Minutes CCQM OAWG Meeting

Date:	**7th and 9th April 2025**
Venue:	**Bureau International des Poids et Mesures (BIPM), Petit Pavillon, Grande Salle (Hybrid Room)**
Presentations:	https://www.bipm.org/en/committees/cc/ccqm/wg/ccqm-oawg/2025-04-07

The presentations are available on the BIPM CCQM-OAWG website and should be accessible to all the OAWG members (as designated by their institutes) after logging in.

Action items are summarised at the end of this document.

Day 1 (Monday, 7th April 2025)

Welcome - Maria Fernandes-Whaley, NMISA

Logistics for the meeting and VC connection – Ralf Josephs, BIPM

Introductions around the room – All and online in chat.

Day 1, Summary

The Organic Analysis Working Group held a meeting to discuss their progress, future plans, ongoing studies, including updates to the strategy document, comparisons, and challenges faced in the comparison studies. The group reviewed various technical aspects of their studies, such as criteria for selecting analytes, harmonization activities, and statistical approaches for calculating reference values. Additionally, they addressed organizational matters, including the group's 30th anniversary, strategic planning, and the importance of institutional support for members.

1. Working group, Strategy 2030+ document and case studies updates – Maria Fernandes-Whaley, NMISA

Summary

The presentation included updates on the group's strategy document, planned comparisons, and the status of current comparisons. The group discussed the challenges they faced in sourcing materials for their studies and the importance of engaging with stakeholders. The group also discussed the need for more Track C and Track D studies to advance measurement science and knowledge transfer. The presentation ended with a discussion on the number of key comparisons that were not delivered on time and the impact of the COVID-19 pandemic on the scheduled comparisons.

MFW provided an overview of the OAWG activities and progress, and what is to come. The status of the recent comparison studies was shown and detailed feedback from the task group sector leads will be received in the course of the meeting. For newcomers, you can read through the OAWG practices and guidelines document, where all the definitions and types of studies are explained and the processes that we follow in the working group. As a reminder, Track A studies are compulsory, and

institutes need to participate in them to underpin CMCs. Track A studies that have been published (3) to the KCDB were highlighted, as well as the studies with draft reports in progress (2). The status of the comparisons for the various sectors were shown. In summary, there were 5 reports published to the KCDB (3x Track A and 2x Track C), there are 2 Draft reports, 1 comparison is underway and 2 comparisons start in 2025. In terms of the overall strategy, the group is behind schedule, for various reasons, for some of the planned comparisons. For the updated strategy document, this picture will change slightly to reflect the reality. Brief updates were provided on the Pure Organics & Solution Track A comparisons, as well as proposals for the Matrix Material Track A key comparisons and the Clinical Sector activities. The Clinical Sector will be looking to include some expert labs in their comparisons and will need consent for these labs to participate. For the Food sector, an update will be provided on the metronidazole study. Juris Meija (NRC) will be assisting with the KCRVs and the implications of dark uncertainty for this study. On the RMO supplementary comparisons, the Food sector relies on RMOs to supplement comparisons that cannot be conducted at CCQM level. The Food sector have had several studies (EURAMET.QM-S15, AFRIMETS.QM-S1, APMP-QM-S21) with RMOs. Feedback will be provided by Kate Rimmer (NIST) on the food measurement task group and from Pui Sze Cheow on consolidating information from the surveys in the Food sector task group. For the Environmental sector, the PAHs in sediment comparison is in progress. The group have had several online discussions to look at the methodologies applied and to try and explain some anomalies. For the Environment sector, Alix Rodowa will provide updates for this sector. Again, we rely heavily on RMOs in this sector to supplement comparisons that are not possible at CCQM level.

For the CCQM strategy document updates, sector task groups were appointed to focus on the different areas. There will be sector focus groups for Food, Clinical, Environment and Improved Stakeholder engagement. Underpinning all of these are the core capabilities for Purity, covered by BIPM. A lot of the horizon scanning and understanding stakeholders' needs will be managed by these groups. In the Food and Environment sectors, don't have this engagement with stakeholders, so will meet more frequently to better anticipate stakeholders needs. Another strategic focus of the group is progressing measurement science to underpin organic analysis. This includes the qNMR workshop (scheduled for 9th April) and UoM dark uncertainties. The plan is that over the next 5 to 10-year period, we will continue to ensure that we keep abreast of all these updates and make sure we remain relevant to our stakeholders. Knowledge transfer is a new addition to the strategy document, and there we will cover matrix reference measurements, BIPM CBKT, RMO training and Track D studies. Additional volunteers to the different task groups members were shown.

An overview of the updates to the planned comparisons for 2030+ were shown. A number of polls were held to gauge interests and needs. Agreed, in principle, that will have 1 to 2 comparisons per year for Track A, Model 2, as identified and driven by the strategic need. For Track C studies, at most 1 comparison per year, and the driver would be the strategic need. Track D studies would be where the driver is research and development needs. An overview of comparisons, completed and planned, up to 2034 was show in Table 6. Some proposals and will be discussed in the course of the week to get agreement. Will also need to consider existing CMCs if intend to underpin them and maintain them, in addition to new services prompted by emerging regulation and international standards. Parallel Track C and Track D studies can be considered for emerging challenges, advancing measurement science and knowledge transfer.

Details for the 150th Anniversary of the signing of the Metre convention on the 20th – 22nd May 2025 were given. The OAWG qNMR workshop – with PAWG will be held on the 8th April 2025 (40 attendees in person and 60 online).

Comments and Questions

Robert Wielgosz – Less studies than originally planned in OAWG. What are we doing about this? Worth contemplating and is this fine according to the strategy? Were original numbers over ambitious? This will be discussed at the plenary meeting. Discussed example of bilirubin and need to have more than one source of the material.

Kate Rimmer – NIST will be happy to support more bilirubin standards.

Gavin O'Connor – There is a slippage of studies. They get more complex and extra work is required when engaging with RMOs. Worth it to keep group engaged with the stakeholders.

Maria Fernandes-Whaley – For clinical sector, also included toxicology. How many studies can you accommodate in a year? Trying to tick as many boxes as you can in a well-designed study, but this adds extra work, time and delays.

Mark Lewin – For comparisons that have not been delivered, there were some significant delays due to COVID, especially for the matrix comparisons.

2. CMC Update, summary reports and KCWG feedback – Mark Lewin, NMIA

Summary

In the meeting, Mark discussed the timeline for the CMCs that have been submitted for JCRB review, CMC approvals and publication of the ones that are approved to occur between now and the end of September. Mark also highlighted general issues found within the current cycle, including uncertainty conventions, inappropriate assignment of uncertainty conventions, and issues with providing certificates of analysis for CRM claims in English. Mark proposed a virtual meeting to further discuss the use of DSC as the major contributing technique for the purity assignments of CRMs and the minimum requirements for measurement techniques. Mark also discussed the proposal for re-review strategies, the re-review process of the category 13 CMCs in the next cycle, and the proposal for an automated system for CMCs screening. Mark concluded by proposing the establishment of a task group for the many aspects of CMCs.

