ORNL/TM-2021/1915

New Capability at ORNL: High-Precision Uranium Titration

Kayron Rogers
Ben Roach
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February 2021

OAK RIDGE National Laboratory

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Nuclear Analytical Chemistry

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Prepared by
OAK RIDGE NATIONAL LABORATORY
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managed by
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for the
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ACRONYMS

ACTS	Assessment and Commitment Tracking System
AM	analytical method
CRM	certified reference material
CSD	Chemical Sciences Division
GUM	Guide to Uncertainty in Measurement
HPT	high-precision titration
NAC	Nuclear Analytical Chemistry
NBL	New Brunswick Laboratory
NBLPO	New Brunswick Laboratory Program Office
ORNL	Oak Ridge National Laboratory
RM	reference material

1. INTRODUCTION

Destructive analytical measurements establish a nuclear facility's nuclear material inventory and inventory differences for Nuclear Material Accountancy and Control. A nuclear laboratory's ability to perform accurate high-precision analytical measurements is key for tracking large inventories within a facility's material balance areas and during production to track material movement through dynamic processes. For uranium, these measurements are made by using several established high-precision measurement protocols. These include isotope dilution mass spectrometry, gravimetry, and potentiometric titrations. Whatever measurement technique a nuclear lab chooses to use, reference materials (RMs) with certified attributes and accompanying uncertainties are used to calibrate measurement systems, and are the cornerstone for accurate results. In addition to calibration, RMs provide for metrological traceability, are used for method development and validation, and thus provide critical evaluations of the appropriateness and performance of analytical processes used. Evaluations may include validation that a method is fit-for-purpose, quantification of systematic and random biases, and the evaluation of long-term and short-term performance metrics. High-precision measurement techniques require that measured attributes be certified to a high degree of precision in the RMs used—ultimately, to a higher degree than that of the measurement technique itself.

The US authority on the production of special nuclear material Certified Reference Materials is the NBL Program Office (NBLPO), formally known as New Brunswick Laboratory (NBL). The NBLPO is responsible for the sales and distribution of existing NBL certified reference materials (CRMs) and for the production of the next generation of nuclear RMs. To accomplish its mission, NBLPO is establishing key base capabilities within the DOE laboratory complex that formerly existed at the NBL laboratory. The Nuclear Analytical Chemistry (NAC) section within the Chemical Sciences Division (CSD) at Oak Ridge National Laboratory (ORNL) is currently working with NBLPO to set up laboratory and measurement capabilities to provide measurements and capabilities for production and/or recertification of existing and future CRMs for uranium assay. The NBL-developed high-precision titration (HPT) method is a critically-evaluated, extremely precise and accurate primary method utilized for the determination of uranium content in a variety of uranium materials. The HPT method, combined with detailed balance weighing protocols, provides for an analytical methodology that is unsurpassed in precision and one in which all sources of error have been evaluated, a requirement of CRM certification. HPT capability within the United States was lost with the closure of the labs at NBL. The NAC has been collaborating with NBLPO to stand up and demonstrate the capability to perform uranium assay via HPT at ORNL. HPT can produce results with an expanded uncertainty of approximately 0.01% for pure uranium compounds, with typical precisions of <0.006%.

The major tasks required to stand up the method at ORNL were the refurbishment of a dedicated lab and equipment setup, procedure development, analyst training, establishing method-specific quality assurance, and qualification of an analyst to perform the method. This report summarizes these tasks, outlines the documents drafted, and gives the outcome of the qualification titrations.

2. LABORATORY SETUP

The NAC manages several laboratory spaces in Building 4501 for its analytical projects and programs. Room 218 is a 308 ft^2 laboratory space that contained legacy equipment, some contaminated with radioactivity, for projects no longer supported by the Radioactive Materials Analytical Laboratory group. The laboratory is designated as a C-zone space and has two chemical fume hoods, one 8-foot and one 4-foot, approved for radiological work. For repurposing into a lab dedicated to HPT, the space was deemed suitable to be cleaned, refurbished, and then equipped. The greatest challenge to the repurposing effort was the disposal of a legacy high-performance liquid chromatography system housed in the 8-foot hood. The instrument was used to perform high-pressure separations of highly radioactive spent nuclear fuel specimens and was internally contaminated. The NAC worked closely as a team with ORNL Waste Services Division and Nuclear and Radiological Protection Division to create a waste disposal plan and Radiological Worker Permit for removal and packaging of the instrument. Once the room was cleared of unusable equipment, the laboratory floors were cleaned, a new output air filter bracket and air filter were installed, and the 8-foot chemical fume hood was cleaned and painted.

