**Report from the CCQM Inorganic Analysis Working Group for the period (April 2019 – June 2020)**

**Michael Winchester, CCQM IAWG Chair**

**Introduction**

Michael Winchester (NIST) was appointed as the new chair of the IAWG during the CCQM plenary meeting held at the BIPM in April 2019; Paola Fisicaro (LNE) continues her tenure as the vice-chair. The new chair hopes to carry on the excellent leadership of the previous chair, Mike Sargent.

The IAWG met at UNIIM on 10-12 September 2019, and the meeting was attended by 33 delegates representing 19 institutes. The first two days constituted the working group meeting, and the third day was devoted to a workshop titled “Direct and indirect approaches to the purity assessment of high purity metals and salts.” This workshop was the latest of a series of activities within the IAWG, ranging from workshops to pilot studies and key comparisons to the development of an IAWG guidance note, to advance the state-of-the-art and share best practices on purity assays of high-purity inorganic materials. This is a topic of importance, because high-purity materials ultimately form the basis for SI-traceability in inorganic chemical analysis. Therefore, the IAWG expects to continue focusing attention on this topic for some time to come.

During the ongoing coronavirus pandemic, the IAWG has met virtually twice so far. The first VC was held on 14 May 2020 and covered general topics, such as the need for additional IAWG guidance notes, KCRV calculations, and scheduling problems caused by delays of comparisons due to the pandemic. The second VC was held on 18 June 2020, during which several comparisons in progress were discussed and two new RMO supplementary comparison proposals were considered. Several more VC meetings are expected over the coming months.

**General Issues**

The IAWG has worked over the past year to develop further its core capability (CC) approach to enable maximized support for CMC claims (both broad and specific claims) with a minimized number of key comparisons. The current CC matrix consists of about 150 combinations of common matrix type, analyte type, and analyte content level. Fortunately, not all of these are relevant to CMC claims desired by the IAWG institutes, reducing the number of relevant combinations to maybe 50. However, this is still too many for the IAWG to support realistically, and so further work is needed. It is unclear at this point how much the CC approach will help with CMC claim sustainability. It is clearer that the approach can cause CMC claim review to be more difficult and to require more expert opinion than the KCWG can provide, at least under the current CMC claim review paradigm.

The IAWG has decided to develop several guidance notes for its work, beginning with templates for new comparison proposals and new comparison reports. While the IAWG has functioned well over the years without such guidance notes, it is believed that such notes will enable better planning, effectiveness, and efficiency. This is especially important, because of the need to support broad scope CMC claims with a minimum of comparisons, making it necessary to get as much out of each comparison as possible. While no decision has yet been made, the IAWG is also considering revising and codifying its approach to KCRV estimation, with one of the goals being to harmonize the approach with the other CCQM WGs.

Similar to most of the other CCQM WGs, the IAWG is experiencing problems, caused by the pandemic, with scheduling of comparisons. Because so many institutes have been at reduced capacity, progress continues to be delayed, especially where preparation, dispatch, and analysis of samples is involved.

An IAWG member institute, the IJS in Slovenia, has been one of the institutes whose CMC claims were jeopardized by the exit of the JRC from EURAMET. Specifically, the IJS depended on an IRMM CRM for calibration and SI-traceability for its *k*0 neutron activation analysis (NAA) measurements. The particular CRM unit was purchased by the IJS prior to JRC’s exit, but was due to expire, which would in turn invalidate the related CMC claims of the IJS. A plan was put in place to allow the IJS to test the IRMM CRM unit for stability and extend the expiration date by 5 y to give the IJS enough time to find another suitable CRM; NIST volunteered to help with this. Fortunately, the JRC has now re-joined EURAMET, solving the problem without the need for such stability testing.

**IAWG Key Comparisons**

The IAWG has 10 key comparisons, some with parallel pilot studies, that have been approved by the CCQM and are currently in progress. In Table 1, they are delineated according to track, model, status, and progress over the time period covered by this report. Note that the last five comparisons in the table are being delayed by the pandemic and will require rescheduling to spread them out over time, so that they do not all occur on schedules that are too similar. A related effect of the pandemic is that the IAWG cannot adopt many new comparisons until the calendar can be cleared a bit.

