Certification Report

for the

Reference Materials

ERM-AE123 and ERM-AE124

Certified for their boron isotope composition

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1. Abstract

Isotope reference materials are essential to enable reliable and comparable isotope data. Besides the correction of mass fractionation or mass discrimination, isotope reference materials are indispensible for validation and quality control of analytical procedures. This report describes the production and certification of two isotope reference materials, ERM-AE123 and ERM-AE124, for boron isotope analysis. The isotopic composition of ERM-AE123 is the unaltered natural-like isotopic composition of the base material. The isotopic composition of ERM-AE124 has been adjusted by mixing two boron stock solutions enriched in ^{10}B and ^{11}B respectively under full gravimetric control. All stock solutions have been prepared fully under gravimetrical control. The corresponding boron isotopic composition has been analysed by TIMS. For both reference materials the isotopic composition obtained on the basis of the gravimetric data agrees very well with the isotopic composition obtained by TIMS.

The certified isotope abundances for ^{10}B are 0.19832 (21) for ERM-AE123 and 0.96006 (6) for ERM-AE124.

Together with the formerly certified ERM-AE101, -AE102a, -AE103, -AE104a, -AE120, -AE121 and -AE122, a unique set of nine certified reference materials (CRM) for boron isotope analysis is now available from BAM covering a range for the ^{10}B isotope amount fraction from 0.2 to 0.96 and for the $\delta^{11}B$ value from -20 ‰ to +40 ‰.

2.   Introduction

Based on its position between metals and nonmetals in the periodic system of the elements, boron features distinct physical properties. Due to its high hardness, its thermal and chemical resistance boron or its carbides are especially suited for various industrial applications (e.g. brake and clutch facings, plating, body armors). The oxidic compounds of boron are mainly used in ceramic and detergent industry or for production of fluxing agents, herbicides or fertilizers (Hollemann *et al.* 1995).

Because of its affinity for oxygen, boron never appears in its elemental form in nature, but always as oxidic compound. Depending on the pH value two dominant boron species occur in aqueous solution: $B(OH)_3$ and the $B(OH)_4^-$ anion. Thereby ^{11}B prefers the triangular planar structure of $B(OH)_3$, whereas ^{10}B prefers the tetrahedric structure of $B(OH)_4^-$ (Palmer *et al.* 1992). In rocks or minerals preferably $B(OH)_4^-$ is assembled so that $B(OH)_3$ stays in the aqueous phase (Palmer *et al.* 1987). The therewith combined isotope fractionation of boron leads to isotopic variations of up to 90 ‰ in nature. This large variation allows the investigation of geochemical processes (Leeman & Sisson, 1996) or anthropogenic influences in water reservoirs (Eisenhut *et al.* 1996, Vengosh *et al.* 1999).

Another important property of boron is the very high neutron cross section of ^{10}B, which amounts 3.84×10^3 barns and therefore exceeds 8×10^5 times that of ^{11}B (Lide 2001). Thus boron is used as neutron absorber according to the reaction shown in Eqn. 1:

$$^{10}B \, (n,\alpha) \, ^7Li \hspace{6cm} \text{Eqn. 1}$$

In nuclear power plants working with pressurized water reactors the thermal power is being controlled that way by keeping a certain concentration of boric acid in the primary cooling circuit. The concentration of ^{10}B has to be monitored permanently, as the overall amount of ^{10}B and consequently the neutron absorption in the primary cooling agent is steadily decreasing. Therefore the boric acid concentration and the $^{10}B/^{11}B$ isotope amount ratio have to be determined on a regular basis to enable the adjustment of the ^{10}B concentration.

Apart from industrial application boron isotopes are increasingly used in geochemical and environmental studies.

Because all mass spectrometric measurements are affected by mass discrimination or mass fractionation, the determined isotope ratios are biased relative to the "true value". To enable comparable isotope data, the measurements have to be carried out in a way that the results are traceable either to the International System of Unit (SI) or to an internationally accepted standard. This means that the determination of isotope amount ratios has to be corrected for mass discrimination or mass fractionation by applying a Reference Material (RM) being certified for its isotopic composition. The determination of relative differences or so-called δ-values can be related to RM being certified for their δ-values. More details on this topic can be obtained from Vogl and Pritzkow 2010a.