The lab was then equipped and set up for the HPT method. The following are highlights of the items purchased and installed:

- a six-place balance with a 22-gram capacity from Mettler Toledo
- a five-place balance with a 220 g capacity (Mettler Toledo, provided by NBLPO)
- a two-place top loader balance, capacity >5000 g, from Mettler Toledo
- a hot plate with ± 2°C surface gradient
- a drying oven
- an OHAUS pH/mV meter
- four saturated calomel electrodes from OHAUS (for later use and safekeeping)
- A platinum (99.95+% purity) wire electrode (3 ft)
- a temperature-controlled refrigerator
- a rod cutter for cutting uranium metal
- a new computer for real-time data processing

Figure 1 is a photograph of the updated laboratory space.

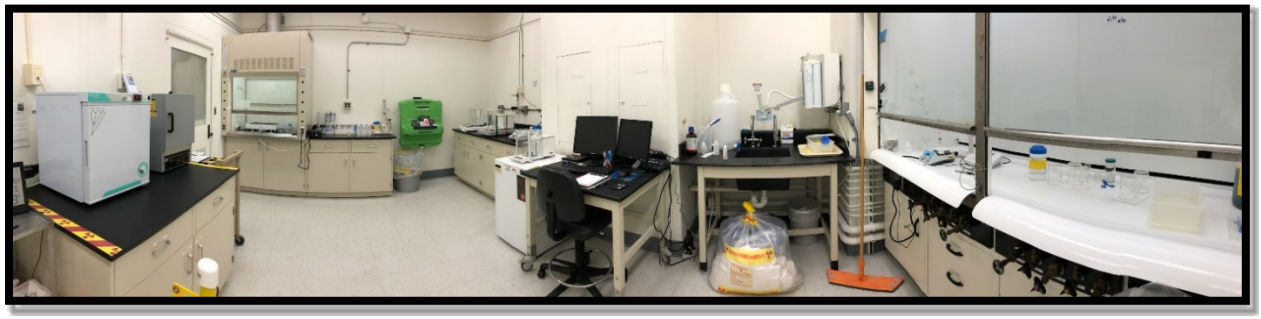

Figure 1. Photo of the HPT laboratory, Laboratory 218 in Building 4501 at ORNL, captured in June 2020.

3. SUMMARY OF THE HPT METHOD

HPT is a scaled-up variant of the Davies & Gray titrmetric method, which is a redox titration completed to a potentiometric endpoint. An oxidizing agent, in this case potassium dichromate, stoichiometrically oxidizes the uranium species. The "high-precision" prefix lends itself to the relatively large sample mass (i.e., 2.5–3.5 g U) used for the method; at this level, random deviations and biases due to titration chemistry are less pronounced. Uranium in phosphoric acid is reduced to U(IV) by ferrous sulfate. The excess Fe(II) is destroyed by molybdate-catalyzed oxidation with nitric acid. The nitrous acid produced in this reaction, which with time would reduce Fe(III) to again form Fe(II), is destroyed by reduction with sulfamic acid. The sample is diluted with water, and vanadyl sulfate is added to increase the rate of the titration reaction. U(IV) is titrated with a potassium dichromate primary standard by adding most of the potassium dichromate as a highly accurately weighed solid, followed by final titration, by mass, with a standard potassium dichromate solution. An endpoint for the chemical reaction has been previously experimentally determined at an mV reading of 600 ± 15 mV. The reactions are as follows:

$$\mathrm{U(VI) + 2\ Fe(II) \xrightarrow{H_3PO_4} U(IV) + 2\ Fe(III)}$$

$$\mathrm{2\ Fe(II) + HNO_3 + 2\ H^+ \xrightarrow{Mo(VI)} 2\ Fe(III) + HNO_2 + H_2O}$$

