PRACTITIONER'S REPORT

Purity determination as needed for the realisation of primary standards for elemental determination: status of international comparability

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Abstract Within the National Metrology Institutes (NMIs) and designated laboratories, an interlaboratory comparison, CCQM-P107, was conducted to verify the degree of international comparability concerning the results of purity analysis. The mass fractions of Ag, Bi, Cd, Cr, Ni, Tl at the lower mg/kg-level in a high purity zinc material were determined, but the real measurand in metrological sense was the sum of the six mass fractions. Homogeneity was investigated by glow discharge mass spectrometry, reference values were obtained using isotope dilution mass spectrometry. Six NMIs participated, contributing eight independent data sets. The agreement amongst the results of the participants, their median and the agreement with the reference values were usually excellent and in almost all cases below the target uncertainty of 30% relative. In this manner, the accuracy of results and the comparability between the participants was demonstrated to be established.

Keywords Interlaboratory comparison · Purity analysis · High purity metals · CCQM-P107 · Zinc

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Introduction

High purity materials with known purity are convenient embodiments of the International System of Units (SI) that can serve as primary standards for elemental determination. Primary solutions prepared from the materials or solutions linked to these primary solutions are widely used for calibration in many fields of application. Ideal purity does not exist for real materials; the actual purity of a high purity material must be determined. Each mass fraction of the possible impurities needs to be ascertained and their sum subtracted from the ideal purity of 100%. There is no method of measurement available to determine the purity of a high purity material with a target uncertainty of 10^{-4} relative, a value which is one order of magnitude lower than the common uncertainty of isotope dilution mass spectrometry (IDMS) or other ratio methods. For inorganic chemical analysis, impurities include all elements of the Periodic Table other than the matrix element.

The realisation and dissemination of primary standards is one of the fundamental tasks of the National Metrology Institutes (NMIs). For inorganic chemical analysis, the NMIs are internationally organised in the IAWG (Inorganic

Analytical Working Group) of the CCQM (Consultative Committee on Amount of Substance), under the diplomatic treaty of the meter convention. Because of its fundamental importance, an interlaboratory comparison CCQM-P62 [1] was organized 2005 within CCQM to verify the degree of international comparability concerning the results of purity analysis. As the effort to comprehensively undertake purity analysis with respect to all possible impurities is huge, a deliberate limitation to six defined metallic analytes was made. The matrix element in CCQM-P62 was high purity nickel. The reported sum of the six mass fractions ranged from 3.1 to 25.4 mg/kg, a discrepancy of not less than eightfold. Inhomogeneity problems were excluded due to the results of extensive glow discharge mass spectrometry (GD-MS) measurements. Reference measurements using isotope dilution mass spectrometry (ID-MS) were additionally applied and confirmed the lower values reported in CCQM-P62. The unsatisfying outcome of the pilot study CCQM-P62 resulted in a decision to repeat the exercise with a different matrix material and different set of analytes.

Herein we report on the outcome of this new interlaboratory comparison organised in 2007–2008 amongst NMIs and designated laboratories, which is the pilot study CCQM-P107. The task was to determine the purity of high purity zinc with respect to the six metallic analytes Ag, Bi, Cd, Cr, Ni and Tl. Reporting the sum of the mass fractions $w(\Sigma_{\text{Imp}}) = w(\text{Ag}) + w(\text{Bi}) + w(\text{Cd}) + w(\text{Cr}) + w(\text{Ni}) + w(\text{Tl})$ and the mass fractions of the individual impurities together with the corresponding uncertainties according to the GUM [2] was mandatory. Zinc was chosen as the matrix because it is considered to be easily solubilised and of high technical and economic relevance. The deliberate limitation to six metallic analytes was made again in order to restrict the measurement effort for this pilot study. The material was selected such that the individual mass fractions of the six requested analytes were in the range of 0.1–5.0 mg/kg. A rather large relative target standard uncertainty of 30% on the individual impurities was considered to be fit for purpose because for a high purity material even large relative uncertainties of the individual impurities contribute minimally on an absolute scale to the final value for purity. Each participant was free to use any suitable method for the determination of the individual impurities. Although the interlaboratory comparison was deliberately limited to only six metallic analytes, it was suggested that multi-element methods such as, inductively coupled plasma with mass spectrometry (ICP-MS), GD-MS and inductively coupled plasma optical emission spectrometry (ICP-OES) are used as they are typically employed for such broad-based applications. Preferred procedures were those which provided traceability of the measurement results in the shortest possible way (e.g., via calibration against sufficiently pure substances of well

known stoichiometry); however, procedures requiring matrix reference materials for calibration (e.g., special types of solid sample measurements) were not excluded if the matrix reference material used was stated and of sufficient metrological quality and if the uncertainties on the values of the matrix reference material were taken into account in the calculation of uncertainty. Although typical mono-element (or oligo-element) techniques such as AAS (atomic absorption spectrometry) and ID-MS were not excluded, they were not the real target group.