ML provided an overview of the CMC Review Cycle XXVI and the OAWG feedback. Overall, 110 CMCs submitted for JCRB review, 13 new broad scope claims within categories 1, 3, 10 and 11 and re-review of all (91) Category 11 CMCs approved prior to 2015. The CMCs approvals and publications are expected to occur between April and the end of September 2025. Some general issues encountered were incorrect assignment of uncertainty conventions, inappropriate assignment of uncertainty convention one, not providing certificates of analysis in English for CRM claims, not providing relevant purity KCs as evidence when traceability is claimed, not providing additional evidence to support claimed MU when the KC used as evidence has the KCRV set more than an order of magnitude greater than the lower CMC value claimed. As well as not documenting changes made to the existing CMCs, CRM stated within the CMC is no longer available, traceability to another NMI not underpinned by a CMC and missing information. More specific issues in the review process was NMIJ's traceability to their own institute and the measurement techniques described for the purity assignment (DSC, GC-FID and KF). The reviewers questioned whether this was sufficient to determine purity and establish

metrological traceability. At this point, the CMCs have been withdrawn and it was proposed to have this discussion within the OAWG meeting. Due to time constraints, propose having a virtual meeting to discuss this and to come to a consensus within the working group with regards to how best to determine purity, or what the minimum requirements are for determining purity of high purity materials.

VNIIM submitted a claim for 6 phthalates in Cat 3. Reviewers viewed this claim as broad scope, without sufficient supporting evidence. Do broad scope criteria need to be reviewed? Should multiple individual claims generated from a single study be discouraged. Discussion around how this situation is handled would be helpful. How will digitalisation efforts affect claims? Do broad scope claims need to be reviewed? In the past there was an excel spreadsheet to assist with the review of broad scope claims. Are these templates still useful and should they be maintained and used?

The KCWG updates that were discussed in the las two days were provided, and covered the review process, additional service categories and uncertainty conventions. The KCWG discussed uncertainty conventions with a suggestion to no longer support uncertainty convention one. For uncertainty convention one, you have a range of uncertainties that would cover the whole measuring range that you're claiming and the uncertainty. For Convention two, you have an uncertainty that you would expect at the lower range of your measurements, and another uncertainty at the higher range of measurements. Would we support removing uncertainty convention one. Existing claims would be modified before the re-review process. Purity claims would be the most affected. The KCWG are planning a CCQM workshop to discuss acceptable evidence for CMCs other than direct key comparison performance. Please consider what types of additional evidence should be acceptable (i.e. peer reviewed publications). The KCWG requested OAWG to review old studies with the view of archiving those that have been superseded. Some groups actively review the studies that can still be used to support claims and archive those that are considered too old.

Looked at setting up a Task Group to produce e-learning modules designed to provide guidance and educate CMC reviewers on how to perform a review. While some working groups have good CMC guidance documents, OAWG does not have such guidance. Could be first steps to developing e-learning modules. Could provide a checklist for writers and for reviewers. Summary reports were proposed to help with the review process. This has happened so reviewers do not need to go through the full comparison report to get the required information. Summary reports have been circulated for feedback. Please can institutes provide some feedback. The greying out procedure for CMCs should also be in the guidance document in the future. Aswell as similar procedures and guidance for underperformance in a key comparison, and for failure to submit CMCs for re-review. The KCWG have discussed the re-review process and the expected new evidence. Expected new evidence in Cycle XXVII was shown, along with studies for future review cycles, studies still in proposal and multiple purity and solution studies not expected to be completed by 2026 and beyond.

A graph was shown with all existing Organic CMCs by category and approval date. The observed jumps are due to re-reviews. There is a proposal for the re-review strategy to re-review Category 13 CMCs in the following cycle for CMCs approved in or before 2019. The OAWG re-review strategy proposal was shown with suggested items for review.

Comments and Questions

Maria - Virtual meeting for CMC discussions would be welcome.
Itoh Nobuyasu - NMIJ would like to discuss thermal analysis (DSC) further as this technique is important for their purity analysis. Would like to discuss further.
Robert – Have an IUPAC paper on what we can use for purity analysis and this includes thermal methods. Should be consistent with what is in the paper.
Bruno – Definitely have to review broad scope claims and their criteria. Numbers show that it is not working if there are so many claims. Not working as, it is too stringent. Needs to be improved and need to consider the digitalisation component. Broad scope claims are not machine readable. For the CMC guidance document, there is some guidance in the OAWG documents.
Mark – Some of the guidance is spread out, so difficult to see all of it. Broad scope isn't working but not sure how effective it is in stopping individual claims.
Gavin – The disparity of information across historical reports makes reviewing difficult and takes a long time. Summary reports must make the reviewing process easier.
Mark – Please look at example summary reports and provide feedback.
Maria – In old template used for CMCs had guidance on uncertainty convention one and two.
Robert – Historically, convention one and two were required as NMIs could not agree. Now have a convention that not everyone understands. Does it help us? We are an outlier in the community and using a convention that no one else does. Has the time come to make it simpler? Historically, was for a lack of consensus, but do we still have that?
Mark – For members that don't submit CMCs that often and don't understand the convention, it is not intuitive. Propose adopting uncertainty convention two.

Action: 1 Please can institutes review the example summary reports and provide feedback to Mark Lewin.

Action: 2 Task group to be set-up for CMCs and suggested to have a representative from RMOs. Led by Mark Lewin.

Purity Assignment – Core Capabilities

3. Criteria for choice of analytes for CCQM OAWG purity comparisons - Ralf Josephs, BIPM Plans for next Track A purity studies (strategy plan update 2030+)

Summary

The group discussed criteria for selecting analytes for organic purity comparisons. They agree to continue with purity studies even when authentic impurity standards are not available, as this reflects real-world challenges. The group reviewed the status of upcoming Track A studies, including a polar pesticide study focusing on glyphosate and related compounds. They also discussed plans for future nonpolar solid organic purity studies, with a call for candidate suggestions from task groups. The importance of timely input for study planning is emphasized, with a two-year lead time typically required.

RJ presented on the criteria for choice of analytes for CCQM OAWG purity comparisons following a customer satisfaction survey. Suggestions for improvement were that key comparisons studies on samples for which authentic related structure impurities are not readily available should be avoided.

This is beyond the scope of the BIPM quality system. The criteria applied by the CCQM OAWG for choice of analyte were provided, and the floor was opened to discuss this further.

Comments and Questions
Bruno – This is reality and what we are dealing with on a day-to-day basis. We are not provided with references of impurities with samples.
Mark – What is required for a purity assessment?
Robert – If only perform comparisons where everything is lined up, it is not that challenging. In the published IUPAC paper, gave examples and guidance where didn't know structures of impurities.
Ralf – In IUPAC paper, describe uncharacterised materials, response factors and structurally related impurities where you don't have authentic standards. VIM 3rd edition also gives guidance on this. Comparisons need to mimic reality and if we were to follow this (only perform studies when impurities are available) materials would be limited.
Maria – Working group supports proposal to continue and proceed with guidance of IUPAC document.

RJ continued with the presentation and showed a table with the core key comparisons (Track A) for pure materials and calibration solutions planned up to 2034. Will need to find analytes and define the goals for these comparisons. For polar pesticides in solution, decided on class of phosphonic acids and metabolites. BIPM have a current joint technical project with NIM for pesticides. There is overlap of glyphosates as analytes for CCQM-K78.a.2027 and an APMP study. The proposed timetable for polar pesticides in solution was shown. For non-polar solid organic, the candidate suggestion for 2028 is quinidine. Will need a solid candidate soon for planning as there is a pre-run phase of approximately two years.

Comments and Questions
Gavin O'Connor – Candidate doesn't have to be in the clinical space. Will look at gaps in clinical space on Wednesday.
Gavin O'Connor – Can provide a list but need to go back to the community to check what would make a difference as a pure primary calibrator.
Ralf – BIPM have sourced the phosphonic acids and metabolites.

Action: 3 Sector leads, please come back with suggestions for purity comparison considerations.

4. CCQM-K148.c (large MW pure organic) - Digitoxin; Ralf Josephs, BIPM

Summary
The group discussed the upcoming Digitoxin study for purity organic analysis. Ralf presented the background, schedule, and current status of the study. The material has been repurified after contamination issues, and a draft protocol has been prepared. The group agrees to extend the deadline for the results submission to March 2026.

