U.S. Department of Energy

CRM 115 History Report

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NBL Program Office

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Origin of Material

New Brunswick Laboratory (NBL) CRM 115, Uranium (Depleted) Metal, was originally used in the Safeguards Analytical Laboratory Evaluation (SALE) Program, at that time administered by Allied Chemical in Idaho Falls, ID (Idaho Falls). The source material originated as one of a number of large uranium metal derbies, formed from green salt converted by reduction to metal, by the National Lead Company of Ohio, Cincinnati, Ohio (NLO). One derby, No. 43590, weighing approximately 72.8 Kg, was shipped to The Dow Chemical Company, Rocky Flats Division, Golden, Colorado (Rocky Flats), in 1974, to process into analytical samples for the SALE Program.

"This derby was preheated in a molten salt bath at a temperature of 630˚C. The derby was then rolled to a nominal thickness of 0.125", resulting in a sheet approximately 21" wide and 93" long. This sheet was then sheared into eight (8) strips, each strip with an approximate width of 2.6". As the strips were sheared they were identified with an alpha designation, i.e., A through H. The strips were then cleaned in a nitric acid solution to remove surface impurities. Each of the eight strips was sheared into approximately 0.750" wide samples. Each strip yielded from 121 to 124 samples and each sample was impression-stamped with its appropriate identification number; for example, A-1 through A-124 were the numbers of the samples from the "A" strip. The final shearing step resulted in samples of the required 75 ± 15 grams."[1]

The final weight of all the samples was approximately 72.1 Kg. Nine samples, identified in a drawing[1], were sent to NLO for analysis. Rocky Flats retained nine samples with adjacent numbers for analysis. All the remaining samples were sent to Idaho Falls. Table I shows the identification numbers of the samples sent to NLO and retained at Rocky Flats.

Table I
Identification Numbers of Samples Sent to
NLO and Retained at Rocky Flats

NLO			**Rocky Flats**		
A-1	D-1	H-1	A-2	D-2	H-2
A-62	D-61	H-61	A-61	D-62	H-62
A-124	D-122	H-122	A-123	D-121	H-121

Analysis of Material

Samples were analyzed by five laboratories from 1974 to 1978: NLO, Idaho Falls, Rocky Flats, NBL and National Bureau of Standards (NBS) (now National Institute of Standards and Technology). NBS standards were used as controls by all laboratories. Note that sample

identification numbers on analysis reports do not always agree with the sample identification numbers given by Rocky Flats above. Records explaining these discrepancies are not available.

NLO reported data December 31, 1974.[2] A reference in the letter accompanying the data indicates that the analyses were done for characterization of the metal. Uranium assay was obtained by potentiometric titrations. Complete isotopic measurements were obtained using thermal analysis and, additionally, gas mass spectrometry was used for U-235 content only. Table II contains these data.

Table II
NLO Uranium Assay and Isotopic Results

Sample ID	U Assay, %	U-234, Wt. %	U-235, Wt. %	U-236, Wt. %	U-238, Wt. %	U-235, Wt. %, Gas
A-1	99.974	0.0007	0.201	0.0036	99.796	0.2003
A-124	99.973	0.0007	0.201	0.0034	99.795	0.2007
D-61	99.969	0.0008	0.200	0.0032	99.797	0.2004
H-1	99.967	0.0007	0.200	0.0033	99.796	0.2004
H-61	99.978	0.0007	0.202	0.0033	99.794	0.2005
H-122	99.976	0.0007	0.200	0.0033	99.796	0.2004

NLO performed impurities analysis on three samples for 47 elements. The test method(s) used were not identified in the report. Table III contains the elements with measurable quantities (excludes hydrogen, oxygen, nitrogen). Data for all elements are found in reference 2.

