National Nuclear Security Administration

Nuclear Reference Material Program

U.S. Department of Energy

Certificate of Analysis

Certified Reference Material 145

Uranium Nitrate Assay and Isotopic Standard, 0.7 % U-235

Description: Certified Reference Material (CRM) 145 is a uranium mass fraction and isotope-amount ratio standard. Each unit of CRM 145 consists of approximately 20 mL of uranyl nitrate solution in 1 mol L^{-1} nitric acid, contained in a sealed glass ampoule. Certified isotope composition and mass fraction values are reported in Table 1.

Table 1. Certified Property Values and Uncertainties [(a) (b)]

	$g\ g^{-1}$		$g\ mol^{-1}$
Uranium Mass Fraction:	0.0101356	Uranium Molar Mass:	238.028918
Uncertainty:	0.0000011	Uncertainty:	0.000012
	$n(^{234}U)/n(^{238}U)$	$n(^{235}U)/n(^{238}U)$	
Isotope-Amount Ratio:	0.000052841	0.0072543	
Uncertainty:	0.000000082	0.0000040	
	$n(^{234}U)/n(U)$	$n(^{235}U)/n(U)$	$n(^{238}U)/n(U)$
Isotope-Amount Fraction (•100):	0.0052458	0.72017	99.27458
Uncertainty:	0.0000081	0.00039	0.00039
	$m(^{234}U)/m(U)$	$m(^{235}U)/m(U)$	$m(^{238}U)/m(U)$
Isotope-Mass Fraction (•100):	0.0051579	0.71114	99.28370
Uncertainty:	0.0000080	0.00038	0.00038

[(a)] ^{233}U and ^{236}U were not detected. The limit of detection of uranium ratios for the technique used is 5×10^{-9}.

[(b)] All uncertainties for the certified values are expressed as expanded uncertainties (U) where $U = k\cdot u_c$, where u_c is the combined standard uncertainty and the coverage factor $k = 2$.

Intended use: CRM 145 is a uranium solution mass fraction and isotope standard intended for use in calibration of and/or quality control for uranium analysis methods. To ensure proper uncertainty propagation, it is recommended that isotope-amount ratios and associated uncertainties be used for calculations incorporating CRM 145 values.

Storage: This material should be stored in its original packaging under normal laboratory environmental conditions.

Period of validity: When stored in its original, unopened ampoule, the certification of this material is valid indefinitely. The certification is nullified if the material or container is damaged, contaminated or otherwise modified. The National Nuclear Security Administration's (NNSA) Nuclear Reference Material Program (NRMP) will notify customers should degradation be detected.

Minimum sample size: The material is considered a pure solution, and thus no minimum sample size is declared.

Instructions for handling: The material in the unit is radioactive. The ampoule should be handled under proper radiologically-controlled conditions at all times. This radioactive material should be handled only by qualified individuals. To minimize personnel exposure, appropriate facilities and personal protective equipment should be used. Refer to the Safety Data Sheet for further information.

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Richard Essex, Director
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Nuclear Reference Material Program
Germantown, Maryland
NRMPsales@nnsa.doe.gov

Users are cautioned that once the vial is opened, the uranium concentration and/or isotopic composition of the material may be affected by evaporative losses or environmental contamination. Users should take appropriate precautions to safeguard the material before, during, and after use to ensure valid certificate values.

Recommended Procedure for Ampoule Opening and Dispensing of Solution

1. The ampoule contains a strongly acidic solution of uranium. Appropriate precautions should be taken.
2. Before opening the ampoule, ensure that any dried uranium or condensed liquid in the neck or body of the ampoule is re-dissolved into solution. This can be accomplished by inverting the ampoule repeatedly.
3. The glass ampoules are scored at the neck for ease of opening. However, glass burrs and fragments pose a cut hazard to anyone opening the ampoules. Appropriate precautions should be taken.
4. Lightly moisten the scored line on the neck with distilled water to help ensure a clean break at the score.
5. Because of the narrow neck of the ampoule, it may be difficult or impossible to pour the solution out. Here is one possible method for dispensing:
    a. Obtain approximately 12 cm length of plastic capillary tubing (e.g. i.d. of 0.1 in, o.d. of 0.16 in).
    b. Insert one end of the capillary tubing fully into the ampoule
    c. Fold the remaining length of tubing along the outside of the ampoule, ensuring that the tube is not crimped and will allow the free flow of air through the tube and into the ampoule.
    d. Holding the ampoule and tubing in one hand, and a beaker or dispensing bottle in another, invert the ampoule over the container allowing the solution to drain into it.
    e. The capillary tubing allows air to flow into the ampoule, eliminating the “airlock” created by the narrow neck of the open ampoule.
6. The user should be wary of evaporative losses once the ampoule is opened and prevent uranium contamination of the sample. It is recommended that the entire solution be accurately weighed and aliquanted as soon as possible after opening the sample. Precautions should be taken (clean glass/plastic ware, air filtration, etc.) to prevent uranium contamination of the CRM with subsequent perturbation of the isotopic composition.

Traceability statement: Certified isotope-amount ratio and amount fraction values are traceable to the SI unit mole. The mass fraction and isotope-mass fraction property values are traceable to the SI unit kilogram.

Measurement uncertainty: Uncertainties were determined according to the JCGM 100:2008 Guide to the Expression of Uncertainty in Measurement. The coverage factor of 2 was chosen to provide an approximate 95 % level of confidence. The input quantities associated with the uranium mass fraction included uncertainties due to weighing, CRM 112A purity, and buoyancy factors. The input quantities associated with the uranium isotopic composition included uncertainties from the certified value for CRM U030A, measurement precision, and background corrections associated with the analytical techniques.

Additional information: The CRM was produced by dissolving uranium metal in a single batch and container, with extensive mixing of the resultant solution followed by dispensation into individual ampoules. Subsequent measurements of a random sampling of the total lot produced did not indicate any inhomogeneity in uranium concentration or isotope composition. The minimum sample sizes taken from packaged units and measured were 30 mg U by titration and 1 µg U by a Thermal Ionization Mass Spectrometer (TIMS). The Nuclear Reference Material Program (NRMP) makes no recommendation as to the minimum sample size to be used to ensure concentration or isotopic homogeneity.

The certified uranium content value is based on the mass of high-purity metal dissolved and diluted to a known solution mass. The stated uranium mass fraction was calculated as the prepared value and verified experimentally by the NBL-modified Davies and Gray titration. The certified uranium isotopic composition and atomic weight is based upon measurements performed on multiple samples by two different measurement techniques on TIMS, calibrated using CRM U030A as primary comparator and CRM 129A as a quality control sample. The isotopic values are shared with CRM 112A, uranium (normal) metal standard, which was the source of uranium used to produce the solutions.

In 2016, the New Brunswick Laboratory facility was transitioned to a program office within the Department of Energy and is now operating within the National Nuclear Security Administration (NNSA) as the Nuclear Reference Material Program (NRMP).