Until 2001 only IRMM and NIST offered boron isotope RM either with natural isotopic composition (NIST SRM 951, $δ^{11}B=0$; IRMM-011 & 611) or highly enriched in ^{10}B (NIST SRM 952, IRMM-610). The operators of nuclear power plants require boron isotope RM in form of aqueous boric acid solutions

with ^{10}B isotopic abundances between those materials. To meet these needs BAM certified in 2001/2002 a set of boron isotope RM, enriched in ^{10}B and labeled BAM-I001, I002, I003 and I004, which became ERM-AE101, AE102, AE103 and AE104 in 2004. When ERM-AE102 and 104 ran out of stock, they have been replaced by ERM-AE102a and -AE104a in 2010. As no RM for boron with δ^{11}B$\neq$0 is available yet and there is an increasing need for these materials due to an increased number of boron isotope measurements for geochemical and environmental applications, a set of boron RM certified for δ-values (ERM-AE120, -AE121, -AE122) has been produced and certified in parallel (Vogl et al. 2010b).

On request of some customers from the nuclear field the production and certification of two additional boron isotope reference materials with nominal ^{10}B isotope amount fractions of approximately 0.2 and 0.96 has been carried out, which is described in detail in the following report.

3.   Experimental Section

3.1. Principal Procedure

Aqueous solutions of boric acid with distinct isotopic compositions can be produced by mixing either a solution enriched in ^{10}B or in ^{11}B with a boric acid solution of natural isotopic composition under full gravimetric control. For the mixing components the boron mass fraction and the isotopic composition should be known exactly. Unfortunately boric acid cannot be used easily for preparing a primary standard, because it contains variable mass fractions of water. Strong drying procedures are not applicable, because boric acid will be dehydrated at around 100°C and will be converted into metaboric acid HBO_2. Therefore no drying procedure can be applied providing a distinct boron compound with an exact stoichiometry offering an uncertainty of less than 0.1% for the elemental mass fraction.

Previous work (Vogl et al. 2010b) has shown that a combination of a gentle drying procedure and a subsequent Karl-Fischer Titration yields gravimetric results being indistinguishable from IDMS results. This has been applied within this project for the stock solutions containing either ^{10}B or boron with natural-like isotope composition.

3.2. Chemicals, Reagents and Labware

All dilutions and manipulations have been carried out by using ultrapure water obtained from a Milli-Q Advantage A10 water purification system. Nitric acid has been purchased p.a. grade and has been purified by twofold subboiling distillation (1st stage quartz still, 2nd stage Teflon still). Only quartz, PTFE or PFA labware have been used in this project. All preparation steps have been carried out in a low boron laboratory environment (Rosner et al. 2005).

For the preparation of the CRMs three boron starting materials with different isotopic composition have been used: a boric acid with natural isotopic composition, a boric acid enriched in ^{10}B and a boron metal enriched in ^{11}B. The boric acid with approximately natural isotopic composition has been purchased suprapure grade from Merck KgaA (99.9999%, Lot. B0481065119). The boric acid

enriched in ^{10}B has been used and characterized previously (Vogl *et al.* 2010b). The ^{11}B solution has been taken from the stock, which has been prepared and characterized in 2010 (Vogl *et al.* 2010b) and has been stored under controlled conditions including weight monitoring. The isotopic composition of these materials is displayed in Table 1.

