UNDERSTANDING METROLOGICAL TRACEABILITY A survey-based exploration of perceptions

Published: October 2022

Eurachem **b**

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* Subject to journal requirements

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Foreword

Eurachem Reports provide summaries of work conducted by Eurachem Working Groups or others on behalf of the Eurachem Executive. Eurachem reports are issued for information only and do not constitute guidance or statements of policy.

This Report summarises responses received in a perception survey undertaken in support of the Eurachem Measurement Uncertainty and Traceability Working Group. The survey was undertaken to provide insight into analytical and other measurement scientists' perception of some metrological issues with a view to providing improved guidance on the topic.

1 Introduction – Why conduct a survey?

Metrological traceability is a fundamental concept in metrology and accreditation. It is the subject of a Eurachem guide [1]. The concept of metrological traceability is simple in principle but can become intricate in practice. In particular:

- Although the Eurachem guide is clear on the role of validation and QC materials in establishing metrological traceability, little guidance is given on the claims of traceability that can be made based on the origin of validation and QC materials;
- The effect of 'recalibration' use of a secondary material to correct a measurement result from a system that has already been calibrated is not considered in most guidance;
- Metrological traceability for reference values obtained by interlaboratory study a common practice in certification of natural matrix reference materials has been widely debated and there remains room for differing opinions.

Eurachem has run a voluntary-response, scenario-based online exercise to probe perceptions of traceability in the metrology community. The intent was primarily to establish the extent of consensus, or otherwise, on the issues listed above. This document provides a summary of the questions and the response counts for information; free text responses are not included. Further publications in the open literature will include critical appraisal as well as free-text responses where given.

2 Conduct and timing of the survey

The survey was an online survey operated via Google Forms. The survey was launched by email and via social media 11 January 2019, with reminders in February 2019. The primary audience was analytical chemists contacted via Eurachem national in international networks. In addition, a link to the survey was distributed directly to available national measurement institute contacts. Contribution from other fields of measurement were invited.

Data collection closed: 22 March 2019; at that time, a total of 464 responses were received.

3 Survey format

The survey included a minimal number of respondent classification questions, and two technical sections. In part 1, respondents were invited to self-classify by Organisation type and Field of study; no other respondent information was collected.

The technical questions each presented a practical scenario with a short set of questions relating to metrological traceability and measurement uncertainty. Part 2 included five laboratory scenarios, increasing in complexity. Part 3: (presented on request only) set of four Reference Material certification scenarios of increasing complexity.

All but closing comment questions were check-box or multiple response with free text 'Other' field. Only the respondent classifications were mandatory. Respondents were permitted to edit responses or submit further responses at a later date.

Details of the scenarios and questions, together with summaries of responses, are given in the following three sections. Section 7 includes a breakdown by organisation type or filed of measurement for those questions where there was a significant effect on response.

4 Part 1. Respondent classification

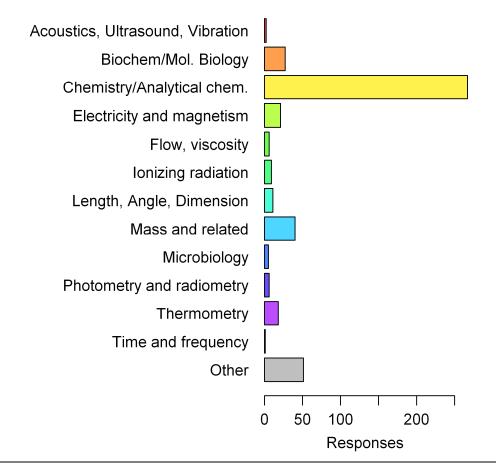
4.1Field of work

Please tick the answer that best describes your main field of measurement work?

Possible responses:

- Acoustics, Ultrasound, Vibration
- Biochemistry, molecular biology
- Chemistry/Analytical chemistry
- Electricity and magnetism
- Flow, viscosity
- Ionizing radiation
- Length, angle or other dimensional metrology
- Mass and related measurement (Force, pressure, hardness, gravity etc)
- Photometry and radiometry
- Time and frequency
- Thermometry
- Other [Free text entry]:

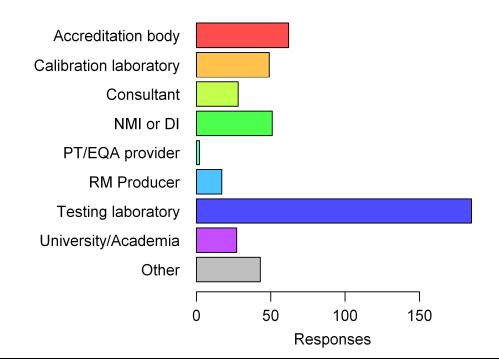
Response counts



4.2Type of organisation

Which of the following best describes your organisation's role? If your organisation fulfils more than one role on this list, use the description most suited to your own work area.

- Routine measurement laboratory
- Academic research institute
- Calibration laboratory
- National Measurement Institute (including Designated Institute)
- Accreditation body
- Reference Material Producer
- Consultant
- Other [Free text entry]:



Response counts*

*Labels are abbreviations of the full list above. In particular, "Testing laboratory" in the Figure refers to "Routine measurement laboratory" in the response list.

5 Part 2: Metrological traceability in practice

[The following introduction to this Part was provided]

The following questions summarise some scenarios in which the concept of metrological traceability is relevant and, sometimes, contentious. Please examine each scenario and choose the answer that best reflects your opinion. An additional comment box is available in most cases.