RJ provided an update on the Digitoxin study. This is a cardio glycoside that fits in the upper sector of the model with a large MW pure organic. The compound classification, application, analytical interest and CRM availability were shown. The study schedule was provided. During initial work on the material, a contamination was discovered that had to be removed to proceed with the material. This

delayed the timelines slightly. The homogeneity and stability study results were shown. The study material will include 4 x 50 mg samples to participants. The distribution of samples will be in June 2025, with results submission in October 2025, initial discussion and Draft A report (2025/2026).

Comments and Questions

NRC and LGC asked for an extension for the submission from October 2025 to Feb 2026.
There was a show of hands for who will participate – approximately 11 participants.
Robert - Pointed out that CMCs based on historical studies in this space will be replaced and that NMIs need to participate in this to maintain their CMCs.
Gavin – What happens to the material after the study?
Ralf – This will be a CRM produced by UME. BIPM always choose a partner NMI who will then produce a CRM.
Maria – Would all the 12 NMIs agree to moving the reporting deadline to March 2026. No objections.

Action: 4 BIPM to update digitoxin study protocol with new results submission deadline of March 2026.

Action: 5 Mark Lewin to analyse database to identify NMIs with CMCs that have molecular mass above 500 and need to participate in digitoxin study.

5. PAWG Cyclosporine comparison – Gustavo Martos, BIPM

GM provided an update on a potential purity comparison on Cyclosporine proposed for the PAWG, but not too far off OAWG activities. A background on Cyclosporine A and its uses were shown. It is important for therapeutic monitoring and dose adjustment to ensure that patients are in the correct therapeutic range. There is plenty of interest across the clinical field and an overview of the clinical relevance was shown. The structure was provided, it is a cyclic peptide with 11 amino acids, neutral, non-polar, several methylated peptide bonds. Produced by fermentation. The challenges for PAWG are that it is cyclic and methylated. There are two conformations depending on the solvent, cis which is the closed form and trans which is the open form. Analytically it is challenging molecule, especially by NMR where you can see different conformers depending on the solvent used. BIPM are discussing sourcing the material with LGC. Call for participation is likely to be next year. Participation will be through PAWG. Cannot have an NMI participating through OAWG and PAWG.

6. CCQM-K78.b 2025 Pesticides in organic solvent standard solution comparison proposal – Ralf Josephs, BIPM

Summary

Ralf provided an update on the CCQM K78.b study for pesticides in organic solvent. The study will include three pesticides: Carbofuran, Atrazine and Dimethoate. New source materials were obtained after the initial materials were found to be unsatisfactory. Analytical methods have been developed and stability studies conducted. The study is planned as a Track C comparison, with participants receiving multiple ampoules to report a combined value. Six institutes have indicated their intent to participate so far. The timeline indicates value assignment and solution preparation by the end of the year, with CCQM approval sought this year for the study to proceed.

RJ provided a background to this study and the issues encountered with the CCQM-K78.b study. Pesticides were selected for their relevance to food safety and the environment, as well as being regulated (controlled/banned) worldwide. BIPM have an ongoing joint technical project with NIM for the study material. There were several proposals for different classes of pesticides and selected Atrazine, Carbofuran and Dimethoate. The materials have been sourced and looked at by qNMR. A feasibility study was conducted in 2024/2025, and the original material was not satisfactory for use and new materials were sourced. BIPM have evaluated analyte stability in single and mixed solutions (multicomponent mixture). The proposed protocol was shown for this as a Track C study. Several ampoules will be provided per participant, no calibrators will be supplied. The proposed timeline was shown and will need to get the study approved by CCQM.

Comments and Questions

Maria – With a show of hands could institutes indicate their interest to participate in this follow up study. Approximately 6 institutes showed their interest. No objections about the proposed analyte and timelines. Can proceed as proposed.

7. Update on Track C CCQM-K154 mycotoxin calibration solution key comparison series – Ralf Josephs, BIPM

Summary

Ralf discussed the formation of 2-hydroxy OTA during the filling process of the ampoules, which was identified as a cause of delay in shipping the samples. The concentration range for the calibration solutions was proposed to be increased from 2 – 50 mg/kg to 2 - 80 mg/kg. The team agreed to the proposed timeframes for the study.

RJ provided an overview of the mycotoxin calibration solutions. This is the last one in the CBKT series and now going into the demonstration of competence phase. This will be a Track C model two comparison, with materials prepared by NMIs and value assigned, and then sent to the coordinator for value assignment. An overview was provided of the study materials and guidelines, and the participant requirements for reporting. The schedule was provided for the initial discussion, stock solution distribution, draft protocol and call for participation.

Gravimetric preparation of stock solutions and calibrant solutions were performed. From the homogeneity studies of the calibrant solutions, there are no trends in filling and analysis sequence. The stability studies show no obvious trends. There appears to be some issues for samples stored at 40°C over time but in principle, they look stable. Observed more variance for the larger numbered vials and have discarded the last 60 ampoules to be safe. Learnt from previous experience with this. RJ provided information on the 2'R-OTA enantiomer discovered during the homogeneity and stability studies of the stock solutions. This doesn't form under the normal storage conditions and is maybe formed through the ampoule sealing process (butane-propane-oxygen flame sealed at about 1900°C). Will correct for the bias of this contamination and continue with this material. It is important for participants to be aware of this as you can create more 2'R-OTA if not careful. Currently, there are very low-level amounts present. The stability of 2R'-OTA has been checked, and overall BIPM are happy to continue with this material. The HFTLS statement was shown. The updated schedule was shown and

need to agree on the draft protocol for the change in concentration. The call for participation will be in May 2025 and the final date to register for participation will be 1st Dec 2025. Will propose new dates for the study as shifted the digitoxin study and this will then coincide with the digoxin study which may be critical for other NMIs. OAWG need to decide if they are comfortable with proposed mass weight range change from 2 – 50 mg/kg to 2 – 80 mg/kg. There are 3 NMIs producing their own calibrators at the higher concentration and would like this to be covered as well.

Comments and Questions

Maria – Is everyone comfortable with the change in the upper limit from 50mg/kg to 80 mg/kg. There were no objections.

Maria - Is everyone in agreement with the changed timelines for this study? Would you prefer the later deadline – a few nods. No one supported the earlier deadline. There were no objections to the revised schedule and deadlines.

Ralf - NMIs were advised to take care with ampouling and the temperature increases. The correction will be to the assigned purity and the uncertainty.

Action: 6 BIPM to update the mycotoxins calibration protocol with the new deadline of March 2026 and expanded concentration range.

8. IAWG-EAWG Comparison of water mass fraction measurements in crystallohydrates - Maria Krasheninina, VNIIM

Maria provided an overview of VNIIM's reference measurements. The laboratory produces certified reference materials for food materials with certified values for moisture. An overview of the materials for the proposed comparison study were given and include $CaC_2O_4.H_2O$, $Na_2MoO_4.2H_2O$ and $C_{12}H_{22}O_{11}.H_2O$. Participants can use different measurement techniques to determine the water content. This comparison is supported by previous comparisons that have been published. The results of these were shown and discussed. All the materials have been certified in Russia using different techniques. The time schedule was provided but has not been approved yet.

Comments and Questions

Cailean – Requested to see timelines again. These will overlap with other studies if the comparison is from April to October 2025. The dates have not been finalised and could change.

CCQM President address to the OAWG – Sang-Ryoul Park, KRISS

Sang-Ryoul Park discussed the growth and development of the organization over the past 30 years, highlighting the expansion of working groups and the organization's role in addressing global challenges. SRP emphasized the importance of strategic planning and the need for institutional support for members.