Table 1: Boron isotopic composition of the starting materials used for the preparation of the RM with their combined standard uncertainties given in brackets and applying to the last one or two digits

Quantity		Boric acid		Boron metal
		enriched in ^{10}B	natural	enriched in ^{11}B
Isotope amount ratio	$n(^{10}B)/n(^{11}B)$	48.019 (34)	0.24739 (17)	0.009239 (7)
	$n(^{11}B)/n(^{10}B)$	0.020825 (15)	4.0422 (27)	108.24 (9)
Isotope amount fraction	$n(^{10}B)/n(B)$	0.979600 (14)	0.19832 (11)	0.009154 (7)
	$n(^{11}B)/n(B)$	0.020400 (14)	0.80168 (11)	0.990846 (7)
Molar mass in g·mol^{-1}	$M(B)$	10.033263 (14)	10.81036 (11)	11.000184 (7)

3.3. Gravimetric Determinations

The preparation of solutions has been carried out under full gravimetric control. To enable this, a few practical aspects have to be considered, as the weighing process is influenced by different effects such as buoyancy, electrostatic effects and evaporation. Moisture content of boron compounds has to be considered separately. Other stability issues do not apply here.

Buoyancy or more exactly air buoyancy expresses the difference in air buoyancy between the object to be weighed and the built-in reference of the balance, which causes a bias. The effect is well-known and corrections are applied according to Eqn.2 whenever reference weighing is performed, e.g. for RM characterisation (Kehl *et al.* 2000, Reichmuth *et al.* 2004). In Eqn. 2 m_{x_obs} is the reading of the balance, ρ_{Air}, ρ_{bal} and ρ_x are the densities of the air, the built-in weights of the balance and of the sample. For objects with a density of around 10^3 kg·m^{-3} (e.g. water) the resulting bias is in the order of 10^{-3} relative and should be corrected for (Pozivil *et al.* 2006).

$$m_x = m_{x\_obs} \cdot \frac{\left(1 - \dfrac{\rho_{air}}{\rho_{bal}}\right)}{\left(1 - \dfrac{\rho_{air}}{\rho_x}\right)}$$

Eqn. 2

Electrostatic charging of plastic containers cannot be corrected for, but it may be reduced or even avoided by specific tools compensating the electrostatic charge. This can be accomplished best by blowing ionized nitrogen onto the container's surface with a ring ionizer combined with a blow-out gun (Type RI 65 P 7187 500, Haug GmbH, Leinfelden-Echterdingen, Germany). This device cannot be used when powders have to be weighed on open plastic trays. Logically, for any handling only cotton gloves should be used, because plastic gloves would produce new electrostatic charges. Gloves in

general are required to avoid fingerprints on the container, which could easily make up a mass bias of a tenth of a mg.

The weighing of solutions into a container seems to be an easy task. However, performing it on a reference level some specific aspects have to be considered. It is advisable to fill a polyethylene syringe with the solution and place a pipette tip on the tip of the syringe to reduce the orifice and a sealed pipette tip to close the reduced orifice. Then the filled syringe is being weighed, the desired mass is being dosed into the container and the syringe is being weighed back. This gives the exact mass of the solution which has been released. When pouring the solution into the container and weighing the mass difference in the container, first the relation of container mass to solution mass is less advantageous (weighing a mouse on an elephant's back) and second during the pouring step solvent will evaporate, which will not be considered. The syringe approach considers this, because the released mass is being determined. Consequently, all prepared solutions have to be monitored for their mass during storage, because from each bottle the solvent (water or dilute acid) will evaporate in small amounts and will change the mass fraction of the solution. Monitoring the mass of the solution can be used to correct for this.

All solutions within this project have been prepared considering these facts and therefore an uncertainty budget can be set up. As an example the full uncertainty budget for the ^{11}B enriched solution is listed in Table 7 (Appendix).

3.4. Mass Spectrometric Determinations

Boron isotope ratios of all samples have been determined by TIMS using the $Na_2BO_2^+$ technique on a Sector 54 instrument (Micromass, Manchester, UK). The $Na_2BO_2^+$ technique has been applied for the characterization of all boron solutions and reference materials within this project.