Experimental

The starting material was prepared by water jet cutting of compact zinc from Alfa Aesar Johnson Mattey, Karlsruhe/DE having a nominal metal based purity of 99.99%. From the material obtained, for each participant six pieces were randomly selected for one bottle containing about 3 g of the material. Before use, chemical etching was recommended to remove surface contamination. Measurements for homogeneity were performed at BAM using GD-MS. Measurements within piece and between pieces were performed under repeatability conditions. For the six individual pieces, five repeat measurements were undertaken. ID-MS after matrix separation using inductively coupled plasma with sector field mass spectrometry (ICP-SF-MS) for Cr and thermal ionisation mass spectrometry (TIMS) was applied for Ag, Cd, Ni and Tl at BAM. In total, six NMIs participated using GD-MS, ICP-SF-MS, Inductively Coupled Plasma with Quadrupole Mass Spectrometry (ICP-Q-MS), Electrothermal Atomic Absorption Spectrometry (ET-AAS) and ICP-OES contributing eight independent data sets. Each laboratory selected its own measurement and calibration procedure. In all cases, except for GD-MS, the sample was chemically dissolved prior to analysis.

Results and discussion

Homogeneity and reference values

The results of the homogeneity testing are presented in the first three lines of Table 1. The nominal mass fractions (line 1, Table 1) obtained from uncalibrated measurements are given for information only. An F-test comparing the relative standard deviation within (line 2, Table 1) and between (line 3, Table 1) the individual samples reveals Cr, Tl and Cd are homogeneously distributed and Bi and Ni are nearly homogeneous whereas silver is not homogeneously distributed throughout the material. However, more important than a purely statistical evaluation is an examination of the

Table 1 Compilation of data from homogeneity study, reference measurements and results from interlaboratory comparison

Line	Quantity		Ag	Bi	Cd	Cr	Ni	Tl	Σ_{Imp}
1	Nominal mass fraction by GD-MS	$w/(\text{mg/kg})$	0.94	0.054	1.27	3.59	4.06	0.79	10.7
2	Relative spread within piece	$s_{\text{rel}}/\%$	2	4	6	10	3	7	
3	Relative spread between pieces	$s_{\text{rel}}/\%$	10	10	11	15	7	7	
4	Relative inhomogeneity contribution	$s_{\text{rel}}/\%$	10	10	11	20	10	7	8
5	ID-MS values	$w_{\text{IDMS}}/(\text{mg/kg})$	1.032	–	1.105	3.0	3.83	0.769	
6	ID-MS uncertainties	$u_{\text{IDMS}}/(\text{mg/kg})$	0.005	–	0.007	0.5	0.14	0.002	
7	Reference values	$w_{\text{Ref}}/(\text{mg/kg})$	1.03	0.053	1.11	3.0	3.8	0.77	9.7
8	Uncertainty of reference values	$u_{\text{Ref}}/(\text{mg/kg})$	0.10	0.009	0.12	0.6	0.4	0.05	0.7
9	Mean of interlaboratory results	$w_{\text{Mean}}/(\text{mg/kg})$	1.00	0.052[a] 0.060	1.1	2.6	3.7	0.76	9.3
10	Spread of interlaboratory results	$s_{\text{Mean}}/(\text{mg/kg})$	0.12	0.006[a] 0.025	0.3	0.5	0.4	0.11	0.9
11	Relative spread of interlaboratory results	$s_{\text{Mean\_rel}}/\%$	12	[a]11 41	29	20	11	15	10
12	Median of interlaboratory results	$w_{\text{Med}}/(\text{mg/kg})$	1.01	0.053	1.11	2.6	3.7	0.80	9.2
13	MAD of interlaboratory results	$s_{\text{Med}}/(\text{mg/kg})$	0.12	0.007	0.13	0.4	0.3	0.03	1.0

[a] Without upper limit value for Bi from Lab #13

uncertainties involved. A maximum relative spread (from lines 2 and 3, Table 1) of about 15% was obtained for Cr. For Cr and Ni also the laboratories ID-MS BAM, #6 and #7 reported an unusually high spread in their results, which suggested the existence of inhomogeneities. From these results, the inhomogeneity contributions to the uncertainty for Cr and Ni represented as relative standard deviation (line 4, Table 1) were set to 20 and 10%, respectively. As the requirements concerning steadiness and purely statistical distribution of the inhomogeneity do not seem to be fulfilled, an evaluation of the homogeneity using ANOVA was not considered to be useful, and the pragmatic approach of a worst case estimate was applied. With a target uncertainty of 30% relative for this interlaboratory comparison, the material was still considered to be sufficiently homogeneous and thus fit for purpose. The relative imprecision of GD-MS at the investigated concentration level is estimated to be 2–5% and negligible for the inhomogeneity statement.

