was again repackaged and isotopic certification and assay verification measurements were performed at NBL. NBL, Argonne, IL, Jon Neuhoﬀ, Laboratory Director, issued a Certified Reference Material Certificate of Analysis for CRM 112-A, Uranium Metal Assay Standard, stating a Uranium Assay of  $0.99975 \pm 0.00006$  g U/g metal and a Relative Atomic Mass of  $238.028918 \pm 0.000012$ .<sup>32</sup> NBL had performed isotopic certification measurements on a random sampling of metal pieces of one (1) gram. NBL does not guarantee uranium isotopic homogeneity for metal pieces smaller than one gram. Prior to use, surface oxide must be removed to ensure accurate uranium assay values. A suggested procedure is given in the certificate. The reference material is described as a uranium concentration and isotopic standard intended for use in calibration of and/or quality control for uranium analysis methods. Each unit of CRM 112-A consists of metal piece of nominal mass as listed on the container. The CRM was priced at \$1,295 per unit.<sup>33</sup>

### THE DINGOT PROCESS<sup>34,35</sup>

In the mid-1950's Mallinckrodt Chemical Works developed a technique to create a large mass of uranium—theoretically up to 3400 lbs—that bypassed a step in the production of an ingot. Work on the “direct ingot” technique took place at a pilot plant in the St. Louis (Downtown) Plant. The word “dingot” was soon coined by pilot plant staff and it stuck.

The process begins by lining a steel bomb shell (the reaction vessel) with crushed  $MgF_2$ . A charge consisting of a blend of  $UF_4$  and magnesium is packed inside and additional crushed  $MgF_2$  is packed on top of the charge. The bomb lid is bolted in place and the bomb is heated for 9-14 hours at 600-700°C. After air cooling for 2-3 days, the bomb is opened and the contents removed simply by inverting it. The  $MgF_2$  slag mass is easily broken off the outside of the uranium metal either by hand or mechanically. Adhering slag is removed using a chipping hammer.

Chemical analysis of the dingots showed that impurities, namely magnesium and nitrogen, are largely concentrated in the surface layers. In a process called surface scalping up to half an inch of

metal on the sides and bottom and one inch on the top is removed by a machining process using a lathe. This produces “a large volume of high-purity metal that has no internal gradient either in density or in the traces of impurities that remain.”<sup>35</sup> In order to achieve this result all traces of occluded  $MgF_2$  slag must be removed. A torch is used on the uranium metal surface which causes it to darken showing any remaining occluded  $MgF_2$  in sharp color contrast. Machining is continued and the flame test repeated until the dingot passes inspection. The dingot is now ready for use.

## REFERENCES

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3. Letter dated September 11, 1959, from Mallinckrodt Chemical Works, Uranium Division, St. Charles, MO, David F. Oltman, Analytical Department, to Dr. C. J. Rodden, U.S. Atomic Energy Commission, New Brunswick Laboratory, New Brunswick, NJ, Subject: Chemical and Spectrographic Analyses of One Normal Extruded Uranium Dingot as per MCW Production Order 370 (AEC Request 192 dated 6/12/59).
4. Shipping form No. 27, Bill of Lading No. B-2129639.
5. Shipping form No. 12-N, Bill of Lading No. NLO-4019.
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7. Letter dated August 23, 1966, from W. Wayne Meinke, Chief, Office of Standard Reference Materials, to S. C. T. McDowell, Assistant Director for Control, Division of Nuclear Materials Management, AEC, Washington, DC.
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13. Nuclear Material Transfer Report (form AEC-741), January 27, 1971, Shipper: Union Carbide Corporation, Paducah, KY, Receiver: National Bureau of Standards, Washington, DC.
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