$Na_2BO_2^+$ TIMS technique

For the $Na_2BO_2^+$ technique the samples have been evaporated to dryness in a cold nitrogen stream and were subsequently dissolved in stoichiometric amounts of NaOH. The mass of 15 µg boron was loaded on Re single filaments (boat design), which were placed on a turret and introduced into the mass spectrometer. After evacuation of the ion source (< $5 \cdot 10^{-8}$ mbar) the automatic measurements started with a high voltage of 8 kV. Each filament was heated up to 1.3 A filament current. Then a series of scanning for analyte signal, peak centering and auto-focusing started, each followed by an additional rise of the filament current. This was carried out until an ion intensity of 1.6 V had been reached at the monitor mass: mass 89, $^{23}Na_2^{11}B^{16}O_2$, for ERM-AE123 and mass 88; $^{23}Na_2^{10}B^{16}O_2$, for ERM-AE124. Then the measurements were started, whereby the filament current was automatically controlled to yield an intensity of 2 V at the monitor mass. Data were recorded in one block of 300 cycles. The achieved repeatability between individual filaments is better than 0.05 % for the ion current ratio.

The multi-collector design allows the simultaneous measurement of the masses 88 ($^{23}Na_2^{10}B^{16}O_2^+$) and 89 ($^{23}Na_2^{11}B^{16}O_2^+$), which enables more precise isotope ratio determinations than a single collector instrument. As the ion $^{23}Na_2^{10}B^{16}O^{17}O^+$ also occurs on mass 89 the observed isotope ratios

have to be corrected using the so-called oxygen-correction (De Bièvre *et al.* 1969). For correcting the mass fractionation always IRMM-011 was used.

3.5. Preparation of the Isotope Reference Materials

Following customer requests two new boron isotope RM have been prepared realizing ^{10}B isotope amount fractions of approximately 0.2 and 0.96. The first candidate RM, ERM-AE123, should reflect a natural-like boron isotopic composition and has been solely prepared from boric acid with natural isotopic composition purchased from Merck (Boric acid, suprapure grade, 99.9999%, Lot. B0481065119). The second candidate material, ERM-AE124, with a ^{10}B isotope amount fraction of approximately 0.96, has been prepared by mixing ^{10}B enriched boric acid, $n(^{10}$B$)/n$(B) ~ 0.98, with a solution containing ^{11}B enriched boron, $n(^{11}$B$)/n$(B) ~ 0.99.

The exact masses of the boric acid amount and the solutions used for the preparation together with their corresponding standard uncertainties are displayed in Table 2. The mixing process has been carried out under full gravimetric control as described above in the section "gravimetric determination". Subsequently ultrapure water has been added to reach a boron mass fraction of approximately 1 g/kg. The exact mass fractions are also displayed in Table 2. These stock solutions have been filled in pre-cleaned PFA-bottles. The bottles have been closed tightly, labelled, sealed in plastic bags and stored in a refrigerator at (5 ± 3) °C. The filling process has been controlled gravimetrically to guarantee a minimum filling quantity of 20 mL. Thus 49 bottles have been produced from each material. Three bottles – one at the beginning, one in the middle and one at the end of each filling sequence – out of each material – have been overfilled by 2-3 mL. This volume has been removed later by pouring to enable the measurements for the characterization of the material.

Table 2: Masses of the solutions and the boric acid used for the preparation of the isotope RM together with the isotope amount ratios and the boron mass fractions; combined standard uncertainties are given in brackets

Material	Mass in g			
	^{10}B boric acid	nat. boric acid	^{11}B solution *	Total solution
ERM-AE123	n.a.	6.21413 (10)	n.a.	1022.51700 (61)
ERM-AE124	6.14368 (10)	n.a.	22.32645 (10)	1031.0930 (12)

	Quantities calculated from gravimetric data		
	B mass fraction	Isotope amount ratio	
	in mg·kg^{-1}	$n(^{10}$B$)/n(^{11}$B$)$	$n(^{11}$B$)/n(^{10}$B$)$
ERM-AE123	1062.93 (11)	n.a.	n.a.
ERM-AE124	1001.51 (10)	23.997 (11)	0.04167 (19)

* Details on the ^{11}B solution can be obtained from Table 7.