Reference measurements using ID-MS were applied whenever possible. Due to its methodology, ID-MS is known to have the potential to establish reference values [3], although it is by nature not infallible just by definition. The results using ID-MS are summarised in lines 5 and 6 of Table 1. For Cr and Ni, an unusually high spread was observed in parallel determinations. This spread is reflected in the rather large relative uncertainties of ID-MS for Cr and Ni as usually about 1% relative can be achieved. The reason for the spread observed is assumed to be inhomogeneity. The ID-MS values were used as reference values (line 7, Table 1) in this study. For Bi, which is a mono-

isotopic element not accessible by ID-MS, the median of the reported results (from line 12, Table 1) of $w(\text{Bi}) = 0.053$ mg/kg was used as reference value. For the uncertainty u_{Ref} of the reference values, calculated in line 8 of Table 1, the relative inhomogeneity contributions s_{rel} from line 4 of Table 1 needed to be taken into account. Note that for Cr and Ni, the contribution due to inhomogeneity should not be incorporated twice as it is already partly included in the uncertainty of the ID-MS values in line 6. For Bi, an uncertainty $u_{\text{Ref}}(\text{Bi}) = 0.009$ mg/kg is obtained from Table 1 line 4 and 13. Compared to the other analytes, the value of Bi and its uncertainty is negligible. The reference value and its uncertainty for the sum of the six mass fractions containing all inhomogeneity contributions is calculated to be $w(\Sigma_{\text{Imp}}) = (9.7 \pm 0.7)$ mg/kg.

Results of interlaboratory comparison

The individual results of the eight data sets reported by the participants have been grouped in ascending order. The value (A) obtained by ID-MS, the arithmetic mean value (M) for all eight participants, the reference value (Ref) and the median (Med) of the eight data sets are depicted in Figs. 1, 2, 3, 4, 5, 6 and 7. The numerical values for the arithmetic mean, the median and the corresponding uncertainties calculated from all eight participants' data are compiled in lines 9–13 of Table 1. The participating laboratories and their methods used are given in Table 2.

Within the known limits of the explanatory power of pure statistics for the reality of interlaboratory comparisons

Fig. 1 Mass fractions w for silver in zinc ($k = 1$). The *solid* and *dottedline* represent the reference value and its uncertainty, respectively. *Numbers* below the institute name represent the laboratory code
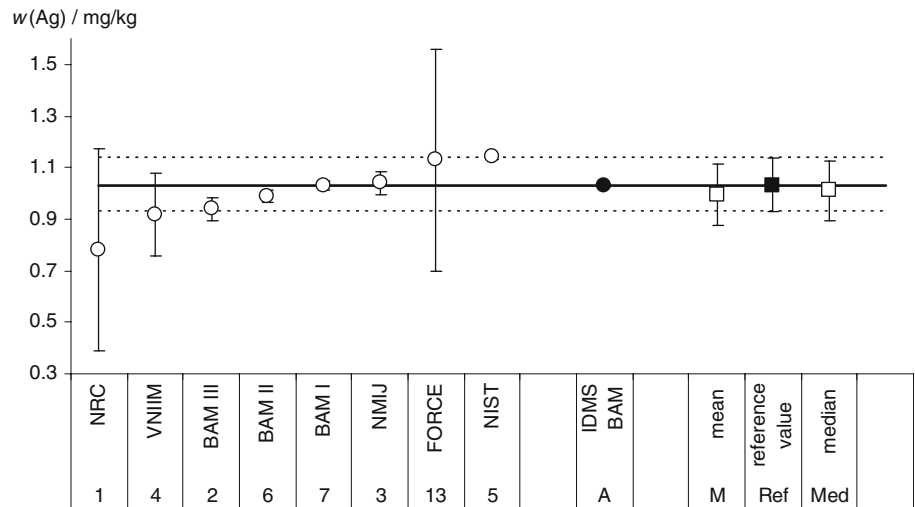

Fig. 2 Mass fractions w for bismuth in zinc ($k = 1$). The *solid* and *dotted line* represent the reference value and its uncertainty, respectively. *Numbers* below the institute name represent the laboratory code

