A random sample of the units was taken for uranium assay (elemental concentration) and isotopic abundance analyses. The uranium assay was determined by the NBL High Precision Titrimetric Method using National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 136e, Potassium Dichromate ($K_2Cr_2O_7$) Oxidimetric Standard, as the titrant. NBL &112A, Uranium Metal Assay Standard, and NBL &829, Uranium Oxide (U_3O_8) Assay Standard were used as controls to verify proper performance of the measurement systems. Uranium assay measurements were performed by two analysts each using independent titration systems. Prior to titration preparation, &829A analysis samples were ignited at 800°C to constant weight to determine the recommended procedure for ignition.

The uranium isotopic composition and the relative atomic mass of uranium were determined by thermal ionization mass spectrometry (TIMS). The following relative atomic masses were used in calculations: ^{234}U - 234.0409456, ^{235}U - 235.0439231, ^{236}U - 236.0455619, and ^{238}U - 238.0507826. Uranium isotopic ratio measurements were performed by two analysts each using a different mass spectrometer. One TIMS instrument, utilizing the Total Evaporation procedure, was used to generate values for the certification of only the ^{235}U/^{238}U ratio. A second TIMS instrument, utilizing the NBL-Modified Total Evaporation procedure, was used to generate values for the ^{235}U/^{238}U ratio. The ^{234}U/^{238}U and ^{236}U/^{238}U UDWLRV ZHUH GHWHUPLQHG XVLQJ WKH 0RGLILHG 7RWDO (YDSRUDWLRQ DQDO\VLV PHWKRG RQ WKH VDPH LQVWUXPHQW 'DWD ZHUH DFTXLUHG XVLQJ KLJK VLJQDO-LQWHQVLW\ VWDWLF PXOWL-FROOHFWRU PHWKRG ZLWK WKH ^{236}U PHDVXUHG XVLQJ D VHFRQGDU\ HOHFWURQ PXOWLSOLHU. The minor ratios were corrected internally using the ^{235}U/^{238}U ratio determined by the Total Evaporation and NBL Modified Total Evaporation methods. Mass discrimination correction factors applied to measured &829A ^{235}U/^{238}U isotopic ratios were determined from multiple analyses of NBL U030A, Uranium Isotope Standard (3% enriched), run sequentially with &829A. Measurements of NBL U500, Uranium Isotopic Standard (50% enriched), were used as a control to verify proper performance of the measurement system for the ^{235}U/^{238}U measurements. Measurements of NBL U010, Uranium Isotopic Standard (1.0% enriched), were used as a control to verify proper performance of the measurement system for the ^{234}U/^{238}U and ^{236}U/^{238}U measurements. Mass spectrometric measurements indicate that there is no detectable ^{233}U and no significant heterogeneity in the isotopic abundances of ^{234}U, ^{235}U, and ^{238}U within and between units. Isotopic heterogeneity was, however, observed in the abundance of ^{236}U. The uncertainties calculated for the ^{236}U isotopic abundance and ^{236}U/^{238}U isotopic ratio incorporate the observed variability.

The expanded uncertainty (U) for a certified property of &829A defines an interval around the value of the property and is calculated according to the ANSI/NCSL Guide[1]. The magnitude of this interval is obtained by multiplying the combined standard uncertainty (u_C) by a coverage factor (k). The coverage factor, k, is the Student's t factor based on the effective degrees of freedom to provide a 95% level of confidence. The combined standard uncertainty (u_C) for uranium assay consists of Type A components derived from standard deviations associated with analyst-to-analyst differences and titration measurements; and a Type B component based on the standard uncertainty taken from the NIST SRM 136e certificate. The combined standard uncertainties (u_C) for uranium isotopic parameters consist of Type A components derived from standard deviations associated with isotopic ratio measurements of the samples and the measurements of the ^{235}U/^{238}U ratio of NBL U030A, and estimates of isotopic inhomogeneity of the samples; and a Type B component based on the standard uncertainty derived from the uncertainties associated with the NBL U030A certified value for the ^{235}U/^{238}U ratio.

Project coordination was provided by A. M. Voeks. High precision titrimetric assay measurements were performed by G. J. Orlowicz and A. M. Voeks. Isotopic abundance measurements were performed by R. M. Essex and S. Richter. Health physics support was provided by H. S. Gruhn. The statistical plan of analysis for assay measurements was prepared by M. D. Soriano and for isotopic measurements by R. M. Essex and S. A. Goldberg. The statistical evaluation of data was performed by M. D. Soriano and reviewed by W. C. Losinger. Technical guidance for C129A certification and issuance was provided by M. A. Legel and U. I. Narayanan. Project supervision was provided by S. A. Goldberg, U. I. Narayanan and J. W. Neuhoff.

Expiration of Certificate: The NBL Program Office has produced and evaluated uranium oxide reference materials stored for extended periods, exceeding 40 years. When stored in its original, unopened container, the certification of this material is valid indefinitely. The certification is nullified if the material or container is damaged, contaminated or otherwise modified. The NBL PO will periodically monitor the materials in inventory and notify customers should degradation be detected.

Stability and Storage: To maintain the integrity of an unused CRM unit, it should remain in the original packaging and should be stored in a dry, temperature controlled location.

[1] American National Standard for Calibration - U.S. Guide to the Expression of Uncertainty in Measurement [GUM], ANSI/NCSL Z540-2-1997.