European Commission

JRC TECHNICAL REPORTS

# Standardization of JRC developed Nuclear Mass Spectrometry Methods with ASTM-International

*Drafting ASTM Standard for the "Double Spike" (DS/TIMS) Test Method by JRC-G.2/METRO*

Stephan Richter (JRC-G.2)

2017

Joint Research Centre

EUR 28812 EN

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**Contact information**
Name: Stephan Richter
Address:
European Commission - Joint Research Centre
Directorate G – Nuclear Safety and Security
Unit G.2 - Standards for Nuclear Safety, Security and Safeguards (SN3S)
Retieseweg 111
E-mail: stephan.richter@ec.europa.eu
Tel.: +32 14 571701

**JRC Science Hub**
https://ec.europa.eu/jrc

JRC108096

EUR 28812 EN

PDF ISBN 978-92-79-73911-8 ISSN 1831-9424 doi:10.2760/759192

Luxembourg: Publications Office of the European Union, 2017

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How to cite: Author(s); title; EUR; doi

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# Abstract

In 2017 the JRC-G.2/METRO team has contributed to standardization of methods developed at JRC for nuclear mass spectrometry in various ways.

Firstly, in early 2017 the revision of the ASTM standard document for the "classical" total evaporation (TE) method, written by K. Mathew (LANL, US-DOE) and S. Richter (JRC-G.2/METRO), was published with designation ASTM C1672-17. The document contains a detailed description of the TE method for U and Pu measurements, which was originally developed at JRC in the late 1980s. S. Richter from JRC-G.2/METRO contributed to the revision of ASTM 1672 in particular to the newly defined requirements for calibration and quality control for TE measurements, as well as by providing new algorithms for uranium and plutonium concentration measurements by IDMS (Isotope Dilution Mass Spectrometry).

Secondly, during the year 2017 a new draft standard document has been written by S. Richter (JRC-G.2/METRO). The document contains a detailed description of the "Double Spike" method (DS/TIMS) for high precision $^{235}U/^{238}U$ isotope ratio measurements. The first draft has been submitted for the C26-05 sub-committee ballot on 04/09/2017. The double spike method for TIMS was used successfully for the validation program of the Cristallini method for sampling of $UF_6$, to which staff from the unit JRC-G.2 in Geel/Belgium and the SGAS/IAEA contributed successfully.

Thirdly, S Richter (JRC-G.2/METRO) revised the draft "New Practice for Sampling and Preparation of $UF_6$ using the ABACC-Cristallini Method", which was drafted by P. Mason (NBL Program Office, US-DOE) and O. Pereira (IPEN, ABACC) and which will include the validation report as an annex. This support from JRC-G.2/METRO takes place within the frame of the new "Action Sheet 43: Collaboration on Certified Reference Material Development and Safeguards Measurements Quality Assurance" signed between JRC and US-DOE in 2017, and the collaboration agreement between ABACC and the JRC signed in 1999.

# 1 Introduction

ASTM International, formerly known as the American Society for Testing and Materials (ASTM), is a globally recognized organization for the development of international voluntary consensus standards. The ASTM Committee C26 on the Nuclear Fuel Cycle was formed in 1969. C26 standards play a significant role in all important aspects to the nuclear fuel cycle. C26 meets twice a year, usually in January and June, with about 50 members attending over one week of technical meetings. C26 has 10 technical subcommittees. One of them, C26.05, is dealing with "Methods of Test", which includes standardized methods for the analysis of nuclear material for the nuclear fuel cycle. This also applies to mass spectrometric methods as there are used and further developed at JRC-G.2 and for which JRC-G.2 provides the material standards (i.e. reference materials) for calibration. The subcommittee C26.05 has implemented several small task groups, e.g. for U and for Pu methods, environmental methods and spectroscopy.