NOTES:

1. Some scenarios are intentionally controversial or subject to interpretation, so more than one statement may seem true or partially true; in those cases, please pick the statement that comes closest to your view.

2. We understand that some cases may be unfamiliar. If you do not feel that you can answer meaningfully, please leave the question blank and move to the next question; none of the questions is mandatory.

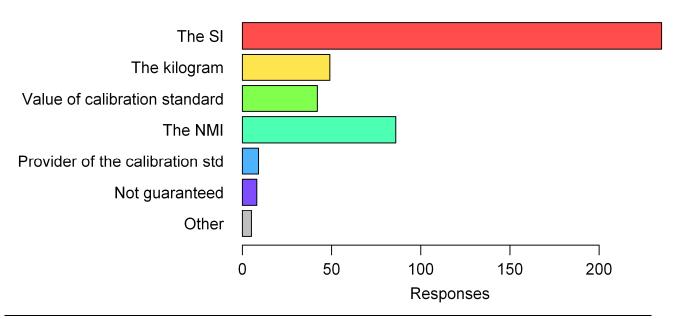
5.1Scenario 1 – A simple measurement

A sample is weighed, following an accredited procedure, using an instrument that has been calibrated at the time of measurement with a measurement standard (in kg) provided by an accredited calibration laboratory. The calibration laboratory calibrates using standards provided by their National Measurement Institute (NMI) (for example, NIST, PTB, NMIJ, ...).

5.1.1 Scenario 1 Q1: Metrological traceability

Which statement below best represents your view of the metrological traceability of the result (measured mass)?

- The measurement result is traceable to the National Measurement Institute
- No guarantee of metrological traceability can be given
- The measurement result is only traceable to the provider of the calibration standard
- The measurement result is traceable to the value of the calibration standard
- The measurement result is traceable to the calibration laboratory
- The measurement result is traceable to the SI
- The measurement result is traceable to the kilogram
- Other [Free text entry]



5.2Scenario 2: Use of a certified check sample for quality control

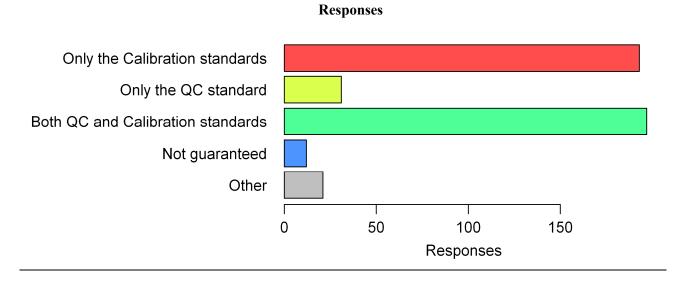
A test piece or sample is measured using a measuring instrument calibrated with properly certified calibration standards, traceable to the SI. The measurement result is calculated using the values of the calibration standards.

In addition, the laboratory uses a certified reference standard (also traceable to the SI) as a quality control (QC) check. The result for the QC standard is within expected QC criteria.

5.2.1 Scenario 2 Q1: Traceability

Which statement below best represents your view of the metrological traceability of the measurement result?

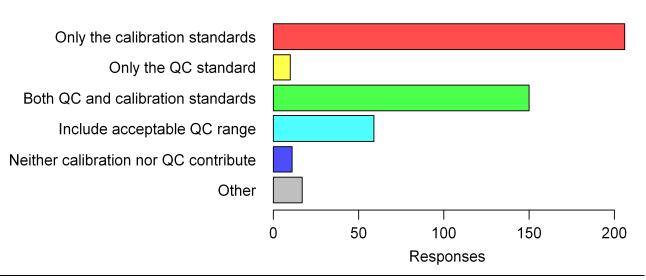
- No guarantee of metrological traceability can be given
- The measurement result is traceableto the value of BOTH the certified QC standard
- AND the values of the calibration standards
- The measurement result is traceable only to the values of the calibration standards
- The measurement result is traceable only to the value of the certified QC standard
- Other [Free text entry]:



5.2.2 Scenario 2 Q2: Measurement uncertainty

The calibration standard and the quality control standard have uncertainties. Which statement below best represents your view of how these two uncertainties should affect the measurement uncertainty in the result?

- The uncertainty budget should include a contribution based on the acceptable range
- for the QC material, as well as other uncertainties
- The uncertainty budget should include contributions from BOTH the certified QC standard AND the calibration standards
- Only the uncertainties for the calibration standards should be included in the uncertainty budget for the measurement result
- Only the uncertainty for the QC standard should be included in the uncertainty budget for the measurement result
- Neither the calibration standards nor the QC check standard contribute to the uncertainty in the result
- Other [Free text entry]



5.3Scenario 3: Correction based on observed value for a certified check sample

A test piece or sample is measured using a measuring instrument calibrated with properly certified calibration standards, traceable to the SI. The result is calculated using the values of the calibration standards.

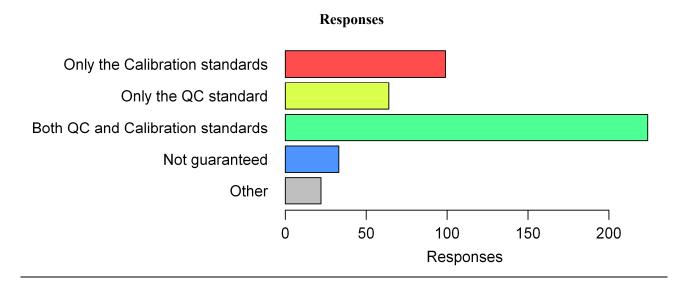
The laboratory includes a certified reference standard (also traceable to the SI) in the measurement run as a quality control (QC) check. The result for the QC standard is within expected QC criteria.