OAWG Environmental Sector Focus

9. Draft A results for Track A environmental key comparison: PAHs in sediment – Tang Hua, NIM China (online)

Summary

The group discussed the statistical analysis of the study results, focusing on the comparison between the different methods. The discussion then moved to the issue of metrological traceability for one laboratory's results. The group considered options for handling this, including allowing the lab to establish traceability post-submission or excluding their results from the KCRV calculation while still keeping them in the study. They aim to finalize Draft B by October 2025, pending resolution of the traceability issues and further discussions on dark uncertainty.

TH presented the progress on the PAHs in sediment comparison. The background was provided for the choice of measurands, sample description and HFTLS statement. The timelines were shown, and they are now ready to discuss the Draft A report in the OAWG. The conclusions for the meeting in Feb 2025 were provided and included labs that withdrew their results. There are several issues with this comparison that require confirmation and discussions. These include the statistical models for the KCRV calculations and some traceability issues. A summary of the results was provided along with the timelines. The KCRV estimation was outlined using the NIST consensus calculator and an overview of the different statistical models for the KCRV were shown. The provisional KCRV values were shown with their associated uncertainties.

Issues for discussion:

- Considering dark uncertainty datasets and the reported uncertainties, was it appropriate to choose Hierarchical Bayes for the KCRV calculation?
- Other statistical issues in the calculation of the KCRV and DoEs
- Traceability of the purity for calibration solutions for Lab 4 is an issue

Traceability discussion:

Joachim – With regards to the traceability of the calibrants. This is a general concern, the aim of the study is to get an accurate KCRV as far as possible, but if you exclude results for formal reasons (i.e. traceability) this is difficult to achieve when excluding results.

Maria – Traceability is a requirement for a calibrant. Refer to the IUPAC document for guidance. There is an expectation of the minimum amount that needs to be done to meet the metrological requirements for calibrants.

Bruno – Not just about applying the right methods but also demonstrating that you have competence in those methods. Demonstration of competence is a CIPM requirement. To do this, will need to participate in comparisons.

Mark – On the demonstration of competence, we have the ability for NMIs to participate in matrix studies as part of demonstration of purity assignment. They have to also participate in the purity comparisons as well.

BJ – Purity by mass balance requires at least two orthogonal techniques. Minimum requirement for mass balance approach is for the purity value to be traceable.

Maria – As an NMI, need to ensure metrological traceability of calibrants.

Joachim – If have multiple analytes, perhaps you can demonstrate your competency for one or two analytes, and use certificates provided for others. It is a challenge for labs to do method development, validation and assignment of all calibrants as well.

Mark – In terms of comparing results, we need to have the same reference point (SI where possible), so comparing to the same unit and not in a situation where you are comparing apples and oranges.
Bruno – With regards to Joachim's comment, it makes sense not to do purity assessments every time for every study. However, will still need to demonstrate capability if assigning purity to a calibrant.
Sang-Ryoul Park – Can have a chain of traceability to another NMI. If you want to claim your own traceability, then you need to do more. This should be part of planning the study and not part of the KCRV discussion, so it is clear from the start.
Maria – Do spend some time upfront on sourcing CRMs.
Gavin – Pure primary calibrators are very important. Had issues in the past with matrix materials.
Ralf – Could provide a common calibrator for the study. Still need to have traceability for that calibrant. Need to do due diligence on calibrants if buying them in.

KCRV discussion:
Mark – In our guidance document there's a description of the choice between ESL and Hierarchical Bayes, and it's not so cut and dry as you have less than or more than 10 participants. There's quite a discussion within that document regarding the choice between the two.
Juris Meija (NRC)– What you just heard is absolutely right, because when we made this flow chart, maybe we didn't explain it well, but the intent was that if n is less than 10, you really need to go, Bayesian, if it's above 10, you can do whatever you want. That was really the idea, and I think this is exactly how it was captured here.
Stephen Ellison (LGC) – The difference is small and get very similar numbers. Recommends doing a sensitivity analysis first before committing to it.
Juris – (in chat) offered to help with the sensitivity analysis and it is unlikely to change the results.
Mark – The issue with metrological traceability for the one lab needs to be concluded before can calculate the KCRV.
Maria - If only met requirements for one analyte, can only claim for that one analyte.
There were suggestions on assigning traceability by purchasing a CRM that is traceable and performing additional measurements and uncertainty contributions, so as not to lose the results from Lab 4. Lab 4 result can stay in the study and they will get a DoE. Can submit a CMC and justify their assignment of the calibrator.

Action: 7 Statistical panel (Juris Meija) to provide guidance on appropriateness of using hierarchical Bayesian method for KCRV calculations in CCQM-K184.

Action: 8 Tang Hua to update KCRV calculations based on the statistical panel's recommendations.

Action: 9 Tang Hua to distribute updated Draft B report to participants after resolving the traceability and statistical methodology issues.

Action: 10 Working group to review and approve the Draft B report for the PAHs in sediments study via email.

10. OAWG Environment Sector strategy, case study update & 2030+ KC discussion Alix Rodowa, NIST

Alix presented updates to the environmental sector strategy document and discussed the definition of the environmental sector. The group agrees that soil, water, and particulate matter should be included

in the environmental sector, while food and clinical matrices are generally considered separate. However, there is recognition of overlap between sectors, particularly for environmental contaminants found in food. The task group leads will collaborate to ensure proper coverage of cross-cutting areas. A survey was conducted to better understand what NMIs consider part of the environmental sector, with the results being soil/sediment (93%), water (91%) and particulate matter (67%).

11. Guidance on OAWG handling of dark uncertainties – OAWG Statistics panel, Juris Meija, NRC Canada (online)

Juris discussed the concept of dark uncertainties in measurement standards, explaining how they are calculated and their significance in ensuring measurement standards are consistent and comparable. He also highlighted the importance of considering dark uncertainties in the calculation of degrees of equivalence. The meeting involved a discussion on how to handle dark uncertainties in the context of CCQM key comparisons and their impact on the uncertainty of the KCRV. The participants also discussed the possibility of using dark uncertainties as a quality indicator for key comparisons and the need for more data to better understand the relationship between dark uncertainties and the performance of laboratories. The conversation ended with a discussion on how to handle dark uncertainties in the context of CMC claims, with some participants expressing concerns about the potential for overestimation of uncertainties.

Comments and Questions

Bruno – To turn Tang Hua's comment into a question. We might be losing some uncertainty components when we do our measurements and estimations, and that is reflected on the consensus. Is it fair to say that in the perfect situation (i.e first example), the dark uncertainty is low.

Juris – Yes, the only problem that I can see, and Steve might comment better on this, is that often the discussions or the problems are political, less so technical, because if there's a discrepancy between the results, it gets captured by dark uncertainty, and then gets redistributed back to the to the participants as a basically top down form of uncertainty evaluation. The problem is that it's a collective decision. The collective has to agree that that they all will pay the price, because sometimes these uncertainty components are missed only by some, not by everybody. But everybody else has to be willing to absorb them. And that's where most of the time people are complaining, especially if you are bang on with the KCRV, you would say, why do I have to pay for this?

Stephen – You are paying the price. May need to understand this better. You are not putting the dark uncertainty to penalise people, but to get a more realistic value on the uncertainty. It is there because the uncertainty needs to be bigger. What's the uncertainty of the DoE and how does this affect the CMC?

Joachim – Each key comparison is a single shot, one value and one sample. Dark uncertainty could be labs dark uncertainty.

Gavin – The studies that we've done over the years have become phenomenally more complicated.

Bruno – There are two comparisons which want to know how to deal with dark uncertainties. Should we combine dark uncertainty with lab uncertainty?