During the recent years, ASTM International has changed from being an American-only organization into an internationally recognized standardization body. In particular, for the standardization of materials and methods for the nuclear fuel cycle used by industry (like URENCO, AREVA) and safeguards organizations (like the IAEA), ASTM International has become the main partner for standardization. In order to strengthen the international character of its standardization efforts, the ASTM C26 committee started organizing some of its sub-committee meetings outside the American continent, e.g. in 2013 a meeting was held in cooperation with CEA/CETAMA in Aix-en-Provence, France, and in 2016 a meeting took place in Vienna, Austria, in collaboration with the IAEA. This latest meeting was combined with a workshop on "Analytical Developments, Reference Materials and Statistical Applications in the Nuclear Fuel Cycle", which included presentations by several staff member of unit JRC-G.2.

The collaboration between JRC-G.2 and ASTM-International was initiated in 2013 due to the invitation of S. Richter (JRC-G.2) by M. v. Wijnkoop (Urenco) to develop an ASTM standard document for the so-called "modified total evaporation" (MTE) method for the determination of uranium isotope abundances by thermal ionization mass spectrometry (TIMS) [1]. Since several years this method is known to provide a significant improvement for measuring in particular the "minor" isotope ratios $^{234}U/^{238}U$ and $^{236}U/^{238}U$ for certification work at JRC-G.2 as well as for routine nuclear safeguards measurements at the IAEA, ORNL and JRC-G.II.6.

The ASTM standard document for the MTE method was approved in late 2015 and published in early 2016 with the designation C1832-16. It can be obtained through ASTM (https://www.astm.org/Standards/C1832.htm). The standardization of the MTE method was also highlighted in the JRC news: https://ec.europa.eu/jrc/en/news/standard-astm-c1832-16-based-method-developed-jrc?search.

During the year 2017, the revision of another important standard document for nuclear mass spectrometry, the document ASTM C1672 for the so-called "classical" total evaporation was published. This method was first developed at JRC [2] and subsequently further described by IAEA (International Atomic Energy Agency) [3] and LANL [4] (Los Alamos National Laboratory, United States). It has become the standard method for analysing the isotopic composition and isotope amount concentrations of uranium and plutonium samples for nuclear safeguards purposes around the world. Therefore, the revision of this standard document ASTM 1672 was considered very important. In order to guarantee accuracy of measurements for nuclear accounting and safeguards purposes, it has to be ensured that these measurements are performed in a reliable and trustable way. The first page of ASTM C1672 is shown in Annex-1.

The ASTM standard document for the TE method can be obtained through ASTM (https://www.astm.org/Standards/C1672.htm). The standardization of the TE method

was also highlighted in the JRC news: https://ec.europa.eu/jrc/en/science-update/newly-revised-standard-astm-c1672-17-based-jrc-methodology

Most recently, within 2017 S. Richter (JRC-G.2) has prepared a new draft standard document for the so-called "Double Spike" method (DS/TIMS) for high precision $^{235}U/^{238}U$ isotope ratio measurements [5, 6]. The first draft has been submitted for the C26-05 sub-committee ballot on 04/09/2017. The double spike method for TIMS was used successfully for the validation program of the Cristallini method for sampling of $UF_6$, to which staff from the unit JRC-G.2 in Geel/Belgium and the SGAS/IAEA contributed successfully [7].

Within ASTM-international an international group of experts from LANL, JRC, IAEA and the nuclear industry are working together in order to provide revised and improved standard documents for frequently used methods, in order to assist the users for performing their analyses in a way that can be relied upon and defended to the safeguards and governmental authorities. The contribution of JRC-G.2 is considered very valuable in this context.