In addition to confirming that the QC value is within limits, the laboratory makes a correction to the measurement result based on the observed value for the check sample (on the basis that, for example, correcting for the QC deviation should remove any 'run' bias)

5.3.1 Scenario 3 Q1: Traceability

Which statement below best represents your view of the metrological traceability of the result?

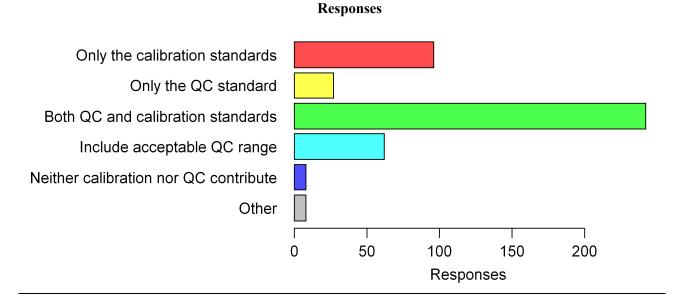
- The measurement result is traceable only to the value of the certified QC standard
- The measurement result is traceable only to the values of the calibration standards
- No guarantee of metrological traceability can be given
- The measurement result is traceable to the value of BOTH the certified QC standard AND the values of the calibration standards
- Other [Free text entry]



5.3.2 Scenario 3 Q2: Measurement uncertainty

The calibration standard and the quality control standard have uncertainties. Which statement below best represents your view of how these two uncertainties should affect the measurement uncertainty in the result?

- The uncertainty budget should include contributions from BOTH the certified QC standard AND to the calibration standards
- Neither the calibration standards nor the QC check standard contribute to the uncertainty in the result
- Only the uncertainty for the QC standard should be included in the uncertainty budget for the measurement result
- Only the uncertainties for the calibration standards should be included in the uncertainty budget for the measurement result
- The uncertainty budget should include a contribution based on the acceptable range for the QC material, as well as other uncertainties
- Other [Free text entry]



5.4Scenario 4: An 'operationally defined' measurement

An 'operationally defined' measurement determines a measurand that is defined _only_ by reference to a standardised procedure, which describes important conditions of measurement (such as standard exposure time or temperature, reagent concentrations, reacton time, impact velocity etc). Many standard test procedures are 'operationally defined'; examples include polymer melt index, fat content or crude fibre in foodstuffs, enzyme activity measurements, indentation hardness, 'flakiness index' for building aggregates and so on.

In this scenario, a laboratory determines the crude fibre content of a foodstuff using a published standard procedure. The 'crude fibre' content is defined as the mass of combustible solid material remaining after digesting a weighed mass of foodstuff in acid solution (of defined acid concentration) for a specified time and temperature. After digestion, the solid residue is weighed, heated to combustion temperature to ensure combustion of 'fibre', and weighed again; the crude fibre content is the difference between the two masses. The result is expressed as the mass lost on combustion, divided by the mass of foodstuff taken (that is, as mass fraction of fibre in the foodstuff, usually expressed as a percentage). Only the initial mass and the mass lost in combustion enter into the calculation of the result. It is, however, known that changes in the digestion time, temperature, and acid concentration and in combustion time and temperature would substantially affect the measured values.

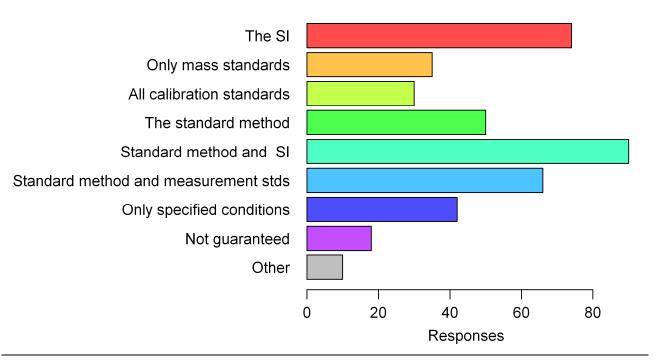
The laboratory calibrates all the equipment used, using appropriate measurement standards or calibration services, and makes up the acid solution using appropriately calibrated equipment. All calibrations are traceable to the SI.

[Question and responses overleaf]

5.4.1 Scenario 4 Q1: Metrological traceability

Which statement below best represents your view of the metrological traceability of the crude fibre result?

- The result is traceable only to the conditions specified in the standard method
- The measurement result is traceable only to the values of the mass standards used to calibrate balance used in the initial and final mass measurement
- The measurement result is traceable to the SI
- The measurement result is traceable only to the standard method
- The measurement result is traceable both to the standard method and to the SI
- The measurement result is traceable to all individual calibration standards used
- No guarantee of metrological traceability can be given
- The measurement result is traceable both to the standard method and to the measurement standards used for calibration
- Other [Free text entry]



5.5Scenario 5: An 'operationally defined' measurand determined using a secondary procedure

(See also Scenario 4)

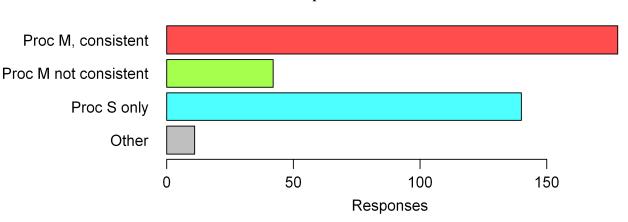
Although an 'operationally defined' measurand is defined by reference to a standardised procedure, it is sometimes possible to calibrate a secondary procedure using reference standards with values assigned using the defining test procedure.