$$\mathrm{HNO_2 + NH_2SO_3H \rightarrow H_2SO_4 + N_2\uparrow + H_2O}$$

$$\mathrm{3\ U(IV) + 2\ Cr(VI) \xrightarrow{V(IV)} 3\ U(VI) + 2\ Cr(III)}$$

3.1 ORNL METHOD DEVELOPMENT

Additionally, for the successful transition of the HPT method from NBLPO to ORNL, guidance documents for the method had to be drafted. With heavy reliance on procedures provided by NBLPO, ORNL staff produced four standard analytical methods, a work aid, and operating instructions. During the drafting process, revisions were made to adjust for NAC's capabilities; Table 1 lists the major revisions. The following documents were written, reviewed, and approved and are now being controlled using ORNL's Integrated Document Management System:

- CSD-AM-NCL-IN02, *Determination of U—High Precision Titrimetric Method*
- CSD-AM-NCL-IN03, *Preparation and Verification or Standardization of Standard Potassium Dichromate Titrant*
- CSD-AM-NCL-IN04, *The Determination of Uranium in Iron (II) Reduction in Phosphoric Acid Followed by Chromium (VI) Titration*
- CSD-AM-NCL-IN05, *Preparation of Uranium Standard Solution*
- NACIL-WA-002, *Flaming Operations for Platinum Labware*
- CSD-OI-NCL-001, *Linearity Check of pH/mV Meters*

Table 1. Revisions to the ORNL documents

Document	Major revision
CSD-AM-NCL-IN02 Revision 0	The analysis for UF_6 was removed from the ORNL analytical method, where it had existed in the NBLPO HPT procedure.
CSD-AM-NCL-IN02 Revision 0	Reference to uncertainty calculations were revised from an NBLPO internal document (FOP-QA-40, *Calculation of Uncertainties for Reportable Measurements*) to the more general Joint Committee for Guides in Metrology 100:2008, *Guide to the Expression of Uncertainty in Measurement*.
CSD-AM-NCL-IN02 Revision 0	Readability specifications were added in the ORNL method for the thermometer, hygrometer, and barometer.
CSD-AM-NCL-IN02 Revision 0	A clause was added for the drying oven to be equipped with a calibrated thermometer with certificate on file.
CSD-AM-NCL-IN02 Revision 0	After phosphoric acid has been added to the solid uranium, and before dissolution, the ORNL method revised 2–3 drops 2% $K_2Cr_2O_7$ to 150 uL; changed 1–2 drops HF to 200 µL HF.
CSD-AM-NCL-IN02 Revision 0	The NBLPO procedure called for a steam bath for dissolutions. The equipment used for dissolution in the ORNL method is a hotplate. Specifically, this statement was made in the ORNL method: "Heat the uncovered beaker on a hot plate at 100°C until the sample has completely dissolved. Heat may be increased to aid dissolution; do not exceed 150°C (H_3PO_4, 85% boiling point = 158°C). Add additional HF dropwise as necessary to aid dissolution."
CSD-AM-NCL-IN02 Revision 0	The use of perchloric acid, $HClO_4$, (for the fuming of HNO_3 dissolved samples) was removed and replaced with the use of sulfuric acid (H_2SO_4). This statement was added: "This part of the procedure would need further testing. NBL laboratory work subsided before work could be completed to see if a substitution of H_2SO_4 could be made for H_3PO_4, only initial work had been done (see 16.14)." The statement references a study preformed at NBLPO in 2010.
CSD-AM-NCL-IN02 Revision 0	The following calculation was added for the tabulation of air density, whereas the NBLPO procedure used a different formula. $$d_a = \frac{0.348444\,p - h(0.00252\,t - 0.020582)}{273.15 + t}$$
CSD-AM-NCL-IN03 Revision 0	For the titrant that will be used in CSD-AM-NCL-IN04, the liquid potassium dichromate can be ***verified*** with six Davies and Gray titrations or can be ***standardized*** with a minimum of eight Davies and Gray titrations. The titrations must be equally split between two independently prepared uranium standard solutions.
CSD-OI-NCL-001 Revision 0	NBLPO used a voltage box that was calibrated by electronics metrology at Argonne National Laboratory. ORNL uses a commercial pH/mV simulator that is calibrated (traceable to NIST) by an outside company.