# 2 Experimental

The "Double Spike" method (DS/TIMS) for high precision $^{235}U/^{238}U$ isotope ratio measurements has been used for many years already, because it provides a remarkably better precision by a factor of about 5-10 compared to other commonly used TIMS methods like the "classical" total evaporation (TE) [2-4] or "Modified Total Evaporation" (MTE) [1]. This is due to the fact that the certified $^{233}U/^{236}U$ ratio of the double spike, e.g. IRMM-3636a, with a ratio $^{233}U/^{236}U$=1.01906(16) certified on 01/07/2007 [5], can be used for an internal mass fractionation correction of the measured $^{235}U/^{238}U$ ratio throughout the entire measurement duration of a sample loaded on a filament. Due to the improved precision the double spike technique is already being used widely for scientific applications, in particular in geochemistry and cosmochemistry, for investigating the age and source of natural uranium samples, geochemical or physical processes [8-9], or for determining important half-life values of long-lived isotopes (i.e. $^{234}U$ and $^{230}Th$) for age determinations [10]. Based on the experience with the double spike method, JRC-G.2 proposed to ABACC and IAEA to use this method as a tool for the investigation of the Cristallini sampling process for $UF_6$ with the best possible precision and accuracy for the $^{235}U/^{238}U$ isotope ratio. For the so-called "minor" ratios $^{234}U/^{238}U$ and $^{236}U/^{238}U$ the MTE method was selected as the best choice at SGAS/IAEA [7].

# 3 Results

The work on the new draft standard document for the "Double Spike" method (DS/TIMS) was proposed and agreed upon during the ASTM-International meeting of subcommittee C26.05, "Methods of Test", on January 31$^{st}$, 2017. (S Richter could not attend the meeting due to other commitments at JRC-G.2). The relevant parts of the meeting minutes are shown below.

Opening of the C26-05-03 Task Group Meeting:

**Agenda for C26.05.03 Task Group Meeting**
Plasma/X-ray Methods in the Nuclear Fuel Cycle
**January 2017, Norfolk VA**

**Members: Mike Brisson, Steve Douglass, Khalil Ghaleb, Joe, Giaquinto, Lawrence Jassin, Donivan Porterfield, Benjamin Roach, Shane Robinson, Kayron Tevepaugh, Rebecca Thomas,**

**Visitors: Anil Thakkar**

**Session opened by Joe Giaquinto for Donna Beals 1:04**

Most of the subsequent discussions were not relevant within the context of this report and therefore not shown here. The meeting was closed at 1:41pm and re-opened (after lunch) at 2:35pm:

Closed the spectroscopy Group 1:41

**Reopened 2:35 – By Mike Brisson**

Re: Email from Stephan Richter – proposed new standard – TIMS Double Spike Standardization, task Group including Non-C26.05 members Rebecca Thomas expressed an interest in joining the Task Group.

"Dear Mike,

Very good. I have got confirmation from Sebastien Mialle also that he will join the TIMS/DS standardization group. I also asked Lars Borg from LLNL, because he is using a 233U/236U Double Spike as well, but he didn't pick up the method and spreadsheet from me (like Sebastien and Joe from the IAEA), so he could be a bit more independent as a contributor and reviewer, I hope his funding will be approved. So this is the task group composition:

Stephan Richter, JRC-G.2.
Kattathu Mathew, LANL
Joseph Hiess, IAEA
Sergei Boulyga, IAEA
Sebastien Mialle, JRC-II.6
Lars Borg, LLNL (to be confirmed)

I wish you a successful January meeting and I hope to be able to join you next time again.
Regards,
Stephan
Dr. Stephan RICHTER"

Reclosed: 2:39

After the meeting, a "collaboration area" was set up on the ASTM website by S Richter, and a task group formed, the description of the collaboration area is shown below.

Products and Services / Standards & Publications / Standards Products

ASTM WK57706

## New Test Method for Standard Test Method for Determination of Uranium Isotopic Composition by the Double Spike Method Using a Thermal Ionization Mass Spectrometer

Developed by Subcommittee: C26.05 | Committee C26 | Contact Staff Manager

MORE C26.05 STANDARDS | RELATED PRODUCTS | COPYRIGHT/PERMISSIONS

Work Item Status

Date Initiated:
02-07-2017

Technical Contact:
Stephan Richter

Status:
Draft Under Development

### WK57706

1. Scope

Measurement of the 235U/238U (or 238U/235U) uranium isotope ratio with high precision (better than 0.005% relative standard deviation) using a Thermal Ionization Mass Spectrometer (TIMS, in combination with a 233U/236U double spike (such as IRMM-3636). This is needed for studies of uranium fractionation effects in isotope geology, for uranium source attribution in nuclear forensics and for investigation of conversion or sampling processes in nuclear industry and nuclear safeguards.