Fig. 3 Mass fractions w for cadmium in zinc ($k = 1$). The *solid* and *dotted line* represent the reference value and its uncertainty, respectively. *Numbers* below the institute name represent the laboratory code
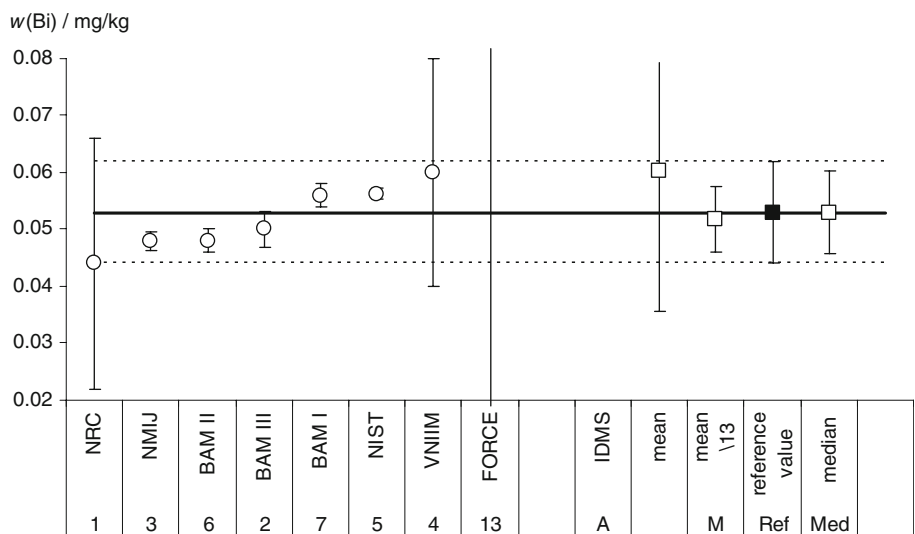
Fig. 4 Mass fractions w for chromium in zinc ($k = 1$). The *solid* and *dotted line* represent the reference value and its uncertainty, respectively. Numbers below the institute name represent the laboratory code
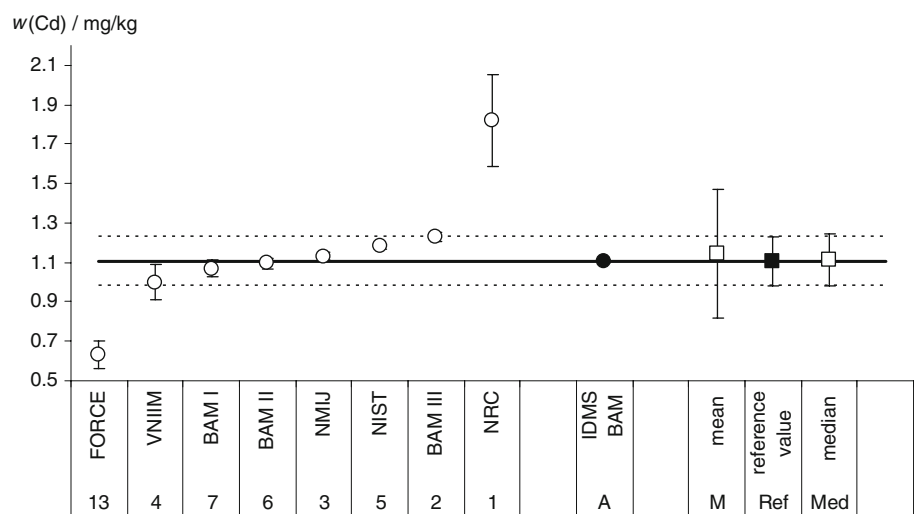

Fig. 5 Mass fractions w for nickel in zinc ($k = 1$). The *solid* and *dotted line* represent the reference value and its uncertainty, respectively. *Numbers* below the institute name represent the laboratory code