3.6. Homogeneity

Dilute solutions of one element in principle are homogenous. However, when mixing different solutions of the same element, but with different isotopic compositions, it is necessary that only one species occurs or the occurring species are equilibrated. Then an equilibrium regarding the isotope distribution can be assumed and the solution is homogenous regarding the isotopic composition of the analyte element.

In all solutions used for the preparation of the boron isotope RM boron is present as boric acid in balance with $B(OH)_4^-$. Therefore all prepared solutions can be assumed homogenous. Experience at BAM over two decades with boron spike and back-spike solutions have shown this. Additionally this has been demonstrated already for the previous nine boron isotope reference materials (Vogl et al. 2002, Vogl et al. 2010b).

Therefore the material can be assumed as homogeneous. The characterization which has been carried out on three units spread over the whole filling sequence supports this, without any hint for inhomogeneity.

3.7. Stability

Previous work (Vogl *et al.* 2002, Vogl *et al.* 2010b) in the field of boron analysis show, that aqueous solutions of boric acid in the $g \cdot kg^{-1}$ range are stable over years for their isotopic composition as well as for their mass fraction. To guarantee stability of the boron mass fraction, contamination, adsorption to container walls and evaporation of water have to be avoided. For the reference materials discussed here this has been achieved by using pre-cleaned PFA bottles and storage under dark and cool, (5 ± 3) °C, conditions. The preceding material ERM-AE104 was monitored for the weight change due to evaporation. This revealed a mass loss of 0.02 % over 6 years, which is much smaller than the uncertainty of 2 % on the boron mass fraction in ERM-AE104.

A more extensive testing has been carried out during the previous certification project to estimate evaporation of water through PFA container walls and the screw cap (Vogl et al. 2010b). Here ten PFA-bottles have been filled with 20 mL water and have been placed in plastic zip bags. Five bottles have been stored at room temperature (24 ± 3) °C and five in a refrigerator at (5 ± 3) °C. For the bottles stored (5 ± 3) °C the evaporation loss was 0.019 % for 24 weeks, for the bottles at room temperature the loss was five times higher. An extrapolation of the evaporation at (5 ± 3) °C to 520 weeks (10 years) gives a loss of less than 0.47 %, indicating a maximum increase of the boron mass fraction of 0.47%. This can be converted into an uncertainty contribution of 0.5 % for the mass fraction, when aiming at a shelf life of ten years. Considering higher temperatures during shipment another 0.5 % will be added. Thus the relative expanded uncertainty for the boron mass fraction is 2 % (Table 6). More details can be obtained from Vogl *et al.* 2010b.

The stability of the isotopic composition is solely compromised by contamination with boron having a different isotopic composition. Pre-cleaned PFA bottles and the high boron mass fraction in the solution (~ 1 $g \cdot kg^{-1}$) reduces the risk of contamination to a minimum. In the preceding series of boron

isotope reference materials, ERM-AE101 to -AE104, a stability monitoring took place over nine years, showing no significant change over this period (Vogl *et al.* 2010b). Boron isotope RM produced under the same conditions (e.g. temperature, packaging material) as ERM-AE101 to -AE104 are expected to be stable at least for ten years.

The repeatability of the analytical technique is already included in the characterization and thus no extra contribution for the stability test has to be added to the uncertainty budget for the isotopic composition.

3.8. Characterization

The isotopic compositions have been calculated on the basis of the gravimetric data and/or on the basis of the TIMS boron isotope measurement results obtained with the $Na_2BO_2^+$ technique (Table 4). Instrumental mass fractionation during TIMS measurements was corrected with a correction factor obtained from concurrently measured IRMM-011. Two outliers have been detected by MYSTAT 12 (Box-Whisker-Plot) for 15 measurements of ERM-AE124. These outliers have been removed due to technical reasons, which are bad filament performance due to repeated use.

The characterization is carried out by a single reference procedure ($Na_2BO_2^+$ TIMS). The results for ERM-AE124 have been verified by a second procedure: calculation of the isotopic composition based on gravimetric data and the isotopic composition of the starting materials.