Method will be used by safeguards agencies such as IAEA, for validation of sampling methods (e.g. Cristallini sampling method for UF6)

Keywords

Uranium, Mass Spectrometry, Reference Material, Spike, Double Spike

The title and scope are in draft form and are under development within this ASTM Committee.

CITING ASTM STANDARDS | BACK TO TOP

Within the following months, the draft was written by S Richter (JRC-G.2) and reviewed by K. Mathew (LANL), S. Mialle (JRC-G.II.6) and J. Hiess (IAEA). The final draft was submitted to the C26-05 sub-committee ballot on 04/09/2017.

At the end of the balloting period, all comments will be retrieved and any questions discussed by email and/or at the next upcoming ASTM C26-05 subcommittee meeting, scheduled for January 21st -24th , 2018, in New Orleans. After that, a new revision will be prepared and submitted to the next following concurrent sub- and main committee ballot in spring 2018. Depending on the approval, the final document can be released in summer 2018.

Besides the drafting of the standard document for the double spike method, S Richter also revised the draft "New Practice for Sampling and Preparation of $UF_6$ using the ABACC-Cristallini Method", which is authored by P. Mason (NBL Program Office, US-DOE) and O. Pereira (IPEN, ABACC). The draft was discussed at the ASTM C26-05 meeting in June 2017 in W Conshohocken (PA). (S Richter could not attend the meeting due to other commitments at JRC-G.2). The relevant parts of the meeting minutes are shown below:

**minutes of meeting:** **ASTM C26-05 U task group meeting W Conshohocken, PA 2017**

| | | | |
|---|---|---|---|
| location | : | ASTM International Headquarter, West Conshohocken | date of meeting: 20-06-2017 |
| author | : | M. van Wijnkoop (URENCO CTG) | date of minutes: 29-06-2017 |
| present | : | S. Robinson (Honeywell), B. Morel (Areva), R. Chomel (Areva Tricastin), M. Brisson (SRNL), D. Porterfield (LANL), J. Giaquinto (ORNL), P. Mason (NBL), K.A. Ghaleb (Areva MOX services), , B. Roach (ORNL), R. Thomas (SRNL), S. Mawn (ASTM International), D. Beals (SRNL), K. Novokshonov (TVEL), G. Mozzherina (TVEL), K. Mathew (NBL), M. Soriano, K. Tevepaugh (ORNL), G. Toury (Areva), O. Pereira de Oliveira (IPEN), E. Gautier (CNEA) | |
| absent | : | J. Agreiter (ETC), D. Bowers (ANL), M. Holland (SRNS), J. Johnson (Cameco), J. Miller (URENCO USA), J. Wilks (URENCO UK), N. Golchert (ANL), R. Foster (Centrus Energy), S. Richter (IRMM), A.R. Fernando (Cameco), S. Maxwell (SRNL), R. Linvall (LLNL), E. Ramon (LLNL), J. Hall (MCL Inc.), M. Sanders (MCL Inc.), R. Warren (Insolves), R. B. Durham, C. M. Johnson (AFRL), J.P. Shukla (Honeywell), D. Dillingham (Honeywell), K. Beckman (Honeywell), H. Russel (Paladin Energy), S. Vogt (NBL), A. Walczewski (Leco Corporation), A. Saprygin (UIEP), S. Carpenter (NuForensics), R. Blevins (Insolves), , K. McCoy (Areva NP), V. Gusev (AECC), V. Podskivalov (Intertech), T. O'Brien (Eichrom), B. Karmiol (Areva MOX services), M.E. Bennet (ANL), E.A. Taskaev (Eckert), S. Jerome (National Physical Laboratory), E. Gautier (CNEA), S. Merx (ETC Juelich). S. Douglass (Cameco), L. Luciani (Areva), L. Jassin (Eckert Ziegler Isotope Products) T. Cross (ANF Richland), A. Thakkar (ANL). | |
| distribution list | : | C26-05 Uranium task group members | |