Fig. 6 Mass fractions w for thallium in zinc ($k = 1$). The *solid* and *dotted line* represent the reference value and its uncertainty, respectively. *Numbers* below the institute name represent the laboratory code
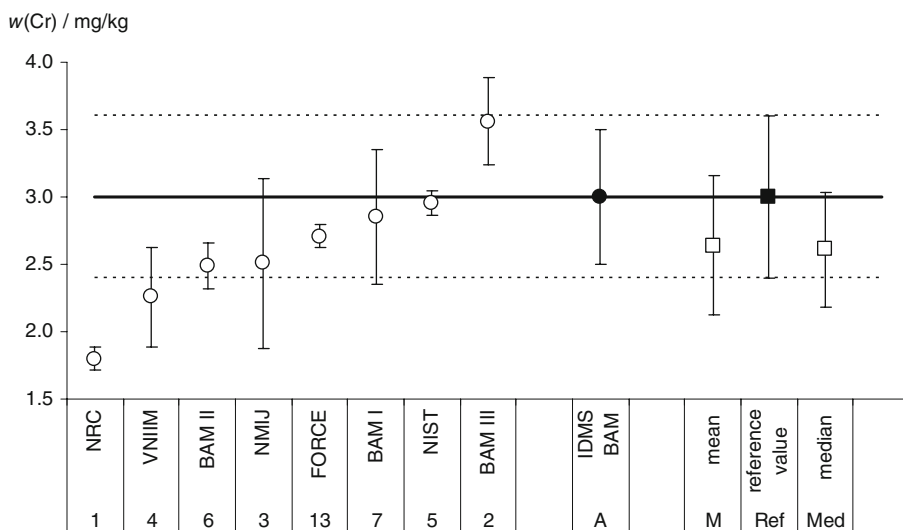
Fig. 7 Mass fractions w for total impurity of zinc with respect to the six defined analytes ($k = 1$). The *solid* and *dotted line* represent the reference value and its uncertainty, respectively. *Numbers* below the institute name represent the laboratory code
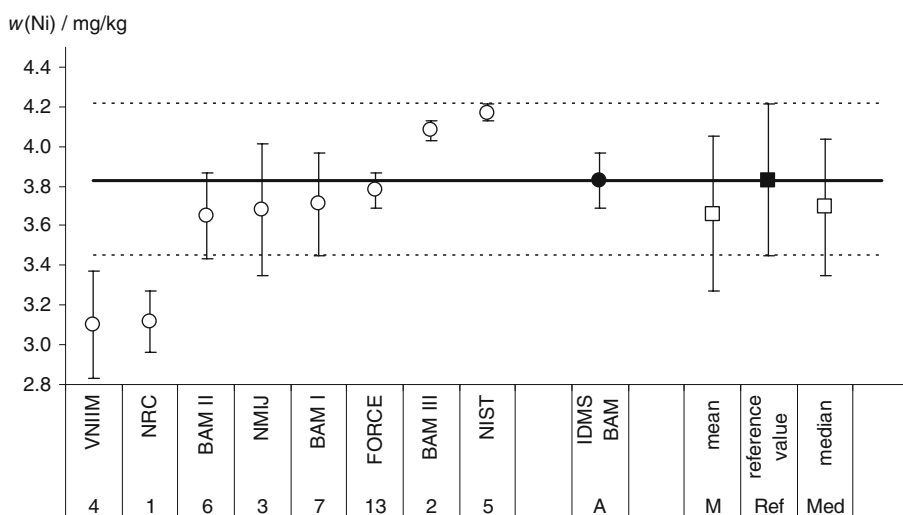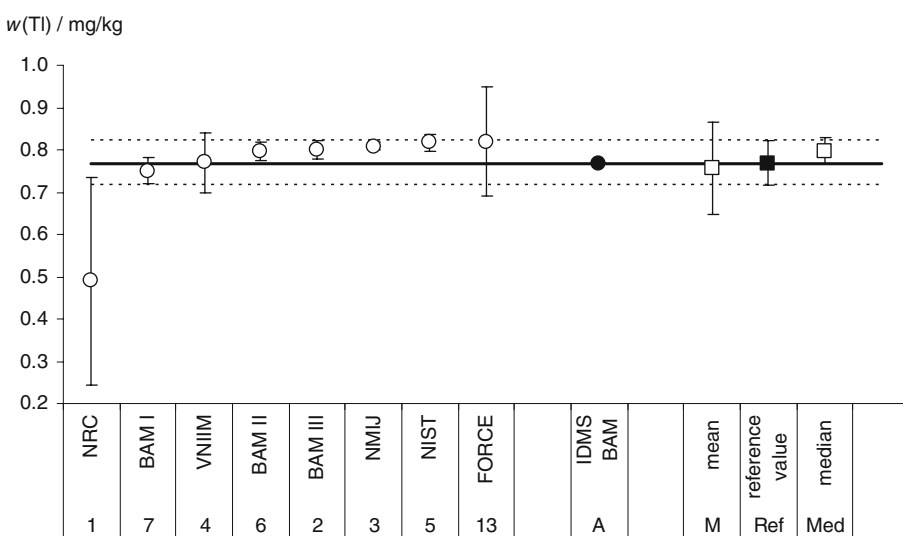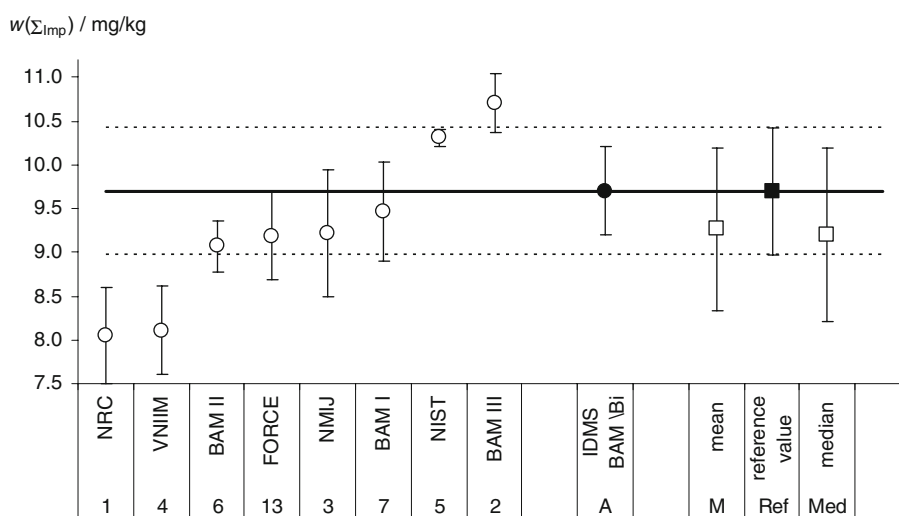
concerning preconditions, a Grubbs test at a significance level of $\alpha = 5\%$ was performed on the measurement results reported by the participants. As the Grubbs test does not take any uncertainty information into account, a value can be detected as a statistical outlier, although it may be technically very consistent with the rest of the data set. Such a case is highlighted for the result for Bi from lab #13. Due to the fact that ICP-OES has poorer detection power compared to the other methods used, a rather high upper limit value (i.e., $w(\text{Bi}) < 0.24$ mg/kg) was reported which, transformed to a statement of $w(\text{Bi}) = (0.12 \pm 0.12)$ mg/kg, carries a rather large uncertainty. For the calculation of the reference value for Bi based on the median, the value obtained by ICP-OES was included. Other statistical outliers occurred for Cd values from lab #1 and lab #13 and for Tl from #1. The latter is also technically not inconsistent with the rest of the data set. The detected statistical outliers are in line with the observation that the relative standard deviation calculated for the mean value of the eight individual data sets from the participants in line 11 of Table 1 was three- and twofold higher for Cd and Tl, respectively, than the inhomogeneity contribution expressed as s_{rel} in line 4 of Table 1. For Ag, Bi (excluding the high upper limit value from lab #13), Cr, Ni and the sum of the impurities, the relative standard deviation (line 11, Table 1) from the interlaboratory comparison was very consistent with the contribution from calculated relative inhomogeneity.