3.2 SPREADSHEET UPDATES

Data-reduction spreadsheets were shared with NAC from NBLPO. These included two controlled spreadsheets—(1) a spreadsheet for air density and buoyancy correction calculations made to the sample weight and potassium dichromate weight and (2) a spreadsheet for recording titration data and calculating the assay result. NAC separately prepared a spreadsheet for the preparation and verification of the liquid potassium dichromate. As a result of a recommendation during a quality assessment (see Section 5.2), with concurrence from NBLPO, the air density, buoyancy corrections, and titration data and calculations were combined into a single spreadsheet. This updated version was verified by hand calculation along

with an independent verification by NBLPO staff. The update was applied for uranium metal, UO_2, UO_3, and U_3O_8 sample types.

3.3 UNCERTAINTY CALCULATIONS

The Guide to Uncertainty in Measurement (GUM) defines measurement uncertainty as a "parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" [1]. A measurement typically has a mathematical model, or calculation, that relates "input values" to the final measurand. Uncertainties associated with these "input values" all contribute to the computation of the final measurand uncertainty. The GUM details a statistical approach to derive the standard uncertainty of the measurand by considering the uncertainty of the input values. In our case, this process is simplified through the use of GUM Workbench Software [2].

Development of a GUM Workbench can be quite involved, but it ultimately is beneficial to the analyst, lab management and sponsors, as it produces an uncertainty budget detailing the key input uncertainties that contribute to overall uncertainty. The uncertainty budget lists all the input values and their respective contributions to the total uncertainty in the form of a percentage. This is a tool that can be used to identify the major contributors to the uncertainty, and allow lab management and sponsors to focus efforts in areas that make significant improvements in the accuracy of measurement systems.. Although there are more than 30 input values in the HPT mathematical model, the major contributors to uncertainty of the uranium assay are the uncertainty of the certificatied value of the potassium dichromate and the weighing of sample and solid potassium dichromate (Figure 2).

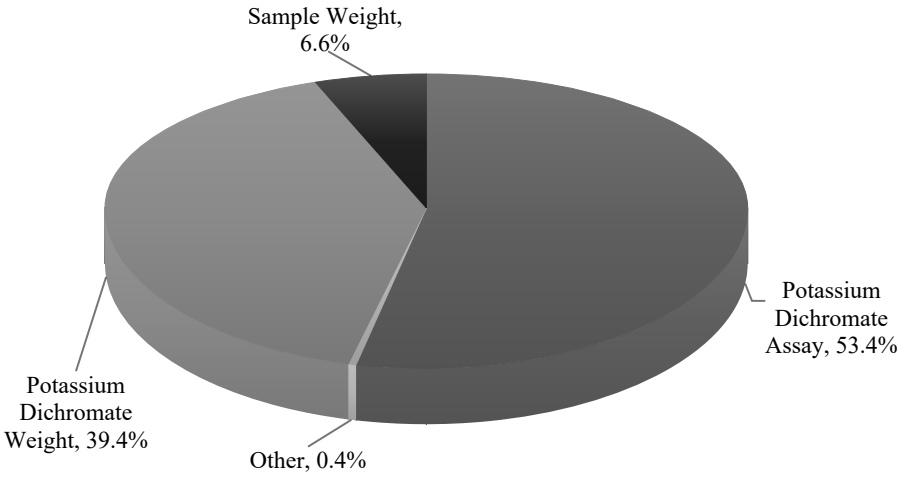

Figure 2. Major contributors to the uncertainty of a single HPT measurement when NIST SRM 136f is used.

4. TRAINING AND PRACTICE TITRATIONS

4.1 TRAINING

During the week of September 9, 2019, NBLPO personnel conducted a training program at the new HPT lab at ORNL. The week-long training included hands-on laboratory work as well as a lecture. NBLPO staff shared knowledge of and demonstrated a linearity check of pH meters, use of data recording and reduction spreadsheets, platinum electrode flaming, preparation of standard potassium dichromate, uranium metal cleaning, weighing, and dissolution. After observing, the trainees all took their turns at practicing each task with guidance from the NBLPO experts. The trainers demonstrated one titration and then assisted and observed as the trainees titrated two to three samples each.