Boron isotope data calculated from the gravimetric results (ERM-AE124) and those obtained from TIMS measurements (ERM-AE123, -AE124) are displayed in Table 4. Gravimetric results and TIMS results agree with one another for ERM-AE124. The difference between both values is smaller than the expanded uncertainty. This small difference between gravimetrical and TIMS value may derive from an incorrect value for the water content of 0.046(2) %. Setting this very low value to zero reduces the difference significantly, the gravimetric value for $n(^{10}B)/n(B)$ shifts to 0.960004. Nevertheless, the TIMS value will be used as certified value as done in previous certifications.

Table 4: Isotopic composition of ERM-AE123 and -AE124 calculated from TIMS measurement results and from gravimetric data; all quantity data are given with combined standard uncertainties in brackets and applying to the last two digits

ERM No.	Proc. [a]	Isotope amount ratio		Isotope amount fraction		Molar mass
		$n(^{10}B)/n(^{11}B)$	$n(^{11}B)/n(^{10}B)$	$n(^{10}B)/n(B)$	$n(^{11}B)/n(B)$	in g·mol^{-1}
AE123	TIMS	0.24739 (17)	4.0422 (27)	0.19832 (11)	0.80168 (11)	10.81170 (11)
AE124	TIMS	24.040 (17)	0.041597 (29)	0.960064 (27)	0.039936 (27)	10.052728 (27)
	Gravim.	23.997 (11)	0.041672 (19)	0.959995 (17)	0.040005 (17)	10.052797 (17)

[a] analytical procedure: TIMS represents PTIMS with $Na_2BO_2^+$ technique and mass fractionation correction with IRMM-011; Gravim. means calculation on the basis of gravimetric data.

4. Certification

For the certification of a reference material all data altering or affecting the quantity value to be certified or its combined uncertainty have to be collected and used for establishing the certified quantity value. According to international guidelines mainly the homogeneity and the stability have to be considered.

Based on experience and results from earlier certification campaigns the isotopic composition of boric acid solutions is not affected by homogeneity and stability issues. Therefore no additional uncertainty contribution is necessary. The certified quantity values of the discussed reference materials (Table 5) derive only from the characterization study. As in previous certifications the $Na_2BO_2^+$ TIMS results are used to certify the isotope abundance ratios of ERM-AE123 and -AE124. The certified boron isotopic compositions as well as the molar masses are being calculated therefrom.

Table 5: Certified quantity values of ERM-AE123 and -AE124 with their expanded uncertainties (k=2)

ERM	Isotope amount ratio		Isotope amount fraction		Molar mass
No.	$n(^{10}B)/n(^{11}B)$	$n(^{11}B)/n(^{10}B)$	$n(^{10}B)/n(B)$	$n(^{11}B)/n(B)$	in g·mol^{-1}
AE123	0.2474 (4)	4.042 (6)	0.19832 (21)	0.80168 (21)	10.81170 (21)
AE124	24.04 (4)	0.04160 (6)	0.96006 (6)	0.03994 (6)	10.05273 (6)

The boron mass fractions in all materials are indicative values only (Table 6). The relative expanded uncertainties of the boron mass fractions have been set to 2 % based on results from previous studies.

Table 6: Indicative quantity values of ERM-AE123 & -AE124 with their expanded uncertainties (k=2)

ERM No.	B mass fraction in solution in mg·kg^{-1}
AE123	1063 (20)
AE124	1002 (20)

The boron isotope reference materials ERM-AE123 and -AE124 are primarily intended to be used as standards for correcting mass discrimination in single collector ICP-MS and as quality control samples. ERM-AE124 can also be used for isotope tracer studies and as a spike for isotope dilution analysis. In the latter case, however, the characterization by reverse IDMS is advisable, when low measurement uncertainties (<1% relative) are aimed at. These reference materials should be stored under dark and cool, (5 ± 3) °C, conditions.