Most of the discussion is not relevant within the context of this report and therefore not shown here. Under the agenda point "new standards", the following comments were made regarding the Cristallini sampling method for $UF_6$:

- ABACC- Cristalini $UF_6$ sampling method (P. Mason, O. Pereira de Oliveira)
  Aim to send 1st draft to sub-committee ballot before summer 2017, no ballot activity yet. In both C26.05 and C26.02 it was discussed which subcommittee should own this sampling method. Because the method is a form of process sampling (comparable to C1703 and C1052) and because C26.05 already has a large number of standards to maintain it was agreed that the standard will reside in C26.02. Because the method also invoked a lively discussion in C26.05 it was proposed to alert the 26.05 members when the standard goes to C26.02 sub-committee ballot, so they can have a possibility to comment.

Subsequently, S Richter also joined the subcommittee C26-02. The work on this document is still ongoing, and it is also planned to include a revised version of the validation report presented at the 58th Annual INMM Meeting 2017 [11] as an annex in order to give evidence for the trustworthiness of this new sampling method to all future users. It is foreseen to submit a draft version to a subcommittee ballot prior to the next upcoming ASTM C26-02 subcommittee meeting, scheduled for January 21st -24th , 2018, in New Orleans.

# 4 Conclusions

As in previous years, JRC-G.2 contributed to the standardization of methods for nuclear mass spectrometry which are relevant and often used in nuclear safeguards measurements worldwide. JRC-G.2 is currently involved in the preparation of two important new documents to be released in 2018.

It is worth mentioning that within November 2017, a visit of the ASTM President K. Morgan, to be companied by the Vice President G. Grove and the European representative of ASMT, Ms S. Gobbi, to the JRC-G.2 Geel site is planned. This will be combined with a meeting between the ASTM President K. Morgan and M. Betti, the Head of Directorate G for Nuclear Safety and Security of the JRC.

The role of standards is high on the policy agenda with the European Commission [12]. The JRC collaborates with international bodies and organisations in the US and across the globe to harmonise scientific techniques and standardise analytical processes. After the publication of the ASTM standard document ASTM 1832-16 for the JRC-developed "Modified Total evaporation" (MTE) technique in 2016, and the ASTM standard document ASTM 1672-17 for the "classical" total evaporation (TE) method in 2017, the draft standard for the double spike method (DS/TIMS) and the contribution to the Cristallini sampling method for $UF_6$ are further examples of JRC's input on standardisation in the nuclear field. The expertise developed at JRC-G.2 on a frequently used standard mass spectrometry technique will lead to the implementation of a significantly improved standard applicable in nuclear safeguards and nuclear industry world-wide.

## References


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12. European Commission – Joint Research Centre Thematic report Science for Standards: a driver for innovation, EUR 25942 EN

## List of abbreviations and definitions

| | |
|---|---|
| ASTM | American Society for Testing and Materials, United States |
| DS | Double Spike |
| DOE | Department of Energy, United States |
| IAEA | International Atomic Energy Agency, United Nations |
| IRMM | Institute for Reference Materials and Measurements (formerly). The nuclear activities of IRMM are continued at JRC - Unit G.2, named "Standards for Nuclear Safety, Security and Safeguards" (SN3S) |
| JRC | Joint Research Centre, European Commission |
| LANL | Los Alamos National Laboratory, Department of Energy, United States |
| METRO | Metrological tools for nuclear Safeguards, Security & Safety |
| MTE | Modified Total Evaporation method, used in TIMS (see below) |
| NBL | New Brunswick Laboratory (formerly), now renamed as "NBL Program Office" |
| SGAS | Safeguards Analytical Services (IAEA), located in Seibersdorf, Austria |
| SRNL | Savannah River National Laboratory, Department of Energy, United States |
| TE | Total evaporation method, used in TIMS (see below) |
| TIMS | Thermal Ionization Mass Spectrometry |