At the conclusion of the training, recommendations were made regarding how to move forward to qualify the method. Table 2 is a list of personnel involved and their roles in the training. Appendix A has a copy of the agenda for the training. A memorandum from the director of NBLPO for completion of the training is also included in Appendix A.

Table 2. List of participants in September 2019 HPT training.

Personnel	Role
Haley Wightman	Secondary trainee at ORNL for the HPT method
Nancy Hui	NBLPO HPT expert
Ben Roach	Secondary trainee at ORNL for the HPT method
Kayron Rogers	Lead HPT and Davies and Gray analyst at ORNL
Anna Voeks	NBLPO HPT expert

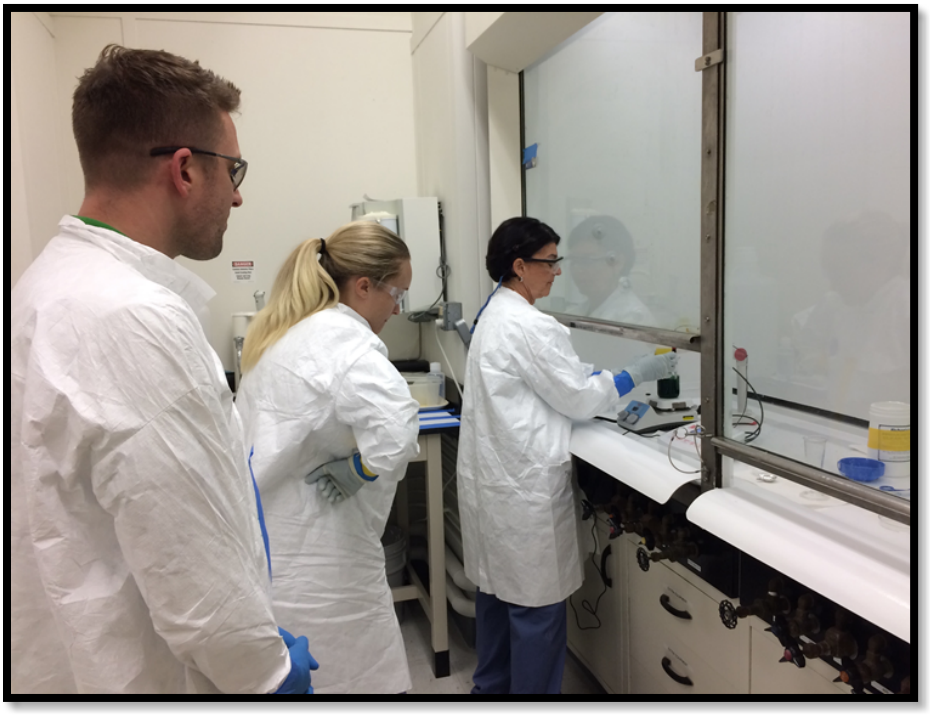

Figure 3. Trainees observe NBLPO personnel perform an HPT titration. From left to right: Ben Roach, Kayron Rogers, Anna Voeks.

5. QUALIFICATION

5.1 QUALIFICATION RESULTS

To qualify and demonstrate proficiency for the method, an analyst is required to prepare and analyze five samples of CRM 112-A, the uranium metal assay standard from NBLPO. To meet the requirement, all acceptable titrations must fall within 0.012% of the certified value of the CRM and achieve a specific precision (defined as the standard deviation of the % relative differences, of ≤0.006%). No more than one result may be removed from the five measurements as a result of handling error. In June 2020, analyst Kayron Rogers attempted to qualify for the HPT method (ORNL Analytical Method CSD-AM-NCL-IN02) by analyzing five CRM 112-A samples. Results of the five titrations are plotted in Figure 4. The mean of the assay values was 0.999726 g U/g with a relative standard deviation of 0.002%. These results fall well within the certified value for CRM 112-A of 0.99975 ± 0.00006 g U/g metal. The results qualified one analyst for the method, and a Demonstration of Capability form was issued (see Appendix A).