In this Scenario, a laboratory first determines the moisture content of a suitable number of typical materials, using the published standard procedure (procedure M) with SI-traceable calibration for all relevant measurement standards. These materials are then used to calibrate a spectroscopic procedure (procedure S) which is intended to provide values for the measurand defined by procedure M. The calibration is checked (using independent materials) and found to provide results that are consistent with the results from the defining procedure, procedure M. The calibrated instrument is then used to measure routine samples.

5.5.1 Scenario 5 Q1: The measurand

Which statement below best describes the measurand ("quantity intended to be measured") for the routine samples?

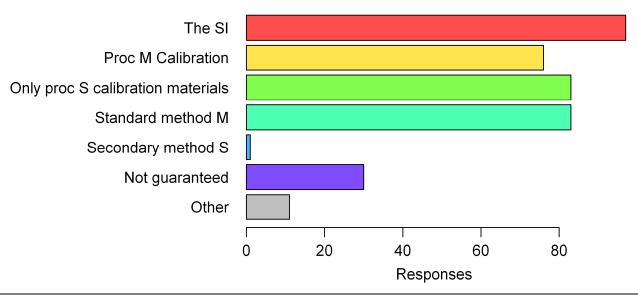
- The measurand is 'moisture content as defined by procedure M' and the spectroscopic procedure does provide values consistent with that definition
- The measurand is 'moisture content as defined by procedure M' but the spectroscopic procedure can not be claimed to provide values consistent with that definition
- The measurand for the spectroscopic procedure can only be described as 'moisture determined by procedure S'
- Other [Free text entry]



5.5.2 Scenario 5 Q2: Metrological traceability

Which statement below best represents your view of the metrological traceability of the results from the secondary procedure?

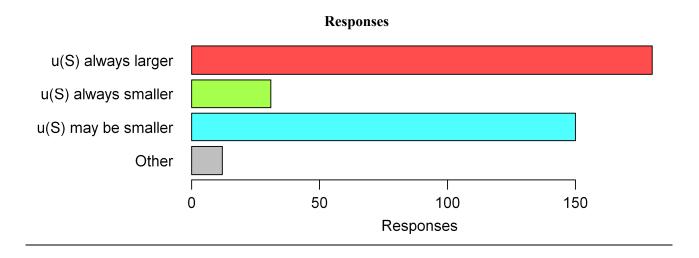
- The measurement result is traceable to the SI
- The measurement result is traceable to the standard method (procedure M)
- The measurement result is traceable only to the values for the typical materials used to calibrate procedure S
- The measurement result is traceable to the standards used to calibrate the measurement conditions for procedure M
- No guarantee of metrological traceability can be given
- Other [Free text entry]



5.5.3 Scenario 5 Q3: measurement uncertainty

For the scenario above, procedure S shows a smaller repeatability standard deviation than procedure M. Which of the following statements best describes your view of the measurement uncertainty for the routine samples measured by the spectroscopic procedure (procedure S)?

- The measurement uncertainty for procedure S must always be larger than for procedure M
- The measurement uncertainty for procedure S must always be smaller than for procedure M
- Procedure S may be able to deliver a smaller measurement uncertainty than procedure M, depending on how many independent materials and results from procedure M were included in the calibration of procedure S
- Other [Free text entry]



6 Part 3: Metrological traceability for reference material certification

[The following introduction to this Part was provided]

Reference materials are often certified by taking a suitable average of results reported by multiple different methods in one laboratory and/or in multiple laboratories. The next few questions explore perceptions of metrological traceability for results determined as an average over multiple methods, laboratories, or both. The particular scenarios included are:

- 3.1 An average of results from a small number of reference measurement procedures, each implemented by a single laboratory
- 3.2 An average of results from multiple measurement methods, implemented across many accredited routine measurement laboratories
- 3.3 An average of results for an operationally defined measurand, determined by multiple laboratories using the defining measurement procedure
- 3.4 An average of results for a measurand that is not operationally defined, determined by multiple laboratories who are all using the same measurement procedure

If you do not feel able to comment on these scenarios, you can skip this section by checking the appropriate box below

- Yes, I'd like to comment on traceability for these reference material scenarios
- No, I'd prefer to skip this section

232 respondents elected to answer the reference material questions.

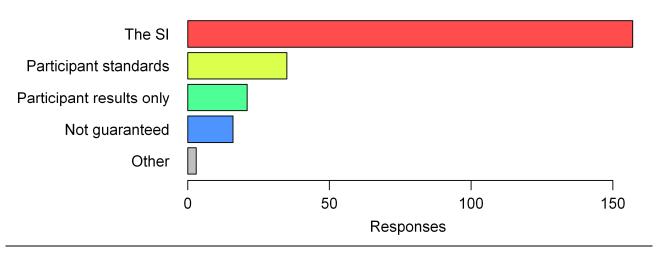
6.1Scenario 6: Reference material certification – reference measurements

A reference material's certified value is assigned as an average of results from a small number of reference measurement procedures, each implemented by a single laboratory. Each result is determined by suitably validated, calibrated procedures, with calibration traceable to the SI. Each laboratory reports a measurement uncertainty. The results all agree within their claimed uncertainties.

