CCQM WG on Surface Analysis (SAWG)

Report on activities April 2024– April 2025

# General

A hybrid meeting of SAWG was held on 24 April 2024 with 12 in-person participants and 14 virtual participants from 15 countries. A joint IAWG / SAWG workshop on the measurement of nanoparticle concentration on 23 April 2023 was attended by SAWG representatives.

Dr Alex Shard from NPL, UK has been SAWG chair from 2023 and Dr Li Lin Tay from NRC, Canada is SAWG vice-chair.

# CCQM-K166/CCQM-P210; Measurement of nanoparticle number concentration in liquid suspension (joint with IAWG, led by LGC, UK)

Key comparison K166 and P210 (number concentration of 60 nm gold particles), led by LGC, received the final reports from participants in November 2023. Preliminary results were circulated on 30 Jan 2024 with good performance from participating NMIs. Ten laboratories submitted results for K166 and seven laboratories submitted results for P166. First inspection demonstrates a symmetrical distribution of results with no clear outliers. The final report is in preparation.

# CCQM-P229; Measurement of measurement of mole fractions in PtxNi1-x alloy films (led by KRISS, South Korea)

Platinum-nickel alloys are an important material in the field of energy storage as a catalyst for polymer electrolyte membrane fuel cells (PEMFC). The objective of this pilot study is to measure the relative mole fractions of Pt and Ni in thin PtxNi1-x alloy films and to check the feasibility as a candidate subject of a key comparison in the Surface Analysis Working Group of CCQM. The measurand of this pilot study is the mole fractions of Pt in alloy films units of mole fraction (cmol/mol). The protocol for the study was circulated in June 2023 and 11 participants from 8 countries are taking part in the study using techniques which include XPS, XRF, EPMA, EDX and APT. Results have been returned to KRISS and discussed at the April 2024 meeting. A final report will be made at the SAWG meeting in April 2025. It is fully expected that a key comparison will commence in 2025.

# CCQM-P230; Measurement of surface elemental mole fractions in ionic liquids (led by BAM, Germany)

Light elements, e.g. C, N, O, F are constituent components of organic and carbon-based materials, polymers, biological and biomaterials. Therefore, reliable quantitative analysis of them is of great interest and is still challenging for surface analysis methods like X-ray photoelectron spectroscopy (XPS), X-ray fluorescence spectroscopy (XRF) or electron probe micro analysis (EPMA). This study aims to underpin core measurement services related to measuring the elemental composition of organic surfaces using a homogeneous, liquid material which has excellent stability under vacuum and ionizing radiation as well as a known composition. This study has been delayed due to the requirement to source sufficient quantities of ionic liquids and validate their suitability. The study will commence after the April 2025 meeting.

# CCQM-K136.2025 and P243; Measurement of porosity properties of nanoporous silica (led by UME, Turkey)

A repeat key comparison of the measurement of gas sorption onto nanoporous media was launched this year. This study uses nanoporous silica rather than the nanoporous alumina which was studied in the original K136 comparison completed in 2016. The purpose of the repeat comparison is to ensure that NMI capabilities are maintained and to enable the participation of NMIs that were not included in the first comparison. Homogeneity and stability testing was complete in November 2024. Samples were distributed to six participants on 12th December 2025. There were some delivery problems leading to samples having to be redispatched. Results will be returned to UME before 15th June 2025.

# Other potential comparisons and interactions with other working groups

Dr. Li-Lin Tay (CNRC, Canada) provided an update on the preparation of quantitative measurement with Raman spectroscopy for mixed polymer blend samples. Dr. Tay reported on the development of the Raman measurement protocol developed at NRC and some potential schemes for the pilot study. It was proposed to run a CCQM pilot study in parallel with a VAMAS TWA42 study to maximise impact. The pilot study is expected to be initiated very soon.

Members of SAWG are participating in P222; *Number concentration measurement of particles for cellular analysis* led by CAWG and P244; *Lipid nanoparticles with encapsulated RNA* led by NAWG. These activities are being covered within the IAWG-SAWG task group on particle metrology.

# Activities of other organizations

VAMAS TWA 2 (surface analysis), TWA41 (graphene and 2D materials), and TWA42 (Raman) are progressing several interlaboratory comparisons that are of relevance to SAWG. These include quantification in atom probe tomography (TWA 2); Measurement of drug dose in biological tissue by SIMS (TWA 2); Analysis of surface functionalized oxide nanoparticles (TWA 2); Interface location in organic SIMS depth profiles (TWA 2); Chemical measurements by Total reflection XRF (TWA 2); Thickness of thin oxide films by XPS and ion scattering (TWA 2); Disorder and number of layers of graphene flakes (TWA 41); Raman measurements of polymorph fraction in TiO2 nanoparticles (TWA 42); Factors affecting the reproducibility in Raman spectroscopy (TWA 42). A number of standards on nanomaterial analysis and instrument calibration are in progress within ISO TC229 (nanomaterials) and ISO TC201 (surface chemical analysis) which are of interest to the SAWG community.

# IAWG / SAWG Task Group on particle metrology

The task group on nanoparticle metrology was established in 2023 and the elected chair is Caterina Minelli (NPL, UK). Several virtual meetings have been held and the terms of reference have been decided. These are outlined below. An update on the activities of the task group will be provided during the joint IAWG / SAWG meeting on 8th April 2025.

*Terms of reference*

To identify activities that the IAWG and SAWG should undertake with respect to particle metrology over the next ten years, including pilot studies, key comparisons, and cooperative research projects. To accomplish this, the TG will:

* Examine the outcomes of the CCQM Workshop on Particle Metrology held 25-27 October 2022;
* Liaise with external stakeholders to understand better the important needs and gaps in particle metrology that can be addressed by the IAWG and SAWG;
* Liaise with the CCL WG-N to leverage knowledge and identify opportunities for cooperation between (nano)dimensional, chemical, and biological activities with respect to particle metrology.

The findings of this TG, including actionable proposals, will be delivered in a written report in the next period.

# Next meeting

The next SAWG meeting will be held on 9th April 2025 at BIPM headquarters, Paris. This will be a hybrid meeting with on-line attendees. The agenda has been circulated.