Juris – Yes, that is what the DoE calculation does.

OAWG Capacity Building and Knowledge Transfer

13. OAWG strategic plan proposed inputs – Maria Fernandes-Whaley, NMISA

Maria provided feedback on the task group for knowledge transfer (KT). The strategic aims were covered and the specific activities to be undertaken by the task group were given. The OAWG knowledge transfer survey results were shared. The proposed updates to the 2030+ strategy were listed.

RMO updates – Comparisons

14. AFRIMETS Pesticides in food update – Laura Quinn, NMISA

Current update is, thanks to NIM China and LGC South Africa, we were able to get the standard. We are working with the individual NMIs now, just to see who needs it and to address procurement. As soon as that's been sorted, we can ship the samples.

15. APMP Histamine in Fish update – Annie WF Wong, GLHK

Annie provided an update on the comparison study. A brief introduction was shown, and this has been presented in other meetings, so not covered in detail. A total of 10 DMIs and NMIs have registered for the study. The homogeneity study was presented, as well as the short-term stability study. This showed that the sample is stable at 20°C. As it is not always possible to ship materials at 20°C or below, due to customs issues, the short-term stability was also performed at 45°C to support shipment. The neat reference material and study material were shipped to participants. The HFTLS statement was covered, along with the proposed schedule. The deadline for submission of the results has been extended to 30th April 2025 (one month later than planned).

16. APMP Proposal for Comparison: pesticide residues in water - Tang Hua, NIM (online)

Tang Hua reported on the progress of the proposal for the APMP supplementary comparisons and pilot studies, focusing on pesticide in water and pesticide in calibration solutions. The proposal was put forward at the last year's OAWG meetings in November and has undergone further discussions. The final selection of components for the calibration solution is based on APMP members' interest and regional priorities. The proposal includes conducting two comparisons for polar and nonpolar pesticides in water first, followed by two comparisons for polar and nonpolar pesticides in calibration solutions. The study materials for pesticides in water include glyphosate in water and lindane in an organic solution. The proposed schedule includes starting the comparisons for pesticides in water in 2025 and for pesticides in organic solution in 2026.

The group discussed the protocol for a pesticide study involving glyphosate and lindane in water. TH explained that glyphosate samples will be provided in drinking water, ready to analyze, while lindane will be in a methanol solution requiring dilution. Concerns were raised about the dilution protocol for lindane, emphasizing the need for a clear specification of the water type and dilution process. The group debated whether to send standardized water samples to ensure consistency across participants. They also discussed the importance of detailing the water matrix composition and the potential impact of drinking water variability on the study results.

Comments and Questions

Bruno – Is your calibration solution a model two comparison? The HFTLS statement should not support preparation.

Mark – Important to have consensus agreement before study goes ahead.

Tang Hua – The matrix is drinking water and this will be sent out. Will be sending water and samples, and participants just need to mix them.

Discussion followed on different grades/types of drinking water, so either have to provide a common drinking water or specify deionised water. If use deionised water, can you claim a CMC for drinking water? Would like to decrease the impact of the water matrix. This discussion will be followed up and clarified on in Day 2.

17. APMP Proposal for Comparison: adulterants in herbal product –Pui Sze Cheow, HSA

PSC showed the proposed analytes, formula and molecular weights. A recap was provided on the proposed study material. The matrix for the comparison will be a dietary supplement powder in sector 5. The HFTLS statement was shown. The proposed schedule was shown with the invitation for participation in June 2026.

18. EURAMET Estrogens in water campaign – Béatrice Lalere, LNE

Béatrice presented on the EURAMET oestrogens in water comparison. The number of compounds has been changed from the proposed 5 to 3. An overview was provided for the molecular weight, mass fraction and traceability. The supplied materials (kit) were described as well as the required preparation of the samples. The protocol was provided and the materials were described. The schedule was shown with sample distribution in September 2025 and the deadline for submission of results in December 2025. The group discussed the preparation of water samples for a comparison study on measuring estrogens. They decided to use mineral water with added suspended particulate matter and organic compounds to create a realistic sample. The study will include two levels of water complexity, with varying amounts of organic carbon and suspended particulate matter. The samples will be distributed in mid-September 2025, with results expected in December 2025. Several institutes have already confirmed their participation, and others are still welcome to join. The protocol is finished and being verified. The team discussed the complexity of water types and how it affects their work. They considered the need for a ranking system to determine the level of complexity in water samples, which would help in deciding which comparisons to participate in. The team also discussed the need for a proposal that outlines the different types of water and their complexity levels. They agreed to finalize the proposal by Wednesday (9th April). The group also discussed the need for a system that allows them to claim more comparisons based on their experience with water analysis.

Comments and Questions

Bruno – May be overthinking the water issue and need to have defined contents.

Béatrice – Have stricter regulations about water as there are more complex analytes in different types of water.

Maria – The food sector space is divided into spaces based on nutritional tables. Similar for clinical.

Perhaps we could put thoughts together to come up with a ranking for different waters, whole water, drinking water, waste water, deionised water. Can rank the water space. Want to minimise studies that people need to participate in to make claims.
Gavin – Can map out the space to give you confidence that people that work in those areas can demonstrate their competence.

19. GULFMET Benzoic acid and sorbic acid in Ketchup – Mine Bilsel, UME (online) 9th April

Mine provided an introduction covering the background for the proposed study and the regulatory requirements. The proposed measurement range is 100 to 2,000 mg/kg of benzoic acid and sorbic acid measurands in ketchup. The presentation included details on sample preparation, homogeneity tests, short-term stability, and measurement uncertainty. The study material will be ketchup, and 4 alumina sachets will be distributed to the participants. Each will contain 200 g of the study material, and the participant is requested to determine the mass fraction (mg/kg) of benzoic acid and sorbic acid with their preferred methods. A list of the available CRMs was shown for benzoic acid and ascorbic acid. The HFTLS statement was shown and a list of the existing CMCs. The timeline for the comparison was discussed, with call for participation in July 2025 and the Draft A report in December 2026. There is an option to push back the timelines to accommodate more participants. The presentation ended with an invitation for participants to express their interest in the comparison.

Comments and Questions
Indicated interest – INMETRO, APMP (will need to read the protocol first), KEBS
Tang Lin – Mentioned that APMP members may be interested, but the timelines are tight if this is the first time people are hearing about this. Can UME's Benzoic acid be used as a CRM?
Mine – Yes, it was certified by mass balance and qNMR. Don't have a CMC yet for benzoic acid. There are other CRMs that can be used for benzoic acid and sorbic acid.
BJ – In the HFTLS statement you have it as a Key Comparison and it should be a Supplementary Study
Mark – Do we need summary reports for the RMO studies as well? If so, then he will need help with this.
Maria - Are people happy with the HFTLS statement?
Comments on the definition of plant-based food matrices. Is this a fruit? Rather define what category it falls in for the food sector.

The team discussed the use of a coulometric value for a purity comparison, specifically for the benzoic acid. UME clarified that the material is certified for both coulometric value and purity, with the assessment done through a combination of results from qNMR and coulometric titration. They also discussed the availability of certified reference materials and the need for a clear protocol regarding the use of these materials. The team agreed to double-check the category of the tomato sauce in the food triangle and to specify the category if they believe the light can shine further than the assigned category.