Use of both the median and arithmetic mean have their advantages and disadvantages for the evaluation of the interlaboratory comparison results. For ideal (or close to ideal) data sets, as in the case of Ag, Bi (excluding the upper limit value from lab #13), Cr, Ni and the sum of the six impurities, there is hardly a difference concerning conclusions drawn from both types of evaluation. In the presence of outlying values, as in the data sets for Cd and

Tl, the median is more robust and the corresponding MAD (median of absolute deviation calculated as $s_{Med} = 1.458 \cdot \text{median}(|X_i - \text{median}(X)|)$) is consequently smaller, here a factor of about three, than the corresponding standard deviation of the arithmetic mean value. From this, the median seems to be more appropriate for the evaluation of the interlaboratory comparison. However, the median is more stringent in detecting inconsistent measurement results (i.e., values with small claimed uncertainty but large bias relative to the reference value) than the arithmetic mean.

The agreement between the ID-MS results, and consequently the reference values derived from them, and the medians of the interlaboratory comparisons is excellent, even within $k = 1$. The uncertainties of the reference values and of the corresponding medians (i.e., the MAD) are about identical for Ag, Bi, Cd, Cr, Ni and the sum of the six impurities, which is a good indication that the median is an appropriate way of evaluating the interlaboratory comparison data. For Tl, the MAD is about half of the uncertainty of the reference value, which can be an indication for an overestimate of the inhomogeneity contribution for Tl. However, this interpretation should not be overstressed. The excellent agreement of the values from the interlaboratory comparison and the reference values is a significant improvement with respect to the outcome of the previous pilot study, CCQM-P62.