Table III
NLO Impurity Content for Selected Impurities

Element	ppm
Aluminum	9
Calcium	2
Carbon	23-26
Copper	6
Iron	34-35
Lead	15-16
Magnesium	6-7
Manganese	7
Nickel	8
Silicon	32-35

Idaho Falls reported data sometime after October 7, 1975.[3] The request, for uranium isotopic analysis, is dated “10-7-75”, and the sheet with handwritten results does not have a date. The analysis method was not identified. It is not clear that this analysis was performed for characterization because the request for sample analysis appears to be a form used for any request for analysis. However, since this is the laboratory that was administering the SALE program, for which this material was intended, one would think that these analyses were for characterization purposes. Table IV contains the reported data.

Table IV
Idaho Falls Isotopic Results

Sample ID	Replicate	U-234, At. %	U-235, At. %	U-236, At. %	U-238, At. %
A-2	1st	0.0010	0.2085	0.0038	99.7867
A-2	2nd	0.0014	0.2054	0.0043	99.7890
A-123	1st	0.0013	0.2050	0.0038	99.7900
A-123	2nd	0.0013	0.2054	0.0042	99.7890
D-62	1st	0.0012	0.2079	0.0040	99.7869
D-62	2nd	0.0012	0.2050	0.0039	99.7899
D-121	1st	0.0012	0.2071	0.0039	99.7878
D-121	2nd	0.0012	0.2055	0.0039	99.7894
H-2	1st	0.0020	0.2235	0.0056	99.7689
H-2	2nd	0.0020	0.2255	0.0057	99.7668
H-121	1st	0.0020	0.2266	0.0057	99.7657
H-121	2nd	0.0019	0.2115	0.0053	99.7813

Rocky Flats, operated by Rockwell International, Atomics International Division, at the time analysis was performed, reported data December 5, 1975.[4] The analysis methods were not identified, but the cover letter states that the "NBL 960 … was used as a primary standard for the ceric sulfate factor.", which implies the assay was performed by titration. Table V contains uranium assay and isotopic values.

Table 5
Rocky Flats Uranium Assay and Isotopic Results

Sample ID	U Assay, %	U-233, Wt. %	U-234, Wt. %	U-235, Wt. %	U-236, Wt. %	U-238, Wt. %
A-2	99.927	0	<.01	.200	.011	99.789
A-61	99.986	0	<.01	.201	.005	99.794
A-123	99.955	0	.000	.202	.010	99.788
D-2	99.950	0	<.01	.204	.008	99.788
D-62	99.939	0	<.01	.203	.005	99.792
D-121	99.925	0	<.01	.203	.009	99.788
H-2	99.973	0	<.01	.201	.004	99.795
H-62	99.957	0	<.01	.201	.006	99.793
H-121	99.948	0	<.01	.207	.012	99.781

NBL reported data in November, 1975 identifying the work as "characterization studies".[5] NBL gave identification numbers to the samples as shown in Table VI. Uranium assay analysis was done by High Precision Titration. The isotopic analysis method was not identified. Table VII contains the results.

Table VI
NBL Sample Identification Numbers

Rocky Flats No.	NBL No.
A-1	DU-280
A-124	DU-281
D-61	DU-283
D-122	DU-284
H-1	DU-287
H-122	DU-288

Table VII
NBL Uranium Assay and Isotopic Results

Sample ID	U Assay, %	U-234, Wt. %	U-235, Wt. %	U-236, Wt. %	U-238, Wt. %
A-1	99.975	0.0007	0.2007	0.0038	99.7948
A-1	--	0.0007	0.2009	0.0038	99.7946
A-1	--	0.0008	0.2005	0.0041	99.7946
A-124	99.982	0.0006	0.2010	0.0037	99.7947
A-124	--	0.0007	0.2013	0.0036	99.7944
A-124	--	0.0007	0.2009	0.0038	99.7947
D-61	99.978	0.0007	0.2004	0.0038	99.7951
D-61	--	0.0006	0.2013	0.0040	99.7941
D-122	99.973	0.0007	0.2009	0.0038	99.7946
D-122	--	0.0008	0.2011	0.0039	99.7942
H-1	99.982	0.0007	0.2003	0.0039	99.7950
H-1	--	0.0007	0.2006	0.0036	99.7951
H-122	99.972	0.0007	0.2007	0.0038	99.7947
H-122	--	0.0007	0.2013	0.0038	99.7942

NBS reported results for uranium assay by the NBL High Precision Titrimetric method on July 27, 1978.[6] Two analysts completed a total of 26 titrations. The material was found to be homogeneous and contain 99.97% ($\sigma = 0.01$) uranium (as natural uranium, correction to atomic weight of the material was not made). Table VIII contains the results.