## List of figures

No figures

## List of tables

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# Annexes

1. First page of the ASTM standard document C1672-17. The complete document can be obtained through ASTM, but cannot be posted completely here: ([https://www.astm.org/Standards/C1672.htm](https://www.astm.org/Standards/C1672.htm)).

This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

**Designation: C1672 – 17**

## Standard Test Method for Determination of Uranium or Plutonium Isotopic Composition or Concentration by the Total Evaporation Method Using a Thermal Ionization Mass Spectrometer[1]

This standard is issued under the fixed designation C1672; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This method describes the determination of the isotopic composition, or the concentration, or both, of uranium and plutonium as nitrate solutions by the total evaporation method using a thermal ionization mass spectrometer (TIMS) instrument. Purified uranium or plutonium nitrate solutions are deposited onto a metal filament and placed in the mass spectrometer. Under computer control, ion currents are generated by heating of the filament(s). The ion currents are continually measured until the whole sample is exhausted. The measured ion currents are integrated over the course of the measurement and normalized to a reference isotope ion current to yield isotope ratios.

1.2 In principle, the total evaporation method should yield isotope ratios that do not require mass bias correction. In practice, samples may require this bias correction. Compared to the conventional TIMS method described in Test Method C1625, the total evaporation method is approximately two times faster, improves precision of the isotope ratio measurements by a factor of two to four, and utilizes smaller sample sizes. Compared to the C1625 method, the total evaporation method provides "major" isotope ratios $^{235}U/^{238}U$ and $^{240}Pu/^{239}Pu$ with improved accuracy.

1.3 The total evaporation method is prone to biases in the "minor" isotope ratios ($^{233}U/^{238}U$, $^{234}U/^{238}U$, and $^{236}U/^{238}U$ ratios for uranium materials and $^{238}Pu/^{239}Pu$, $^{241}Pu/^{239}Pu$, $^{242}Pu/^{239}Pu$, and $^{244}Pu/^{239}Pu$ ratios for plutonium materials) due to peak tailing from adjacent major isotopes. The magnitude of the absolute bias is dependent on measurement and instrumental characteristics. The relative bias, however, depends on the relative isotopic abundances of the sample. The use of an electron multiplier equipped with an energy filter may eliminate or diminish peak tailing effects. Measurement of the abundance sensitivity of the instrument may be used to ensure that such biases are negligible, or may be used to bias correct the minor isotope ratios.

1.4 The values stated in SI units are to be regarded as standard. When non-SI units are provided in parentheses, they are for information only.

1.5 *This standard may involve the use of hazardous materials and equipment. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

2.1 *ASTM Standards:*[2]

C753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder
C757 Specification for Nuclear-Grade Plutonium Dioxide Powder for Light Water Reactors
C776 Specification for Sintered Uranium Dioxide Pellets
C787 Specification for Uranium Hexafluoride for Enrichment
C833 Specification for Sintered (Uranium-Plutonium) Dioxide Pellets for Light Water Reactors
C859 Terminology Relating to Nuclear Materials
C967 Specification for Uranium Ore Concentrate
C996 Specification for Uranium Hexafluoride Enriched to Less Than 5 % $^{235}U$
C1008 Specification for Sintered (Uranium-Plutonium) DioxidePellets—Fast Reactor Fuel (Withdrawn 2014)[3]
C1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry
C1156 Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials

[1] This test method is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

Current edition approved Jan. 1, 2017. Published January 2017. Originally approved in 2007. Last previous edition approved in 2014 as C1672 – 07 (2014). DOI: 10.1520/C1672-17.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

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doi: 10.2760/759192

ISBN 978-92-79-73911-8