6.1.1 Scenario 6 Q1: Traceability of the assigned value

Which statement below best describes your view of the metrological traceability of the assigned value?

- The certified value is traceable to the SI
- The certified value is traceable only to the values provided by the different laboratories
- The certified value is traceable to the measurement standards used by the participants
- No guarantee of metrological traceability can be given
- Other [Free text entry]



6.1.2 Scenario 6 Q2: Uncertainty of the assigned value

Which statements below best describe your views of the uncertainty of the assigned value? (considering only the uncertainty arising from the inter-laboratory exercise). For this question, you can tick multiple responses.

Check all that apply.

- The uncertainty may be smaller than all of the individual uncertainties reported by the participants
- The uncertainty can not be smaller than the smallest uncertainty reported by participants
- The uncertainty is not related to the reported uncertainties
- The uncertainty must be based _only_ on the reported uncertainties
- The uncertainty can consider additional contributions, such as the dispersion of reported results
- Other [Free text entry]

MU CAN be smaller than min. u[i] MU can NOT be smaller than min. u[i] MU based ONLY on participant uncertainty info MU not related to reported uncertainties MU CAN consider dispersion of values Other 0 50 100 150 Responses

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6.2Scenario 7: Reference material certification by multiple routine measurements

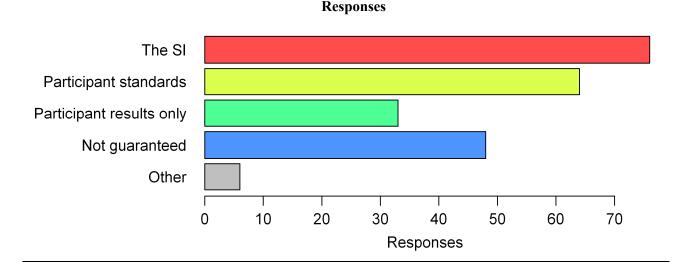
A reference material's certified value is assigned as an average (possibly a robust or outlier-rejected average), from many routine measurement laboratories, each reporting a single measurement result. All of the laboratories are working in accordance with ISO 17025 or an equivalent standard that assures adequate metrological traceability for routine measurements.

Each laboratory reports a measurement uncertainty. The results do not necessarily agree within their claimed uncertainties.

6.2.1 Scenario 7 Q1: Traceability of the assigned value

Which statement below best describes your view of the metrological traceability of the assigned value?

- The certified value is traceable to the SI
- The certified value is traceable only to the values provided by the different laboratories
- The certified value is traceable to the measurement standards used by the participants
- No guarantee of metrological traceability can be given
- Other [Free text entry]

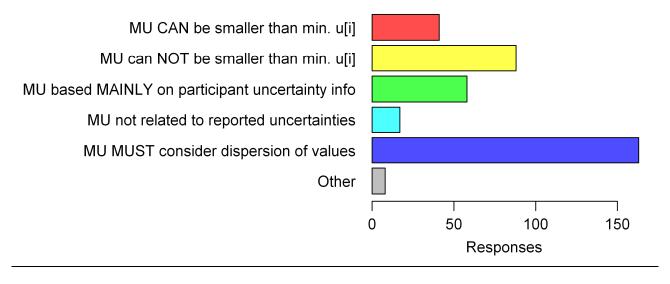


6.2.2 Scenario 7 Q2: Uncertainty of the assigned value

Which statements below best describes your view of the uncertainty of the assigned value? (considering only the uncertainty arising from the inter-laboratory exercise)

Check all that apply.

- The uncertainty may be smaller than any of the individual uncertainties reported by the participants
- The uncertainty can not be smaller than the smallest uncertainty reported by participants
- The uncertainty is not related to the reported uncertainties
- The uncertainty for the certified value should be based mainly on the uncertainty information reported by the participants
- The uncertainty must consider the dispersion of reported results
- Other [Free text entry]



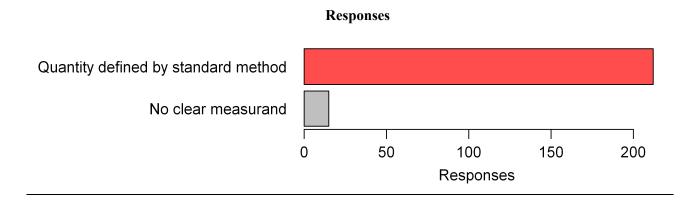
6.3Scenario 8: RM certification for an operationally defined measurand

A reference material is certified for an operationally defined measurand (like the crude fibre discussed previously), by an interlaboratory study among routine measurement laboratories who all use the correct standard method for the measurand. All of the laboratories are working in accordance with ISO 17025 or an equivalent standard that assures adequate metrological traceability for routine measurements.

6.3.1 Scenario 8 Q1: The measurand

Which statement below best describes your view of measurand that can be claimed on the RM certificate?