Day 2 (Wednesday, 9th April 2025)

Welcome – Maria Fernandes-Whaley NMISA

OAWG Clinical and Toxicology Sector Focus

20. Feedback from CCQM Nano - and microplastics task group – Marina Ricci, JRC and Andrea Giovannozzi, INRIM

Summary

The presentation focused on the ongoing activities and future directions of the task group on micro and nano plastics measurement and standards. The challenges in standardizing and harmonizing the measurement of micro and nano plastics were discussed, including the lack of clear definitions and the need for more research on the impact of organic contaminants on plastics. The results of an interlaboratory study conducted by the VAMAS Technical Working Area 45 were discussed. This aimed to quantify the accuracy of different labs and evaluate the comparability between labs. The study focused on the characterization of candidate reference materials in the microplastic range. The group discussed the development of reference materials, including the release of a new reference material and the ongoing work on aging microplastics. The possibility of launching a pilot study, depending on the interest of the NMI's in the field, was presented.

Marina provided a background and need for the task group. The task group started in 2022 and in 2024 they were granted an extension, as it was felt that there was more work to be done. The goals of the virtual stakeholder workshop were shown and the topics covered were listed. The task group members were listed, along with the input/polls from the workshop participants. The future directions were discussed, and the task group will ask for another one-year extension at the plenary meeting.

Andrea covered the activities and interlaboratory comparisons in other groups such as VAMAS, CUSP, PLasticsFatE and Polyrisk. For the testing suitability of the reference materials, a summary was provided of the different techniques used and the types of samples provided to the labs. The materials produced by BAM were covered in detail and the homogeneity was shown. A summary of the ILC was shown and included size distribution: microscopy versus laser diffraction (manuscript: Analytical Chemistry under review). The conclusions of the ILC were covered as well as lessons learnt. The ISO standards initiatives were shown: ISO/TC 147/SC 2/JWG 1. PLasticTrace – ISO 16094-2 standard: Interlaboratory validation. A summary of the materials distributed were shown and an overview of the results were provided, along with the general conclusions. There was a call for participation for the PLasticTrace ILC.

Marina covered new reference material for the analysis of microplastics particles. EURM-060 has been released as a reference material but not a certified reference material. The material is a surfactant solution, and particles are embedded in a freeze-dried salt cake (NaCl carrier) and mineral water. They are working on more materials, EURM-062: reference material of microplastics particles for Manta Net sampling consisting of PE and PP >250 µm immobilized in a NaCl carrier and surfactant for analysis in water. Also looking into exploratory research stage: aged MP PE/PET particles. Looking into artificially aging plastics and characterising the ageing. Proposed a CCQM study, pilot study on microplastics and nanoplastic characterisation. Marina shared the QR code to express interest in this pilot study.

Comments and Questions

Maria – Interest and focus appears to be in number of particles and composition. Is there any interest in tracking organic components?

Andrea – Not much in terms of regulation.

Enrica – Lack of relevant refence materials.

21. Update Track A clinical key comparison: Estradiol in serum – Marina Ricci, JRC

MR showed the mapping of the clinical space and Estradiol falls within sector 2. She provided a recap of the new candidate CRMs for 17β-Estradiol and the need to replenish the current low stock. The production of the material was finalised in 2023 and they have conducted a preliminary commutability study. The material was prepared in different ways, and they decided to use a frozen form for the samples. The production of ERM-DA576, 577, 578 were shown, as well as a summary of the homogeneity and stability studies. Sample dispatch to KC participants (12) was shown and everyone should have all the samples now. The original plan was that the study would run alongside the RELA study. Many of the labs in RELA could not accept a frozen fresh sample. Due to the logistic problems that RELA couldn't use the samples, they looked at the JCTLM database and selected some labs for a pilot study. The participation of guest labs needs to be considered carefully and requires justification for their contribution. The labs selected from the database for the pilot study were shown and link the two studies. Observations/recommendations for analysis of BCR-576:

- Ampoules are difficult to break and are made of borosilicate glass
- Cannot dispatch more than 7 ampoules to participants
- If not able to report 6 independent measurements, please report as many independent measurements as possible
- Minimum sample intake declared on the certificate as 4 mL but 'real' sample intake is lower
- Stability on reconstituted material, do not store for more than 4 days
- Reporting form has been sent and since been revised, the density value of BCR-576 is needed for the evaluation

The candidate CRMs value-assignment, technical competence and general quality assurance were shown. The study timeline was provided, with sample dispatch in 2025 Q1/Q2 and the results submission on 30th September 2025. The HFTLS statement was shown. For the pilot study participation, do the participants in the pilot study cause any issues locally for NMIs/DI?

Comments and Questions

Mark – Timeline is optimistic. Can have results discussions between participants separately.

Gavin – The RELA data is hard to go back and check. Don't foresee any issues. Participants will be anonymised and concerns about reputation will be protected.

No objections for updated protocol and pilot lab participation. Gavin will check for any regional bias and will report back to OAWG.

Maria – Not seeing any objections, so on behalf of the Organic Analysis Working Group, we give our consent for the pilot labs to participate.

Action: 11 Gavin O'Connor will check for any regional bias for the pilot study participants and will report back to OAWG.

22. OAWG Clin Tox TG strategy inputs, case study update & 2030+ KC discussion – Gavin O'Conner

Gavin reported back on the activities of the clinical sector focus group – need new members. Met once in June 2024. Gavin provided an overview of the task group, and the specific activities undertaken were listed. The activities carried out by Gavin were shown and he requested that other task members cross check these. The stakeholder needs and lists of materials were mapped. This showed prioritisation for measurands in blood, serum or plasma. The new rankings in terms of ICHLR were shown. Nothing has really moved and no real update in this space. Listed CCQM studies – 18 key comparisons and 10 measurands. The mapped-out sectors were shown, and an updated graph of the dot diagram was shown, with red dots for high priority for stakeholders. Gavin showed the grouping of measurands. The current availability of CRMs (JCTLM Database) was shown as well as the reference material procedures on the JCTLM database. Projected about 50 years to meet the requirements for the stakeholders and will take a community effort to fill the space for stakeholders.
The planned Track A OAWG studies were shown. We have a lot of claims and need to back those claims with studies in those areas. The key comparisons were shown, and there is a hole in Category 3 where stakeholders have said are important. Gavin showed the CCQM studies, CMCs and JCTLM listed CRMs. The planned Track A OAWG studies were shown and there were discussions around dry blood spot sampling, and the need to support this. KRISS have a CRM of dried blood spot

Comments and Questions
Maria – Do we want to have a forensics study?
Bruno - Depends on HFTL statement.
Ralf – There are two calibrators in sector 3.
Marina – Contaminants in residues?
Mark – There have been requests to feedback on the categories.
Gavin – Task groups to review categories and feedback.
Maria – CCQM strategy document will be finalised in May 2025. This needs checking and feedback on what is being proposed. Please forward comments and input.

Action: 12 Task Group members to check and validate the clinical sector data mapping presented by Gavin O'Connor.

OAWG Food Sector Focus

23. Proposal for Joint Isotope Ratio Mass Spectrometry Workshop / Joint IAWG workshop, Zoltan Mester

This meeting segment discussed a proposed joint workshop between the Isotope Ratio Working Group and the Organic Analysis Working Group, planned for April next year. Zoltan Mester, chair of the Isotope Ratio Working Group, presented the idea of a workshop on site-specific isotope ratio measurements using NMR and high-resolution mass spectrometry technologies. The workshop aims

to explore these techniques for food authenticity and adulteration detection. Members expressed interest in the workshop, recognizing its potential for knowledge sharing and future collaborations. The discussion concluded with a call for volunteers to form an organizing science committee.