These certified reference materials (CRM) are traceable to the International System of units (SI) in the shortest possible way, by calibrating all instruments (balance, mass spectrometer) against SI-traceable calibrators. For isotope measurements the SI traceable calibrator was IRMM-011, which is a primary isotope reference material. Mass fractions are determined gravimetrically by using SI-traceable weights (ERM-AE123 & -AE124) and by isotope dilution mass spectrometry (IDMS) applied

as primary method of measurement (ERM-AE124). In the latter case traceability is established via SI-traceable weights and high-purity boron.

Measurements calibrated against these ERM will, therefore, also be traceable to the SI.

5. Appendix: Exemplary uncertainty budgets

Table 7: Uncertainty budget for the boron mass fraction in the ^{11}B solution

Quantity	Value	Standard uncertainty	Unit	Type A/B	Description	Index
p_{air}	1023.000	0.577	mbar	B	air pressure	0.0 %
T_{air}	298.150	0.289	K	B	temperature of the air	0.0 %
H_{rel}	87.000	0.577	%	B	relative humidity	0.0 %
$p_{svp\text{-}water}$	26.4400	0.0500	mbar	B	saturation vapour pressure of water at T_{air}	0.0 %
ρ_{air}	1.18557	0.00133	kg/m^3	B	density of the air	*
ρ_{bw1}	8000.0	10.0	kg/m^3	B	density of the balance weights of the microbalance	0.0 %
ρ_{bw2}	7950.0	10.0	kg/m^3	B	density of the balance weights of the analytical balance	0.0 %
ρ_z	2350.00	5.00	kg/m^3	B	density of the boron metal	0.0 %
ρ_{Lsg}	1045.00	2.89	kg/m^3	B	density of the solution	0.1 %
k_{mz}	1.00035648	0.00000116		B	buoyancy correction factor for microbalance	*
k_{sol}	1.00098651	0.00000334		B	buoyancy correction factor for analytical balance	*
P_z	0.9999950	0.0000900		B	purity of boron metal	89.1 %
$m_{total\text{-}obs}$	326.94300	0.00275	g	B	observed mass of bottle with solution	2.1 %
$m_{bottle\text{-}obs}$	126.91720	0.00275	g	B	observed mass of bottle	2.1 %
$m_{z\text{-}obs}$	203616.00	5.00	µg	B	observed mass of boron metal	6.6 %
m_z	203687.6	19.0	µg	B	mass of boron metal	*
$m_{z\text{-}sol\text{-}obs}$	200.02580	0.00389	g	B	observed mass of the solution	*
$m_{z\text{-}sol}$	200.22313	0.00395	g	B	mass of solution	*
w_z	1017.3029	0.0970	µg/g	B	Mass fraction boron in the back-spike solution	
w_z	1017.30	0.19	µg/g	B	Mass fraction boron in the back-spike solution, expanded unc. ($k=2$)	

*   Intermediate result

Equation system:

$\rho_{air} = (0.3485 * p_{air} / T_{air}) - 0.00132 * p_{svp\text{-}water} * (H_{rel}/T_{air});$
$k_{mz} = (1 - (\rho_{air} / \rho_{bw1})) / (1 - (\rho_{air} / \rho_z));$
$k_{sol} = (1 - (\rho_{air} / \rho_{bw2})) / (1 - (\rho_{air} / \rho_{Lsg}));$
$m_z = m_{z\text{-}obs} * k_{mz} * P_z;$
$m_{z\text{-}sol\text{-}obs} = (m_{total\text{-}obs} - m_{bottle\text{-}obs});$
$m_{z\text{-}sol} = m_{s\text{-}sol\text{-}obs} * k_{sol};$
$w_z = m_z / m_{sol};$

Table 8: Uncertainty budget for the isotope amount ratio $n(^{10}B)/n(^{11}B)$ of ERM-AE123 as determined by TIMS

