NBL Program Office

U.S. Department of Energy

Certificate of Analysis

Certified Reference Material C145
Uranyl Nitrate Assay and Isotopic Solution, 0.7% U-235

Uranium Mass Fraction: **0.0101356 g U/g solution ± 0.0000011 g U/g solution**
[10.1356 mg U/g solution ± 0.0011 mg U/g solution]

	$^{234}U/^{238}U$	$^{235}U/^{238}U$
Atom Ratio:	**0.000052841**	**0.0072543**
Atom Ratio Uncertainty:	**0.000000082**	**0.0000040**

	^{234}U	^{235}U	^{238}U
Atom Percent:	**0.0052458**	**0.72017**	**99.27458**
Atom Percent Uncertainty:	**0.0000081**	**0.00039**	**0.00039**
Weight Percent:	**0.0051579**	**0.71114**	**99.28370**
Weight Percent Uncertainty:	**0.0000080**	**0.00038**	**0.00038**

Relative Atomic Weight: **238.028918**
Relative Atomic Weight Uncertainty: **0.000012**

Note: ^{233}U and ^{236}U were not detected. The detection limit of uranium ratios for the technique used is 5×10^{-9}.

This Certified Reference Material (CRM) is a uranium concentration and isotopic solution standard intended for use in calibration of and/or quality control for uranium analysis methods. Each unit of C145 consists of approximately 20-mL of uranyl nitrate solution in 1M nitric acid, contained in a sealed glass ampoule.

NOTE: The vial should be handled under proper radiologically-controlled conditions at all times.

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 concentration 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 a Thermal Ionization Mass Spectrometer (TIMS), calibrated using CRM U030-A as primary comparator and CRM 129-A as a quality control sample. The isotopic values are shared with CRM 112-A, uranium (normal) metal standard which was the source of uranium used to produce the solutions.

All uncertainties for the certified values are expressed as expanded uncertainties (U) where $U = k*u_C$, where u_C is the

combined standard uncertainty and the coverage factor k = 2. 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 content included uncertainties due to weighing, CRM 112-A purity, and buoyancy factors. The input quantities associated with the uranium isotopic composition included uncertainties from the certified value for CRM U030-A, measurement precision, and background corrections associated with the analytical techniques.

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 bottles. Subsequent measurements of a random sampling of the total lot produced did not indicate any inhomogeneity in uranium concentration or isotopic composition. The minimum sample sizes taken from packaged units and measured were 30 mg U by titration and 1 µg U by TIMS. The NBL makes no recommendation as to the minimum sample size to be used to ensure concentration or isotopic homogeneity.

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. User's should take appropriate precautions to safeguard the material before, during and after use to ensure valid certificate values.

Recommended Procedure for Ampoule Handling 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:
    a. Obtain approximately 12-cm length of plastic capillary tubing (e.g. i.d. of 0.1", o.d. of 0.16").
    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.

Expiration of Certificate: When stored in its original, unopened container, the certification of this material is valid indefinitely. The NBL PO will periodically monitor the materials in inventory and notify customers should degradation be detected.

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

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