- The measurand is the operationally defined quantity defined by the standard method used
- No clear statement can be made about the measurand
- Other [Free text entry]

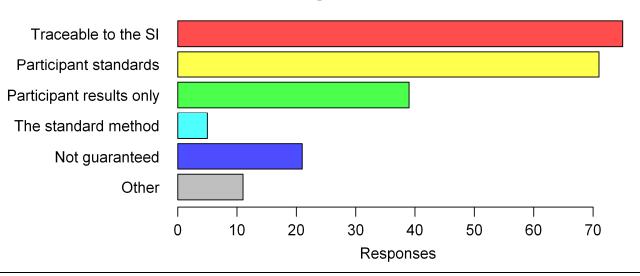


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6.3.2 Scenario 8 Q2: Traceability of the assigned value

Which statement below best describes your view of the metrological traceability of the assigned value?

- The certified value is traceable to the SI
- The certified value is traceable only to the values provided by the different laboratories
- The certified value is traceable to the measurement standards used by the participants
- No guarantee of metrological traceability can be given
- Other [Free text entry]

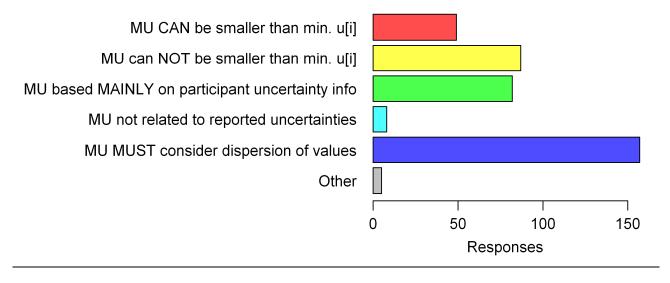


6.3.3 Scenario 8 Q3: Uncertainty of the assigned value

Which statements below best describes your view of the uncertainty of the assigned value? (considering only the uncertainty arising from the inter-laboratory exercise)

Check all that apply.

- The uncertainty may be smaller than any of the individual uncertainties reported by the participants
- The uncertainty can not be smaller than the smallest uncertainty reported by participants
- The uncertainty is not related to the reported uncertainties
- The uncertainty for the certified value should be based mainly on the uncertainty information reported by the participants
- The uncertainty must consider the dispersion of reported results
- Other [Free text entry]



6.4Scenario 9: RM certification using a single measurement procedure

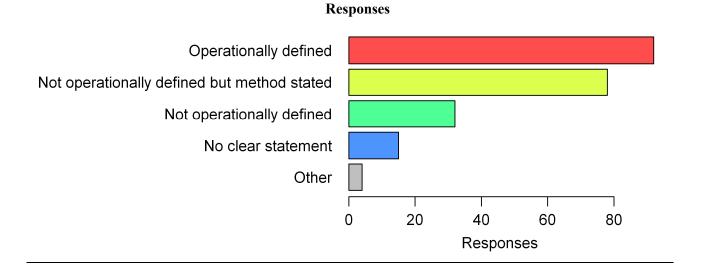
A reference material is certified, by inter-laboratory study, for a measurand (such as concentration of an element in drinking water) for which many, very different, measurement procedures could reasonably be used. The participants are allowed to choose their preferred procedure; because all work in a similar sector, all of the laboratories by chance choose to use the same standardised procedure.

All of the laboratories are working in accordance with ISO 17025 or an equivalent standard that assures adequate metrological traceability for routine measurements.

6.4.1 Scenario 9 Q1: The measurand

Which statement below best describes your view of measurand that can be claimed on the RM certificate?

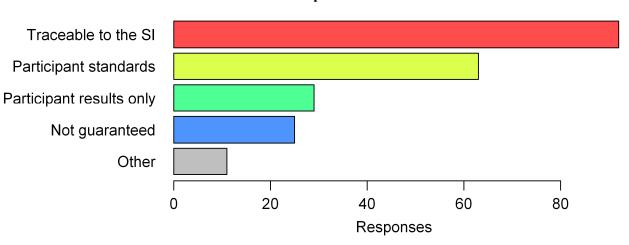
- The measurand is not operationally defined and the certificate does not need to state the procedure used
- The measurand is not operationally defined but the certificate should state the procedure used
- The measurand is operationally defined by the standard procedure used.
- No clear statement can be made about the measurand
- Other [Free text entry]



6.4.2 Scenario 9 Q2: Traceability of the assigned value

Which statement below best describes your view of the metrological traceability of the assigned value?

- The certified value is traceable to the SI
- The certified value is traceable only to the values provided by the different laboratories
- The certified value is traceable to the measurement standards used by the participants
- No guarantee of metrological traceability can be given
- Other [Free text entry]

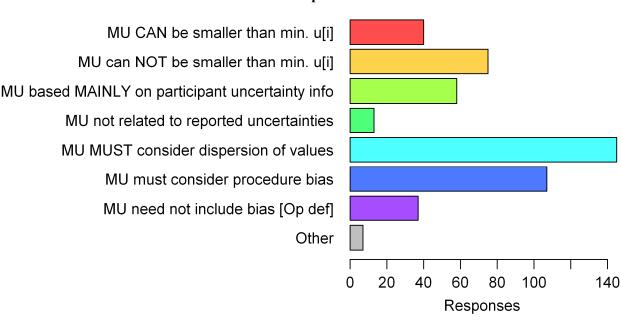


6.4.3 Scenario 9 Q3: Uncertainty of the assigned value

Which statements below best describes your view of the uncertainty of the assigned value? (considering only the uncertainty arising from the inter-laboratory exercise)

Check all that apply.