Quantity	Value	Standard uncertainty	Unit	Type A/B	Description	Index
$I_{c88/89}$	0.24744217	0.00000899	-	A	Ion current ratio 88/89 of standard IRMM-011	0.3 %
$I_{123-88/89}$	0.2475694	0.0000225	-	A	Ion current ratio 88/89 of candidate reference material ERM-AE123	1.9 %
$I_{c89/88}$	4.041348	0.000147	-	A	Ion current ratio 89/88 of standard IRMM-011	*
$I_{123-89/88}$	4.039271	0.000368	-	A	Ion current ratio 89/88 of candidate reference material ERM-AE123	*
K_S	0.0007900	0.0000100	-	B	Oxygen correction	0.0 %
$R_{co10/11}$	0.24749055	0.00000902	-	B	Observed, oxygen corrected isotope ratio 10/11 of standard IRMM-011	*
$R_{123o10/11}$	0.2476179	0.0000226	-	B	Observed, oxygen corrected isotope ratio 10/11 of ERM-AE123	*
$R_{c10/11}$	0.247260	0.000160	-	B	Certified isotope amount ratio 10/11 of IRMM-011	97.8 %
$K_{10/11}$	0.999068	0.000648	-	B	Factor for correcting mass fractionation	*
$R_{123-10/11}$	0.247387	0.000162	-	B	Isotope amount ratio $^{11}B/^{10}B$ in ERM-AE123, standard uncertainty	
$R_{123-10/11}$	0.24739	0.00032	-	B	Isotope abundance ratio $^{11}B/^{10}B$ in ERM-AE123, expanded uncertainty (k=2)	

*   Intermediate result

Equation system:

$I_{c89/88} = 1 / I_{c88/89}$;
$I_{123-89/88} = 1 / I_{123-88/89}$;
$R_{co10/11} = 1 / (I_{c89/88} - K_S)$;
$R_{123o10/11} = 1 / (I_{123-89/88} - K_S)$;
$K_{10/11} = R_{c10/11} / R_{co10/11}$;
$R_{123-10/11} = K_{10/11} * R_{123o10/11}$;

Table 9: Uncertainty budget for the isotope amount ratio n(^{10}B)/n(^{11}B) of ERM-AE124 as determined by TIMS

Quantity	Value	Standard uncertainty	Unit	Type A/B	Description	Index
$I_{c88/89}$	0.24744217	0.00000899	-	A	Ion current ratio 88/89 of standard IRMM-011	0.3 %
$I_{124\text{-}88/89}$	23.61369	0.00172	-	A	Ion current ratio 88/89 of candidate reference material ERM-AE124	1.9 %
$I_{c89/88}$	4.041348	0.000147	-	A	Ion current ratio 89/88 of standard IRMM-011	*
$I_{124\text{-}89/88}$	0.04234832	0.00000308	-	A	Ion current ratio 89/88 of candidate reference material ERM-AE124	*
K_S	0.0007900	0.0000100	-	B	Oxygen correction	0.0 %
$R_{co10/11}$	0.24749055	0.00000902	-	B	Observed, oxygen corrected isotope ratio 10/11 of standard IRMM-011	*
$R_{124o10/11}$	24.06257	0.00606	-	B	Observed, oxygen corrected isotope ratio 10/11 of ERM-AE124	*
$R_{c10/11}$	0.247260	0.000160	-	B	Certified isotope amount ratio 10/11 of IRMM-011	97.8 %
$K_{10/11}$	0.999068	0.000648	-	B	Factor for correcting mass fractionation	*
$R_{124\text{-}10/11}$	24.0402	0.0167	-	B	Isotope amount ratio ^{11}B/^{10}B in ERM-AE124, standard uncertainty	
$R_{124\text{-}10/11}$	24.040	0.033	-	B	Isotope abundance ratio ^{11}B/^{10}B in ERM-AE124, expanded uncertainty (k=2)	

*   Intermediate result

Equation system:

$I_{c89/88} = 1 / I_{c88/89}$;
$I_{124\text{-}89/88} = 1 / I_{124\text{-}88/89}$;
$R_{co10/11} = 1 / (I_{c89/88} - K_S)$;
$R_{124o10/11} = 1 / (I_{124\text{-}89/88} - K_S)$;
$K_{10/11} = R_{c10/11} / R_{co10/11}$;
$R_{124\text{-}10/11} = K_{10/11} * R_{124o10/11}$;

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