Though not shown in Table 1, the IAWG also has two standalone pilot studies that have been completed and for which journal publications are being prepared. These include CCQM-P160 (Isotope ratios / molar mass measurements of Si isotopes in isotopically enriched silicon) and CCQM-P194 (Number concentration of colloidal particles in solution).

Also not included in Table 1 are studies which are yet to be formally approved by the CCQM, but for which designations have been attained from the KCWG, including CCQM-K166/P210 (Measurement of nanoparticle number concentration in liquid suspension; joint with the SAWG) and CCQM-P215 (Arsenic speciation in seafood – aquatic animals). CCQM-K166/P210 is considered a Track C comparison under the terminology employed by most of the CCQM WGs (the IAWG has not traditionally used this terminology). Both CCQM-K166/P210 and the preceding CCQM-P194 are unique in the IAWG in that the measurand is nanoparticle number concentration in liquid suspension, not a measurand that would usually be considered in the realm of inorganic chemical analysis. The reason for this work being in the IAWG is that one of the measurement methods is single-particle (sp)ICP-MS, which naturally belongs in the IAWG. Because other applicable techniques reside in the SAWG, institutes from both the IAWG and the SAWG participated in CCQM-P194, and CCQM-K166/P210 will be registered under both WGs.

**Advancing Measurement Science in the IAWG**

The IAWG continues to undertake activities to advance inorganic chemical metrology. Several of the comparisons shown in Table 1 include measurements of elemental speciation, which is still not an advanced field. The IAWG studies are helping to improve the state-of-the-art and to build capacity amongst its member institutes. As noted in the preceding section, the IAWG also has activities in the area of nanoparticle metrology. Indeed, nanotechnology will continue to be a focus of the IAWG for the next several years.

Finally, it should be noted that the IAWG is working with other CCQM WGs in some of these endeavours. Specifically, the nanoparticle metrology comparisons are being carried out cooperatively with the SAWG for reasons explained previously. Another case is CCQM-K151/P191 (Mass fraction of a purity-assessed recombinant protein in an aqueous calibration solution using amino acid-based ID-LC-MS and/or sulfur-based ID-ICP-MS). All participants in K151 are members of the PAWG, and only methods belonging in the PAWG’s purview were used. In contrast, all participants in P191 are in the IAWG, and only methods belonging within the IAWG were employed. The key point is that K151 and P191 are intended to support one another, because they involve two completely different approaches for quantitation. This work is expected to be published in a scientific journal.

Table 1. Official key comparisons and parallel pilot studies of the IAWG for the period (April 2019 – June 2020), including progress made.

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| --- | --- | --- | --- | --- |
| **CCQM Designation** | **Title** | **Track a** | **Model b** | **Status** |
| **April 2019** | **June 2020** |
| K34.2016.1 c | Assay of KHP | A | 1 | Protocol and sample development | Draft A |
| K143/P181 | Copper calibration solutions | A | 2 | Draft A | Draft B |
| K144/P182 | Trace elements in alumina powder | A | 1 | Measurement | Draft A |
| K151/P191 d | Mass fraction of a purity-assessed recombinant protein in an aqueous calibration solution using amino acid-based ID-LC-MS and/or sulfur-based ID-ICP-MS | A | 1 | Draft B | Draft B |
| K152/P192 c | Assay and purity of potassium iodate | A | 1 | Draft A | Draft B |
| K155/P196 | Elements in seawater | A | 1 | Protocol and sample development | Measurement |
| K158/P200 | Elements and inorganic As in rice | A | 1 | Protocol and sample development | Protocol and sample development |
| K160/P203 | Platinum group elements in automotive catalyst | A | 1 | Protocol and sample development | Protocol and sample development |
| K161/P207 | Anions in seawater | A | 1 | Approved at CCQM plenary | Protocol and sample development |
| K162/P208 | Selenoproteins in serum | C | 1 | Approved at CCQM plenary | Protocol and sample development |

a The IAWG does not use the “Track” nomenclature, but it is included here, because many of the CCQM WGs do use it. “Track A” refers to measurements that constitute core functions performed routinely by the member institutes; “Trace K” refers to more specialized measurements that are not yet commonly used to provide measurement services.

b “Model 1” means the pilot institute sends samples to the participants to be measured; “Model 2” means the participants send samples to the pilot institute for measurement.

c Joint with the EAWG.

d Joint with the PAWG.