- The uncertainty may be smaller than any of the individual uncertainties reported by the participants
- The uncertainty can not be smaller than the smallest uncertainty reported by participants
- The uncertainty is not related to the reported uncertainties
- The uncertainty for the certified value should be based mainly on the uncertainty information reported by the participants
- The uncertainty must consider the dispersion of reported results
- The uncertainty must consider any possible bias arising from the particular measurement procedure used
- If the measurand is claimed to be operationally defined by the method in use, procedure bias does not need to be considered in the uncertainty on the certificate
- Other [Free text entry]



7 Effect of Respondent classification

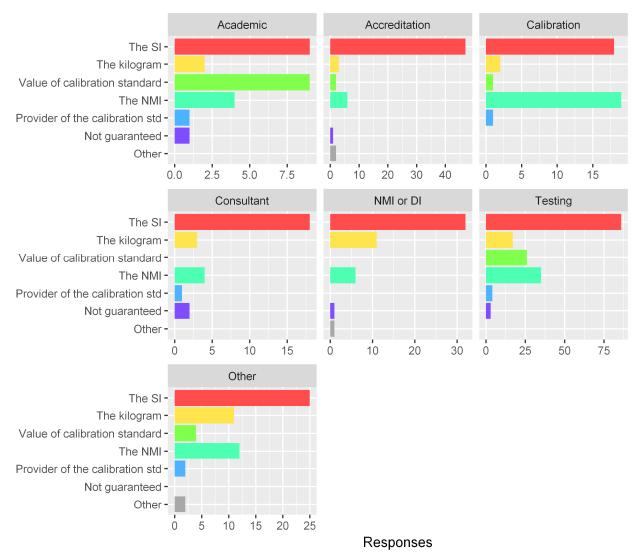
All single-answer responses were checked for association with respondent classification groups using χ -squared tests, with *p*-values corrected for multiple testing using Holm's correction [2]. None showed any significant association with field of work. Responses for five questions showed significant association (*p*<0.05 after correction) with organisation type; these were:

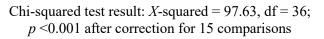
- Scenario 1 Q1: Metrological traceability (Which statement below best represents your view of the metrological traceability of the result (measured mass)?)
- Scenario 2 Q1: Traceability (Which statement below best represents your view of the metrological traceability of the measurement result?)
- Scenario 3 Q1: Traceability (Which statement below best represents your view of the metrological traceability of the result?)
- Scenario 4 Q1: Metrological traceability (Which statement below best represents your view of the metrological traceability of the crude fibre result?)
- Scenario 5 Q2: Metrological traceability (Which statement below best represents your view of the metrological traceability of the results from the secondary procedure?)

All five relate to the answers for metrological traceability. Cross-tabulation results are plotted in the following figures. Note that smaller groups of respondents [fewer than 20 respondents] were merged to form a larger "other" class, so that not all possible groups are represented separately.

7.1.1 Scenario 1 - Single calibration standard

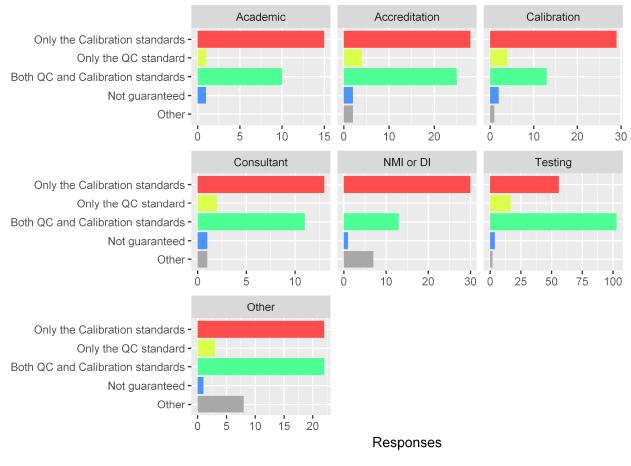
Q1 Which statement below best represents your view of the metrological traceability of the result (measured mass)?





7.1.2 Scenario 2 - Use of a certified check sample for quality control

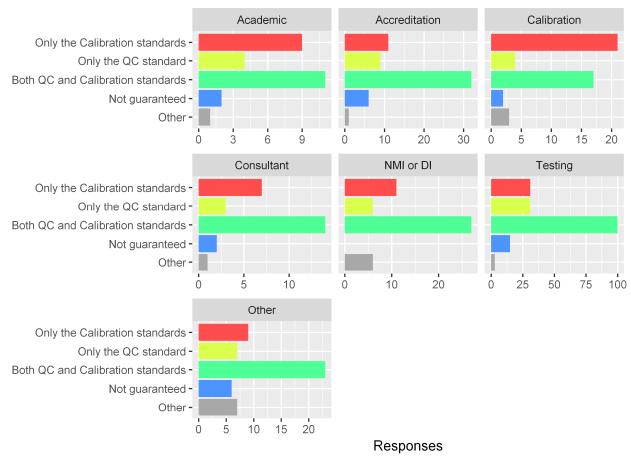
Q1: Which statement below best represents your view of the metrological traceability of the measurement result?)