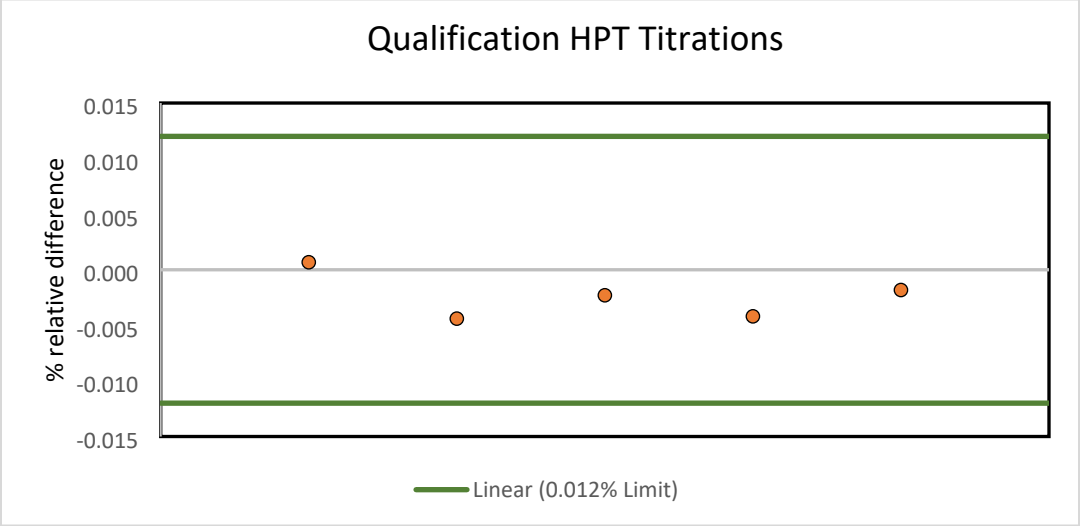

Figure 4. High-precision titration qualification results.

5.2 ASSESSMENT AND COMMITMENT TRACKING SYSTEM

The Assessment and Commitment Tracking System (ACTS) is a quality assurance measure at ORNL in which line managers and quality personnel assess tasks performed at all work levels to identify deficiencies or improvements in programs, systems or processes. During qualification, the NAC section head and quality representative assessed potassium dichromate weighing, uranium metal dissolution, and qualification titrations. This review included an in-laboratory observation during the potassium dichromate weighing and qualification titrations, as well as a review of spreadsheets used for data analysis.

Findings from the assessments identified one non-compliance: “Is software used tested, validated and approved for the calculations performed?” To address this issue, hand calculations for data reduction were documented and an independent verification of the spreadsheet was performed by NBLPO. Spreadsheet updates mentioned in Section 3.3 of this report were a result of an issue filed by the ACTS program. Once the requirements were satisfied, the ACTS issue was closed and the quality assessment was complete.

6. CONCLUSION

The NAC has completed a successful collaboration with NBLPO to establish a unique capability to perform the HPT method for the certification of existing and future uranium CRMs at ORNL. This capability is currently unique to ORNL and provides the NBL PO the ability to produce the next generation of uranium CRM's utilizing the expertise and ability developed through this collaboration. The method is highly accurate and reproducible for the determination of uranium content in metal or oxide forms. The development of this capability positions the NAC section to contribute to measurements that provide attribute values to critical uranium certified RMs under the direction of NBLPO.

Details of the HPT lab and procedure development and the training were presented at the 60th Institute of Nuclear Materials Management Annual Meeting in Palm Desert, California in July 2019, accompanied by a conference proceeding.

- Rogers, K., Roach, B., Giaquinto, J., Ilgner, R., Wightman, H., Chattin, M., Hexel, C., Voeks, A., Hui, N., Mason, P. "Establishing a High-Precision Titrimetric Laboratory at Oak Ridge National Laboratory to Support the NBL Program Office's Mission," Institute of Nuclear Materials Management 60th Annual Meeting, Palm Desert, California, July 14–19, 2019.