Table VIII
NBS Uranium Assay (as Natural Uranium) Results

Sample ID	Average U Assay, %
A-1	99.966
A-124	99.967
D-61	99.963
D-122	99.976
H-1	99.970
H-122	99.966

Certification
NBL issued a Certificate of Analysis for NBL Reference Material No. 115, Uranium (Depleted) Metal (Uranium and Uranium-235 Standard) in June, 1978.[7] The data used to establish certified values were the NBL analysis data (see above). The material was certified for uranium weight percent, uranium-235 weight percent and relative atomic weight. NBL later reformatted Certificates of Analysis and re-issued the 1978 certificate.[8] The material name changed to CRM 115, Uranium (Depleted) Metal (Uranium and Uranium-235 Standard).

1993 Packaging
In 1993 NBL had no stock of packaged units of CRM 115. By this time, the source material, 75-gram pieces, originally at Idaho Falls, was all located at NBL and identified as NBL number 82DU0914. All pieces from LIMS subsplit numbers 004, 008, and 009 were used to package 102 units.[9] The letter/number designation of the pieces were not recorded. Each piece was placed into a Nalgene vial, the vial labeled, heat sealed in a plastic bag, and placed in a screw-cap cardboard tube. The outer tube was labeled.

2001-2002 Packaging and Verification of Uranium Assay and U-235 Content
In 2001 once again NBL no longer had stock of packaged units of CRM 115. At this time about 30 Kg of source material, NBL number 82DU0914, was available. A Scope of Work was written for packaging approximately 100 units, performing uranium assay and isotopic verification analysis, and density and impurity measurements.[10]

Packaging
Bottle number 18 containing pieces from strip "B" was selected from the source material. Each piece was placed into a Nalgene vial, the vial labeled, heat sealed in plastic, and placed in a large size blue cardboard container. The outer container was labeled. As each of the 103 pieces was packaged, they were sequentially numbered in the order of packaging and the letter/number designation was recorded. In two cases, two pieces were found with the same letter/number designation (B11 and B23).[11]

Sampling, Uranium Assay and Isotopic Measurements
Four of the packaged pieces were selected at random for all analyses. Uranium assay and isotopic measurement results are discussed in a statistical report.[12] Uranium assay was performed using the NBL Titrimetric Method and the results were in agreement with the certificate value. Measured uranium-235 values performed on the Triton and the MAT 261 instruments were in agreement with each other, but not in agreement with the certificate value. Table IX summarizes the isotopic data.[13]

Table IX
NBL 2001 CRM 115 Isotopic Data

	Atom % U-235	**Uncertainty**
Triton	0.202913	0.000052 (2 Std Dev)
MAT 261	0.20278	0.00036 (2 Std Dev)
Certificate	0.2034	0.0002 (95% C.I.)

Impurities Analysis

Trace element analysis for 13 elements, aluminum, cadmium, calcium, chromium, copper, iron, magnesium, manganese, molybdenum, nickel, sodium, vanadium, zinc, was performed by ICP-AES.[14] All elements, except aluminum, calcium, sodium and zinc, showed results reasonably close to the impurities analysis performed by NLO in 1974.[2]

Density Measurement

Density measurements were performed using a Micromeritics/AccuPyc 1330 Pycnometer, yielding a result of 18.9 g/cm^3.[15]

2002 Recertification

On July 31, 2002, NBL issued a new certificate for CRM 115, certifying the uranium assay and relative atomic weight as they were on the original 1978 certificate.[16] Isotopic values were given as information only because the data obtained in the 2001 analysis did not verify the 1978 certificate values.