24. Draft A results for CCQM-K180 Metronidazole in pork – Joachim Polzer, BVL

Summary

The presentation focused on the results and discussions of the analysis of metronidazole in animal muscle tissue. The study involved multiple laboratories and used different extraction methods. The results showed good agreement between the laboratories, with two outliers that were later excluded. The presentation also discussed the challenges faced during the shipment of the pure standard reference materials and the difficulties in obtaining veterinary certificates for the animal samples. The presentation ended with a discussion on the final key comparison reference value (KCRV) calculation and the need for a separate meeting with the participants. The study also highlighted the importance of considering the dry mass of the material and the need for additional analytes in future studies.

JP presented the timeline, the study started in August 2022 and there was a presentation of the Draft B report in April 2025. There were 18 registered participants, and 17 participants received samples.
JP summarised the previous meetings and decisions, including the calculated KCRV. An overview of the results was shown, and two labs were outliers compared to the other participants. A comparison of the different datasets was shown. The questions/outcomes from the meeting in October 2024 were summarised. The moisture content was focused on and questions to participants were should KF be used for water content. The follow up activities from VNIIM were reported on. They received additional material and did more extractions, and the third extract still gave some native metronidazole. The different extraction techniques were shown. The KCRV discussion from previous meeting was summarised, and the KCRV options and NIST decision tree were shown. The values from the different models were compared and are quite similar. Overall, there is good agreement of participants values. The DSL and DoE results were shown. For the DoE, most labs are overlapping. Outstanding activities include revision of the data by the statistics team and the selection of the KCRV. JP summarised the experiences of the study that could be helpful as learnings for future studies. Question, what KCRV would be appropriate to use.

Comments and Questions

Mark – NMIA would support the lower expanded uncertainty.
Maria – No objections for Der Simonian Laird estimate (DSL). The Draft B report can be circulated and then the study can be finalised.
Mark – Is the dark uncertainty due to the inhomogeneity of the material. This could be compared to the homogeneity study results.
Mark – Despite the extra delay of 1 year, Mark thanked the laboratories that performed additional work to get to this point.

Action: 13 Joachim Polzer to proceed with the Draft B report for K180 using the DSL model.

25. Feedback CCQM Food measurements task group – Kate Rimmer, NIST

Kate provided feedback on the task group activities and the membership was shown. A workshop was held in Feb 2025 for 4 days, 2 hours a day with talks/discussions. One of the topics was food authenticity. The challenges in assuring food authenticity were summarised. In the presentation, Kate discussed the progress of the food sector in the CCQM OAWG, highlighting the increase in plant matrix reference materials and PT materials for chemical food hazards. The strategic focus is adequate and there are appropriate measurements related to key classes like mycotoxins, pesticides, and veterinary drugs. Emerging areas of interest include environmental contaminants, food processing, packaging, emerging contaminants, and the impacts of climate change on chemical contaminant profiles. Kate presented updates on case studies, including activities and studies for pesticides and veterinary drugs. The group discussed the frequency of participation in studies, with a consensus of every 5 years or as coordinated by the working group. The group also discussed the possibility of linking purity comparisons to matrix comparisons, such as PFAS in food, environmental, and clinical matrices.

26. OAWG food strategy, case study update & 2030+ KC discussion – Pui Sze Cheow, HSA

PSC showed the task group membership, and they met last July. PSC provided a general overview of the CCQM OAWG strategy for 2030+. Some changes are required to reflect the current trends and emerging challenges. The suggested changes were listed for the relevant texts and figures. New figures were provided. Table 6 was updated to cover up to 2034. The Case Study III was updated and at the end of the document, the updates were listed.

Comments and Questions

Mark – The CMCs in this area are increasing and it is worth checking whether the proposed upcoming studies will support the CMCs.

27. Update on materials for Track A comparison: Fat-soluble vitamins in infant formula (2025/26) - Xiaomin Li, NIM (online)

XL presented a proposal for a Track A comparison for fat soluble vitamins in infant formula. The proposal included a brief background on the nutrients in infant formula, technical background, and information on the target analyte. XL also discussed the sample treatment, method of analysis, quantification method, homogeneity, long-term stability, and short-term stability of the samples. The proposal also included information on the participants, reference materials, and the proposed schedule for the comparison. The HFTLS statement was covered. The proposed schedule was shown with invitation for participation in November 2025 and discussion of results in October 2026.

Comments and Questions

Kate – What is the base of the formula. Xiaomin responded that it is milk based.

Bruno – Asked to see the homogeneity graph. There appears to be a large variation in the standard uncertainty.

Mark – If the material is not that homogeneous, that will limit the KCRU.

This is not a commercial product, but from a collaboration with NIM. It was proposed to remix and repackage the material to get better homogeneity. It has already been mixed and repackaged.

It was pointed out that only KRISS have a CMC in this area. This will be a Track A study, and institutes will need to participate to maintain CMCs or make a broad scope claim.

BJ – For the scattering on homogeneity, the scattering may be due to analytical techniques. Do you have homogeneity test results for compounds other than for vitamin D?
Checked for the homogeneity for other vitamins measured and all have similar uncertainty values for homogeneity.
Maria – If the study uncertainty is greater than institutes', can still use for CMCs but may be problematic.
BJ – For vitamin D in milk, CoA has U of 7%
Maria – Can NIM look at the homogeneity and if can reduce the variation?
XL – If they can find a way to increase the homogeneity, would NMIs be interested in this study? At the last OAWG meeting, 7 NMIs showed interest. What is the timeframe for trying to increase the homogeneity? Can send it out by email for communication.
Maria – Please can the calibrator be shipped with the samples.

Action: 14 NIM to conduct additional homogeneity testing for vitamin D3 in infant formula material (to reduce uncertainty) within 3 months and report back to the working group.

Action: 15 Working Group members to review current CMCs and determine if they fall within the vitamin D3 in infant formula comparison space.

Action: 16 NIM to prepare draft protocol for the vitamin D3 in infant formula comparison study after completing homogeneity analysis.

Action: 17 NIM to consider shipping vitamin D3 calibrator along with samples for the comparison study.

28. Proposals for next Track A/ Track C comparisons – Pui Sze Cheow, HSA

PSC provided an overview of the past comparisons and regional comparisons from 2017, to show any gaps. Table 6 was shown with proposals for 2027 and 2032. For 2029, still looking for a coordinating institute. The results from the food survey were show for KCDM. There is a proposal by KRISS for a Track A study with pesticides in strawberry. The initial proposal for consideration was show, along with the available CRMs, polarity and molecular weight. The mass fraction range was shown. Other proposals by KRISS for consideration were listed. There is a proposal by NRC for a Track A Mycotoxins in a high carb matrix (2032). The proposal was shown with the availability of CRMs, the polarity and molecular weight. An update was provided on the edible insects proposal by HSA which would be a Track C contaminants in edible insects. The available calibrants, polarity and molecular weight were shown. The preparation of the black soldier fly larvae material was shown, and the fly larvae were fed with the contaminants. There is sufficient homogeneity for mycotoxins, sulfadiazine and PAHs (except chrysene). Other proposals for PFAS in food studies were shown. The floor was opened for comments on the proposed studies:

1. Pesticides in Strawberry (2027, KRISS) – several analytes on offer in a freeze-dried material. KRISS can provide more information on material, analytical techniques, CRMS and performance of analytes in the material.
2. Mycotoxins in High Carb Matrix (2032, NRC) – tentatively reserve for 2032 and agree in principle.
3. Contaminants in edible insects (2027) – Track C. Would be at the same time as Strawberry study. How many NMIs would be interested? METAS, NIM, BVL, INMETRO, HSA, NIMT

Action: 18 HSA to continue investigation of high chrysene results in black soldier fly larvae powder due to potential blank contamination.