6.1 ACKNOWLEDGMENTS

This work was funded by the US DOE, National Nuclear Security Agency's NBL Program Office. NBLPO director Peter Mason identified ORNL as the first laboratory to establish the method and capability. Nancy Hui, Anna Voeks, Alma Stiffin, and Paul Croatto provided information and training that were invaluable to the success of the project. Robert Watters and Michael Holland reviewed spreadsheets and procedures and provided advice throughout the process. Lisa Duncan from the Nuclear and Radiological Protection Division provided assistance during the laboratory cleanup. Jimmy Selph from Transportation and Waste Management Division worked with NAC to develop a plan and documentation for disposal of legacy equipment for the lab refurbishment.

7. REFERENCES

1. *Evaluation of Measurement Data–Guide to the Expression of Uncertainty in Measurement*, JCGM 100:2008, Joint Committee for Guides in Metrology, 2008.
2. GUM Workbench Professional Version 2.4 [Computer Software] Metrodata, http://www.metrodata.de/ver24_en.html

APPENDIX A. TRAINING AND QUALIFICATION DOCUMENTS

APPENDIX A. TRAINING AND QUALIFICATION DOCUMENTS

A.1 TRAINING AGENDA

HPT Meeting Agenda

Event contact	Kayron Rogers, 865-574--2444 (office); 931-704-2514 (mobile); rogerskt@ornl.gov		
Time	**Event**	**Attendees**	**Place**
Monday, September 9, 2019			
8:30-9:00am	Meet at Visitor Center, Badge In, Transport to 1005 Room 84	Kayron Rogers, Anna Voeks, Nancy Hui	Visitor's Center
9:00-10:00am	Progress Update Discuss Plan for the Week	Kayron Rogers, Anna Voeks, Nancy Hui, Haley Heese, Ben Roach (referred to as All)	Building 1005 Room 84
10:00-10:20am	Break		
10:20am-11:30am	Talk Procedures Set up notebook pages for U Metal Dissolution	All	Building 1005 Room 84
11:30am-12:45pm	Lunch	All	ORNL Cafeteria
12:45-1:00pm	Transport to Training	Haley Heese, Anna Voeks, Nancy Hui	
1:00-2:00pm	Site Specific Training	Ed Turnington Anna Voeks Nancy Hui	
2:00-2:30pm	Transport to 4501, Building Tour, Changing, RWP Coverage	Lisa Duncan All	Building 4501
2:30-5:00pm	Enter Lab, Lab Tour Discuss Metal Dissolution *(Weigh and dissolve uranium metal. Anna demonstrates one piece. Kayron and Haley do one piece each. Enter data in spreadsheets for buoyancy correction and titration. Explain spreadsheet use as necessary. Review spreadsheets for previously dissolved metal).*	All	4501 Lab 218

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HPT Meeting Agenda

Time	Event	Attendees	Place
	Tuesday, September 10, 2019		
8:30-10:00am	Questions/Review on metal dissolution. Set up notebook pages for weighing potassium dichromate.	All	4501 Lab 218
10:00-10:15am	Break		
10:15am-12:00pm	Weigh potassium dichromate for all metal samples, except two for gel demo. Anna demonstrates; Kayron and Haley follow.	All	4501 Lab 218
12:00-1:00pm	Lunch	All	ORNL Cafeteria
1:00-4:30pm	**Review Kayron's** Standard Dichromate prep (Nancy). Continue weighing potassium dichromate. Enter data in spreadsheets for buoyancy correction and titration.	All	4501 Lab 218
	Wednesday, September 11, 2019		
8:30-10:00am	Set up notebook pages for titrations.	All	4501 Lab 218
10:00-10:15am	Break		
10:15am-12:00pm	Perform High Precision Titrations. Anna demonstrates; Kayron and Haley follow.	All	4501 Lab 218
12:00-1:00pm	Lunch	All	ORNL Cafeteria
1:00-4:30pm	Questions/Review Continue High Precision Titrations Questions/Review/Spreadsheets	All	4501 Lab 218