Each result reported by the eight participants for the six measurands was tested with respect to the reference values and also with respect to the medians of the interlaboratory comparison. The results are compiled in Table 2.

The column headed "S" in Table 2 indicates whether the measurement result reported is consistent with the reference value according to Eq. 1, i.e., if the difference between the reported measurement result w_i and the reference value w_{Ref} is less than the expanded uncertainty

Table 2 Compilation of results from comparing each measurement result reported to the reference values and medians of the interlaboratory comparison

No	Origin of Value	Method	Ag S	Ag R	Ag C	Ag U	Bi S	Bi R	Bi C	Bi U	Cd S	Cd R	Cd C	Cd U	Cr S	Cr R	Cr C	Cr U	Ni S	Ni R	Ni C	Ni U	Tl S	Tl R	Tl C	Tl U	ΣImp S	ΣImp R	ΣImp C	ΣImp U
1	NRC	GD-MS	+	+	+	−		+	+	−	−	−	+	+	+	+	−	+	+	+	+	+	+	+	−	−	+	+	+	+
2	BAM III	GD-MS	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
3	NMIJ	ICP-SF-MS	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
4	VNIIM	ICP-Q-MS	+	+	+	+	+	+	+	−	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
5	NIST	ICP-Q-MS	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
6	BAM II	AAS	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
7	BAM I	ICP-SF-MS	+	+	+	−	+	+	−	−	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
13	FORCE	ICP-OES	*	*	*	+					*	*	*	+	*	*	*	+	*	*	*	+	*	*	*	+				
A	IDMS BAM	ID-MS					+	+	+	+																				
M	Mean		+	+	+	+	*	*	*	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	*	*	*	+
Ref	Reference value		*	*	*	+	*	*	*	+	*	*	*	+	*	*	*	+	*	*	*	+	*	*	*	+	*	*	*	+
Med	Median		+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
	Homogeneity					+				+				+				+				+				+				+

*, automatically consistent as a consequence of definition of Eqs. 1–3.

The column headed "S" indicates whether the measurement result reported is consistent with the reference value according to Eq. 1. The column headed "R" indicates whether a reported measurement result is consistent with the median of all participants according to Eq. 2. The column headed "C" indicates whether a reported measurement result is consistent with the reference value within a target uncertainty of 30% as calculated using Eq. 3. The column headed "U" indicates whether the reported measurement uncertainty is below the target uncertainty of 30% relative

with a coverage factor of $k = 2$. This expanded uncertainty contains the reported measurement uncertainty u_i and the uncertainty u_{Ref} of the reference value. The uncertainty of the reference value itself includes the uncertainty of its determination by ID-MS (or for Bi from the calculation of the median) and a contribution for inhomogeneity. A slight complication arises as the measurement results of some participants also include a contribution for inhomogeneity, effectively accounting for inhomogeneity twice. Irrespective of this, for the known cases of laboratories #6 and #7 for Cr and Ni, the outcome of the test would not be different.

$$(w_i - w_{Ref})/\left(2 \cdot \sqrt{u_i^2 + u_{Ref}^2}\right) < 1 \qquad (1)$$

The column headed "R" in Table 2 indicates whether a reported measurement result is consistent with the median w_{Med} of all participants according to Equation 2. Here, the expanded uncertainty contains contributions from the reported measurement uncertainty u_i, the uncertainty of the median u_{Med} (i.e., MAD) and the uncertainty contribution u_{hom} for inhomogeneity, i.e.:

$$(w_i - w_{Med})/\left(2 \cdot \sqrt{u_i^2 + u_{Med}^2 + u_{hom}^2}\right) < 1 \qquad (2)$$

The column headed "C" indicates whether a reported measurement result is consistent with the reference value w_{Ref} within a target uncertainty of 30% irrespective of the uncertainties reported for the measurement results, as calculated using Eq. 3.

$$(w_i - w_{Ref})/(0.3 \cdot w_{Ref}) < 1 \qquad (3)$$

The column headed "U" indicates whether the reported measurement uncertainty is below the relative target uncertainty of 30% of the reference value.