Advances In Measurement Science

29. Future actions following on Mass Spectrometry workshop (22 April 2024) - Track D comparisons – Gavin O'Connor, PTB

As a follow up for the IDMS workshop and possible Track D studies, there were no suggestions, so some suggestions were proposed by GOC. These include an update to the paper/monologue to include: basic concepts and benefits of IDMS, single IDMS, double IDMS and impact of new instrumental approaches. Gavin proposed a collaborative study/experiment to assess the capabilities of various mass spectrometers for doing isotope dilution mass spectrometry (IDMS) measurements. The experiment would involve preparing gravimetrically prepared blends, running samples on different platforms, and processing the data. The goal is to evaluate the platforms for doing IDMS measurements and provide a set of recommendations for future instrument purchases. Gavin suggested that the experiment could be designed by a study oversight group, which would also include a statistics expert. The experiment would involve running samples on as many platforms as possible, including different mass spectrometry modes. The data generated would be used as examples in the IDMS pamphlet. The suggested timelines and discussions were provided with a start in April 2025. Gavin's email address was provided and he encouraged interested participants to email him.

Comments and Questions

Mark - Would like to contribute to the document and study design (and oversight group).

Action: 19 Gavin O'Connor to form an oversight group for the IDMS Track D study design.

Action: 20 Working Group members interested in contributing to the IDMS document to contact Gavin O'Connor.

30. Future actions following on qNMR workshop (8 April 2025) - Track D comparisons – Bruno Garrido, NRC Canada

Summary of qNMR workshop

The Organic Analysis Working Group and the Protein Analysis Working Group held a joint workshop on quantitative nuclear magnetic resonance (qNMR) to progress measurement science, with a focus on the use of alternative nuclei like fluorine and phosphorus, and the application of deconvolution and quantum mechanical approaches for data handling. The team discussed strategies for achieving high accuracy qNMR measurements in complex samples and calibration solutions, the use of solvent suppression techniques and external standards in qNMR for analysing toxin samples at low concentrations, and advanced data processing techniques for NMR spectroscopy, focusing on deconvolution methods. The meeting covered advancements in NMR spectroscopy, including

quantum mechanics and artificial intelligence applications, as well as the development of new standards for biological medicines and vaccine polysaccharides. Discussions focused on various techniques for enhancing measurement selectivity in qNMR, comparing different methods for qNMR analysis, and potential studies. These will be discussed further at the upcoming October meeting.

31. OAWG advances in measurement science, case study update & 2030+ KC discussion – Bruno Garrido, NRC Canada

Bruno presented a summary of the Advances in Measurement Science Task Group's activities and membership. The group has identified several gaps in measurement science, including quantitative NMR with crowded spectra, qualitative analysis, non-targeted mass spectrometry, and ultra-trace analysis of compounds without CRMs. They recommend expanding comparison materials, fostering knowledge exchange, conducting pilot studies, and developing new measurement uncertainty approaches.

32. Update BERM/ ISO TC 334 and qNMR summit, other events – Bruno Garrido, NRC Canada

Bruno invited attendees to the upcoming BERM16 meeting in Halifax Canada. Three events will be hosted:

1) BERM16: International Symposium on Biological and Environmental Reference Materials
2) qNMR Summit 2025
3) ISO TC 334 (by invitation)

33. Any other business or topics to cover (outstanding discussions that were not concluded) – Maria Fernandes-Whaley NMISA

Definition of a water matrix – Alix Rodowa, NIST

The environmental sector lead, Alix Rodowa presented a proposed categorization of water matrices into low and high complexity, based on human exposure. The group agrees to this categorization, with CMCs obtained in high complexity matrices also applying to low complexity, but not vice versa. For soil, sediment, and particulate matter, they propose to categorize based on human-related exposure. The group discussed how this aligns with service categories in the KCDB and agrees to provide clarity on the categorization in the strategy document.

Action: 21 Alix Rodowa to provide further clarity and breakdown on the two water categories (low and high complexity) for environmental matrices.

APMP – Tang Hua, NIM

Tang Hua presented the options for the water matrix in their pesticide study, including deionized water, lab-selected drinking water, or distributed bottled drinking water. After discussion, APMP decided to provide bottled drinking water to participants along with an ampoule for dilution, to ensure consistency across laboratories. The protocol will be updated to reflect this decision and include the environmental sector's recommendation on complexity categorization in the HFTLS statement. The

working group discussed approving the study with amendments, including updating the protocol to reflect bottled drinking water distribution and considering it a low complexity water sample.

Comments and Questions
Alex – Asked about lindane in water and whether this would be a CRM. Lindane belongs to the low polar pesticides and is not stable in water for a long time.
Maria – How will APMP prepare the samples. Will you be sending the sample, so participants are diluting it into the same water sample.
Tang Hua – Participants will be provided with 1 -2 bottles of drinking water. The protocol will be updated to reflect that bottled water will be provided and include the environmental sectors guidance on the water considered to be a low complexity sample.

Action: 22 Tang Hua to update the protocol for the pesticides in water study to reflect that bottled drinking water will be distributed and considered a low complexity water sample.

Action: 23 Tang Hua to share the updated protocol for the pesticides in water study with the working group.

35. Next meeting (on-line October 2025)

The group tentatively scheduled their next meeting for late September or early October, with exact dates to be confirmed due to potential conflicts with national holidays.

Proposed dates for 12h00 – 15h00 (UTC+2) meetings:
Tue, 30 Sep
Thu, 02 Oct
Tue, 07 Oct
Thu, 09 Oct

Action: 24 Maria Fernandes-Whaley to circulate proposed dates for the next Organic Analysis Working Group meeting.

Action Items - Summary

Action: 1 Please can institutes review the example summary reports and provide feedback to Mark Lewin. 5
Action: 2 Task group to be set-up for CMCs and suggested to have a representative from RMOs. Led by Mark Lewin 5
Action: 3 Sector leads, please come back with suggestions for purity comparison considerations..... 6
Action: 4 BIPM to update digitoxin study protocol with new results submission deadline of March 2026. 7
Action: 5 Mark Lewin to analyse database to identify NMIs with CMCs that have molecular mass above 500 and need to participate in digitoxin study 7

Action: 6 BIPM to update the mycotoxins calibration protocol with the new deadline of March 2026 and expanded concentration range. 9
Action: 7 Statistical panel (Juris Meija) to provide guidance on appropriateness of using hierarchical Bayesian method for KCRV calculations in CCQM-K184. 11
Action: 8 Tang Hua to update KCRV calculations based on the statistical panel's recommendations. . 11
Action: 9 Tang Hua to distribute updated Draft B report to participants after resolving the traceability and statistical methodology issues. 11
Action: 10 Working group to review and approve the Draft B report for the PAHs in sediments study via email. 11
Action: 11 Gavin O'Connor will check for any regional bias for the pilot study participants and will report back to OAWG. 18
Action: 12 Task Group members to check and validate the clinical sector data mapping presented by Gavin O'Connor. 18
Action: 13 Joachim Polzer to proceed with the Draft B report for K180 using the DSL model. 19
Action: 14 NIM to conduct additional homogeneity testing for vitamin D3 in infant formula material (to reduce uncertainty) within 3 months and report back to the working group. 21
Action: 15 Working Group members to review current CMCs and determine if they fall within the vitamin D3 in infant formula comparison space. 21
Action: 16 NIM to prepare draft protocol for the vitamin D3 in infant formula comparison study after completing homogeneity analysis. 21
Action: 17 NIM to consider shipping vitamin D3 calibrator along with samples for the comparison study. 21
Action: 18 HSA to continue investigation of high chrysene results in black soldier fly larvae powder due to potential blank contamination. 22
Action: 19 Gavin O'Connor to form an oversight group for the IDMS Track D study design. 22
Action: 20 Working Group members interested in contributing to the IDMS document to contact Gavin O'Connor. 22
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