HPT Meeting Agenda

OAK RIDGE National Laboratory

AGENDA

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Time	Event	Attendees	Place
Thursday, September 12, 2019			
8:30-10:00am	Questions/Review Continue High Precision Titrations; Anna demonstrates gel formation	All	4501 Lab 218
10:00-10:15am	Break		
10:15am-12:00pm	Morning Continues	All	4501 Lab 218
12:00-1:00pm	Lunch	All	ORNL Cafeteria
1:00-4:30pm	Questions/Review Continue High Precision Titrations Finalize titration spreadsheet	All	4501 Lab 218
Friday, September 13, 2019			
8:30-10:00am	Questions/Review	All	4501 Breakroom
10:00-10:15am	Break		
10:15am-12:00pm	In lab, Questions/Review	All	4501 Lab 218
12:00-1:00pm	Lunch	All	ORNL Cafeteria
1:00-3:00pm	Discuss Results (136f, other topics as needed)	All	4501 Breakroom

A.2 MEMORANDUM FOR TRAINING FROM NBLPO

NNSA

NBL Program Office
U.S. Department of Energy

Memorandum

To: Oak Ridge National Laboratory

From: Peter Mason, NBL Program Office

Date: December 12, 2019

Re: Completion of High Precision Titration Training

This memo is to document the successful completion of training for three Oak Ridge National Laboratory (ORNL) chemists. This training was conducted the week of September 9-13, 2019 at ORNL. The trainers were Nancy Hui, NBL Program Office (NBL PO) chemist and Anna Voeks, former NBL Program Office chemist and current contractor to the NBL PO. The trainees were Kayron Rogers, Ben Roach and Haley Wightman.

The training consisted of lecture and hands-on demonstration including the trainee's each completing a number of titrations. The training included the following NBL PO procedures and techniques:

- Linearity check of pH meters
- Use of buoyancy correction and associated spreadsheet
- Electrode flaming procedure
- High precision titrimetric method, including all calculations
- Preparation of standard potassium dichromate
- Uranium metal cleaning, weighing and dissolution

The NBL PO trainers concluded that the ORNL participants demonstrated adequate ability and understanding of the methods and that ORNL should proceed with implementing the methods and eventual formal qualification of their chemists.

Peter Mason

NBL Progam Office Director
NA-121 Office of Stockpile Production Integration
US Department of Energy/National Nuclear Security Administration

Cc: J. Giaquinto, ORNL
K. Rogers, ORNL
To file, NBL PO

A.3 DEMONSTRATION OF CAPABILITY FORM

OAK RIDGE NATIONAL LABORATORY
MANAGED BY UT-BATTELLE FOR THE US DEPARTMENT OF ENERGY

Physical Sciences Directorate
Chemical Sciences Division
Nuclear Analytical Chemistry and Isotopics Laboratories Group

Demonstration of Capability

This document is to certify that **Kayron Rogers** has demonstrated acceptable performance in the analysis of **Uranium (g/g)** using procedure **"Determination of U-High Precision Titrimetric Method", CSD-AM-NCL-IN02**. This demonstration of capability involved the preparation and analysis of at least 5 known laboratory controls, prepared and analyzed in the same manner as samples. The laboratory controls were prepared using NIST-traceable material, **NBL, CRM112-A**, processed per approved analytical methods, in accordance with the NACIL Quality Assurance Plan (QAP-X-96-CSD/RML-001) and reported below. Acceptability is determined by calculating the percent recovery as well as the relative percent difference or standard deviation between replicates as applicable. Project or procedural requirements shall be documented as acceptance criteria.

Date of Analysis: **06/18/2020** Acceptance Criteria: **%RD < 0.012; mean SD < 0.006**

Data File/ Batch ID: **Excel file: HIGH-PRECISION-CALC-Final for ORNL Qualification 06-15-2020**

Criteria source: **CSD-AM-NCL-IN02, NBL-SA-U(E)-2-2**

Nominal standard concentration: **0.99975 ± 0.00006 (k=2)**

Sample ID	Result (g U/g)	% RD	Acceptance
202000923	0.99976	0.0007	Pass
202000924	0.99971	-0.0044	Pass
202000925	0.99973	-0.0023	Pass
202000926	0.99971	-0.0042	Pass
202000927	0.99973	-0.0018	Pass
Mean	0.99973	-0.0024	Pass
SD	2.1E-05	0.0021	Pass

Kayron Rogers 3012236 6/19/2020
Analyst

34733 6/29/2020
Laboratory Group Leader or delegate

Jennifer Parikh Digitally signed by Jennifer Parikh Date: 2020.06.29 15:31:54 -04'00'
Quality Representative