Methodical aspects

In the frame of this study, several methods of measurement were applied, namely: AAS, ICP-Q-MS, ICP-SF-MS, ID-MS, ICP-OES and GD-MS. More important than the method of measurement, the quality of a measurement result depends very much on the actual measurement procedure applied. Just by definition, no method is superior to another. AAS can deliver very good measurement results, however, for the purpose of analysis with respect to many analytes the capabilities of AAS are slow and it requires higher effort because it is a mono-element technique and limited in dynamic range. Nevertheless, AAS is an important supporting measurement technique for purity analysis. ICP-MS, with multi-element capability and wide dynamic range, can overcome the stated limitations of AAS. However, MS suffers from potential interferences which need to be overcome. ICP-Q-MS is, compared to

ICP-SF-MS, less expensive and requires less effort; however, in some cases there are spectral interferences which are difficult to deal with. Such interferences as (^{65}Cu, ^{64}NiH) and (^{56}Fe, ^{40}Ar^{16}O) are examples arising from a previous similar interlaboratory comparison (CCQM-P62 [1]) on the purity of Ni with respect to Ag, Al, Cu, Fe, Pb and Zn. From the point of view of ICP-Q-MS, the study reported here was easier. Interferences in ICP-Q-MS can often be overcome by use of collision cells but at the cost of reduced multi-element capability. ICP-SF-MS can overcome many problems of spectral interferences and is usually more sensitive than ICP-Q-MS, but it is a rather expensive and complex technique and requires additional effort. ID-MS after matrix separation, used here with TIMS and also in combination with ICP-MS, is an established calibration strategy for performing reference measurements. The intrinsic advantage concerning internal standardisation with the best internal standard possible (i.e., an isotope of the same element) resulting in small uncertainties is well-known. As ID-MS is a mono- or multi-element method, the effort using ID-MS for purity analysis with respect to many analytes would be large. Additionally, ID-MS is not applicable for some ten analytes as they are mono isotopic and technically there are no spikes available. Similar to ICP-MS, ICP-OES is a multi-element technique with a wide dynamic range. The different detection principle results in different characteristics concerning matrix effects, memory effects and (optical line) interferences. Compared to ICP-MS, a drawback of ICP-OES is the lower sensitivity and hence the higher limits of determination. As an example, for Bi present at a 50 μg/kg level in the material investigated in this interlaboratory comparison, ICP-OES could merely deliver a fivefold higher upper limit value as result. All the methods discussed above are based on solution sample introduction. Working with solutions has the advantage of easy calibration, but it also requires that the sample be dissolved without any contamination or losses. This is generally difficult to verify.

Solid sampling techniques, such as GD-MS, have significant advantages concerning avoidance of contamination and losses by chemical sample preparation processes. GD-MS is sensitive, multi-elemental—even for analytes such as the non-metals which are generally not accessible via ICP-MS and, compared to ICP-MS, fast relative to the entire analytical procedure. However, solid sampling methods have the disadvantage that sample adapted solid standards are needed for calibration. This can be achieved in different ways. One is the concept of standard relative sensitivity factors (standard RSF) which does not involve a measurement standard of similar composition, but is used at the cost of introducing uncertainties of a factor of two for metallic analytes. A second approach is the use of Certified Reference Materials (CRMs) of the same or similar matrix composition as the sample to be analysed. This approach can result in much smaller uncertainties, as evidenced partly in this study. A drawback of this approach is that the traceability of the measurement result is not established in the shortest possible way, as it runs over a matrix adapted measurement standard, the CRM, which itself is normally linked to the SI by the calibration solutions and applying the methods used for its certification. An additional and more practical problem associated with this approach is that certified matrix reference materials for impurities in high purity metals usually only exist for few analytes and for few concentrations. A third new calibration approach is based on the use of matrix adapted synthetic standards prepared from high purity base metal powders by gravimetrically doping them with liquid standards of known concentration [4]. A major advantage is that these standards provide traceability to the SI in the shortest possible way and that the uncertainties can be rather small. A drawback of this approach is that the preparation of such standards requires considerable effort, and there are also intricacies concerning losses, homogeneous distribution and contamination, that need to be obeyed. The feasibility of this approach was successfully demonstrated in this interlaboratory comparison by the data submitted as BAM III. These results may open a gate for a fast, directly traceable purity analysis with small uncertainty. It is noteworthy that the design of the current commercially available GD-MS is based on SF-MS, thereby offering the same possibilities of overcoming spectral interferences as does ICP-SF-MS.

Conclusions

Based on the median, the observed spread of the measurement results reported by the NMIs and designated laboratories for Ag, Bi, Cd, Cr, Ni, Tl and the sum of these impurities in the zinc material are 12, 14, 12, 16, 9, 4 and 11%, respectively, which is well below the target uncertainty of 30% relative. As a consequence, comparability between the participating NMIs and designated laboratories is demonstrated to be established. The individual measurement results and the medians derived were in all cases consistent with the reference values. As a consequence, accuracy of the measurement results for the NMIs and designated laboratories participating is demonstrated to be established. In particular, having the results of the previous study CCQM-P62 in mind, the outcome of this study can be seen as a big step forward for the community.

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