Chi-squared test result: *X*-squared = 63.25, df = 24; p < 0.001 after correction for 15 comparisons

7.1.3 Scenario 3 - Correction based on observed value for a certified check sample

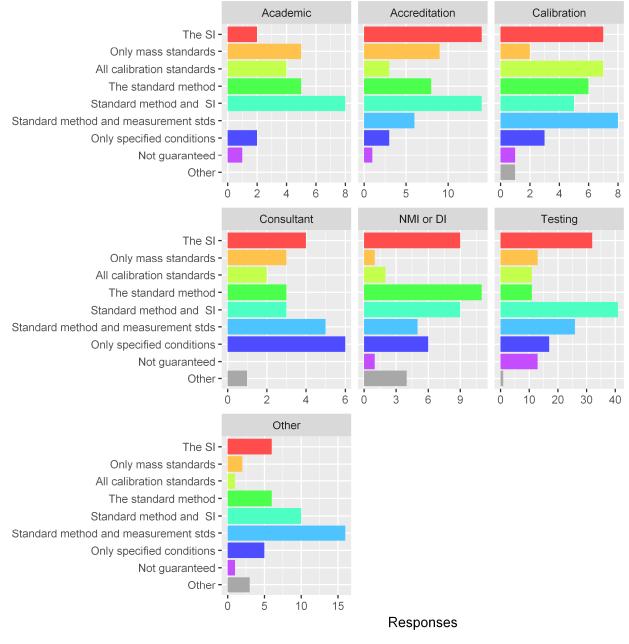
Q1: Which statement below best represents your view of the metrological traceability of the result?

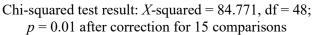


Chi-squared test result: X-squared = 46.106, df = 24; p = 0.047 after correction for 15 comparisons

7.1.4 Scenario 4 - An 'operationally defined' measurement

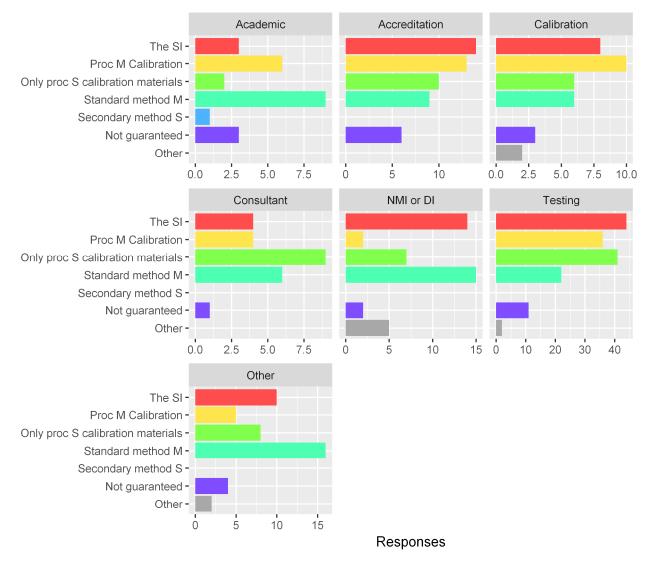
Q1: Which statement below best represents your view of the metrological traceability of the crude fibre result?

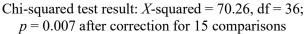




7.1.5 Scenario 5 - An 'operationally defined' measurand determined using a secondary procedure

Q2: Which statement below best represents your view of the metrological traceability of the results from the secondary procedure?





8 Principal findings

The main findings fall into four broad areas:

Understanding of metrological traceability

- All respondent groups offered a range of responses; there was little consensus on the 'best' description except in simple cases.
- Traceability "to the NMI" is still a common but not dominant concept
- Traceability to local calibration standards is often not interpreted as implying higher traceability
- The role of QC checks in establishing traceability is not clear

Measurement uncertainty and metrological traceability

- The great majority of respondents correctly expect standards to contribute to uncertainty
- Many labs expect QC material uncertainties and/or QC limits to contribute to uncertainty
- Doubts or misconceptions about metrological traceability may be impeding understanding of contributors to uncertainty or *vice versa*.

Metrological traceability for RMs certified by interlaboratory certification

- Traceability chains for 'consensus' values from interlaboratory certification are not clear to respondents;
- Accreditation body respondents appeared significantly more likely to identify results as SI traceable when participants are accredited;
- Operationally defined measurands and methods showed by far the least consensus.

Effect of organisation type and field of measurement

- No significant association between response and field of measurement was found, indicating that (at least for this respondent set) measurement scientists from all the fields of measurement represented here share a similar range of perceptions.
- Organisation type was found to materially affect responses relating to statements regarding metrological traceability. Examples of differences included:
 - Calibration laboratories were particularly likely to describe metrological traceability in terms of traceability "to an institute" rather than to the SI;
 - In the simplest scenario, a higher proportion of academic respondents described metrological traceability in terms of traceability "to the value of the calibration standard" than other organisation types;
 - Routine measurement laboratories were more likely than other organisation types to describe metrological traceability as arising from both calibration and quality control standards when both were in use.

Bibliography

References cited in this document are listed here. For a summary of additional relevant references on metrological traceability, please refer to the Eurachem *Reading List* placed under *Publications* at the Eurachem website, <u>www.eurachem.org</u>.

- 1. S L R Ellison and A Williams (Eds) Eurachem/CITAC Guide: Metrological Traceability in Analytical measurement (2nd ed. 2019). ISBN: 978-0-948926-34-1. Available from <u>www.eurachem.org</u>.
- 2. Holm, S. (1979). A simple sequentially rejective multiple test procedure. *Scandinavian Journal of Statistics*, **6**, 65-70. <u>http://www.jstor.org/stable/4615733</u>

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