2011 Packaging, Uranium Assay Verification and Isotopic Characterization

In 2009 another packaging and certification of CRM 115 was proposed. To be completed in 2012, this effort will provide users with the availability of approximately 1-gram pieces of CRM 115 in addition to the 75-gram pieces as well as complete isotopic data. The previously certified uranium assay value was verified through analysis by high precision titration. Full isotopic characterization was obtained though analysis by MAT 261 and Triton mass spectrometry instruments. A final report of this work, *Project Report for Certified Reference Material 115: 1-Gram Unit Production and Certification of U Amount Content and U Isotope-Amount Ratios*, will be available in the NBL CRM 115 file. [NOTE: Report inserted at end of this document on May 23, 2013.]

2012 Recertification

In 2012, NBL will issue a new certificate for CRM 115, *New Brunswick Laboratory U.S. Department of Energy Certificate of Analysis CRM 115 Uranium (depleted) Metal Assay and Isotopic Standard*, certifying the uranium assay and complete isotopics and molar mass (relative atomic weight) based on the 2011 characterization.

References

1. Letter and attachment dated July 17, 1974, from L. E. Wilson, QC-Quality Engineering, The Dow Chemical Company, Rocky Flats Division, Golden, CO, to L. M. Levy, Manager, Advanced Planning Department, National Lead Company of Ohio, Cincinnati, OH.
2. Letter and attachments dated December 31, 1974, from J. W. Robinson, National Lead Company, Cincinnati, OH, to L. M. Levy.
3. Report with attachment on Allied Chemical Corporation, Idaho Chemical Programs Operations Office, Request for Analysis by Analytical Chemistry Branch, SALE Depleted U Metal, log no. 75-5441, submitted by D. M. Lund on 10-7-75.
4. Letter with attachment dated December 5, 1975, from H. C. Anderson, Manager, Service Laboratories, Rockwell International, atomics International Division, Rocky Flats Plant, Golden, CO, to Mr. Dave Lund, Allied Chemical Corporation, Idaho Falls, ID.

5. Letter with attachments dated November 19, 1975, from Carleton D. Bingham, Director, U. S. Energy Research and Development Administration, New Brunswick Laboratory, New Brunswick, NJ, to Mr. D. M. Lund, Analytical Research Section Allied Chemical, Idaho Falls, ID.
6. Memorandum with attachments dated July 27, 1978, from J. R. Moody, Inorganic Analytical Research Division, U. S. Department of Commerce, National Bureau of Standards, Washington, D.C., to I. L. Barnes.
7. U.S. Department of Energy, New Brunswick Laboratory, Certificate of Analysis, NBL Reference Material No. 115, Uranium (Depleted) Metal (Uranium and Uranium-235 Standard), June, 1978, Argonne, IL.
8. U.S. Department of Energy, New Brunswick Laboratory Certified Reference Materials Certificate of Analysis, CRM 115, Uranium (Depleted) Metal (Uranium and Uranium-235 Standard), June 1978, Argonne, IL.
9. CRM 115 Packaging, November 3, 1993.
10. U.S. Department of Energy, New Brunswick Laboratory, Scope of Work for Packaging and Verification of CRM 115, April 25, 2001, from Usha I. Narayanan, Reference Materials Program Manager, to Jon Neuhoff, NSND Director.
11. Bottling C115 in C068.
12. Verification of CRM 115, Willard C. Losinger, 15 May, 2001.
13. CRM 115-NDA Report of Verification Analysis, Date of Revised Report, 03/08/02, S. Richter.
14. Report of Trace Element Analysis of C115 Samples by ICP-AES, Pat Santoliquido.
15. Department of Energy, New Brunswick Laboratory, Density Determination, September 7, 2001, from Marie E. Morales-Arteago to Iris Frank.
16. U.S. Department of Energy, New Brunswick Laboratory, Certified Reference Material Certificate of Analysis, CRM 115, Uranium (Depleted) Metal (Uranium Assay Standard), July 31, 2002, Argonne, IL.