**Final Minutes**

**Surface and Micro/Nano Analysis Working Group (SAWG)**

**23rd Meeting**

Hybrid Meeting on April 9th, 2025

Myria Room (BIPM) and Online

Draft Version 1, April 25th, 2025

**NOTE: All documents related to this meeting are on the SAWG website**  
<https://www.bipm.org/en/committees/cc/ccqm/wg/ccqm-sawg>

**Participants:**

CCQM-SAWG Chair

Dr Alexander Shard, NPL

CCQM-SAWG Vice-Chair

Dr Li-Lin Tay, NRC

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| In-person participants:  Dr Benjamen Reed (NPL, UK)  Dr Seung Mi Lee (KRISS, South Korea)  Dr Yaxuan Yao (NIM, China)  Dr Hiroyuki Matsuzaki (NMIJ/AIST, Japan)  Dr Andrea Mario Rossi (INRIM, Italy)  Dr Ute Resch-Genger (BAM, Germany)  Dr Michael Krumrey (PTB, Germany)  Dr Rainer Stosch (PTB, Germany)  Dr Egor Sobina (UNIIM, Russia) | Online Participants:  Dr Abdullah Othman (NMIM, Malaysia)  Dr Ali Enis Sadak (TÜBİTAK, Türkiye)  Dr Angela Hight-Walker (NIST, USA)  Dr Braulio Archanjo, (INMETRO, Brazil)  Dr Caterina Minelli (NPL, UK)  Dr Charles Clifford (NPL, UK)  Dr Donald Windover (NIST, USA)  Dr Froy Martinez (CENAM, Mexico)  Dr Hai Wang (NIM, China)  Dr Jeong Won Kim (KRISS, South Korea)  Ms Katherine Cárdenas Daza (INM, Columbia)  Ms Maribel Moreno Ferero (INM, Columbia)  Mr Adlan Akram Mohamad Mazuki (NMIM, Malaysia)  Dr Norma González(CENAM, Mexico)  Dr Rania Sayed (NIS, Egypt)  Dr Victoria Coleman (NMIA, Australia)  Mr Werner Jordaan (NMISA, South Africa)  Dr Jörg Radnik (BAM, Germany) |

**Actions from this meeting for SAWG members:**

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| **#** | **Action** | **Responsible Person/NMI/DI** |
| 1 | Dr Shard to correct the pilot study number in the SAWG Activity Report (2) slide from P229 to P248 before uploading to the SAWG documents directory (or edit afterwards). | A. Shard (NPL) |
| 2 | Dr Shard to speak to Dr Julian Braybrook to inform him that Dr Caterina Minelli has volunteered to represent SAWG in the TG for gene delivery systems, and the discuss the possibility of a liaison with CCQM-IAWG-SAWG-TG-PARTICLE. | A. Shard (NPL)  *Complete* |
| 3 | SAWG members to contact Dr Shard and inform him what sorts of CMC claim would be most useful and relevant to them and suggest activities that SAWG that be doing to help them with their measurement and/or calibration services before the 24th SAWG Meeting in September 2025. | All SAWG members |
| 4 | SAWG members that wish to participate in P230 must inform Dr Jörg Radnik before the end of May 2025. | All SAWG members |
| 5 | Dr Shard to request a study number for the traceable quantitative measurements of polymer weight fraction pilot study by the end of August 2025. | A. Shard (NPL) |
| 4 | Dr Tay to update Dr Shard on the progress of resolving the solvent issue for the future traceable quantitative measurements of polymer weight fraction pilot study by the end of August 2025. | L. L. Tay (NRC) |
| 7 | Dr Stosch and Dr Shard to clarify whether strain measurands are placed under SAWG or another CC by the end of August 2025. | R. Stosch (PTB) |
| 8 | Dr Shard to set up a TG in SAWG on 2D materials and appoint a TG lead by the end of May 2025. | A. Shard (NPL) |
| 9 | Dr Hight-Walker and Dr Shard to define terms-of-reference for new SAWG TG on 2D materials by the end of July 2025. Ideas for pilot studies can be generated afterwards. | A. Hight-Walker (NIST) |
| 10 | Dr Resch-Genger to circulate qNMR and XPS protocol to the SAWG member for comment by the end of July 2025. | U. Resch-Genger (BAM) |
| 11 | Participants of P248 to check and calculate their uncertainties using new certified values of Pt-content and uncertainties from the reference alloys acquired using ICP-MS and report back to Dr Lee before the end of June 2025. | P248 Participants |
| 12 | Participants of P248 to review Draft A report and provide comments and corrections before the end of June 2025. | P248 Participants |
| 13 | Dr Lee (and KRISS) to complete Draft B report of P248 by end of August 2025 and launch the KC. | S. M. Lee (KRISS) |
| 14 | Dr Shard to contact the chair of the TG on microplastics and express SAWG’s interest in a leading a study in microplastics. In addition, Dr Rossi to suggest a possible study that SAWG could lead including what materials and measurands such a study would focus on before the end of August 2025. | A. Shard (NPL) A. Rossi (INRIM) |
| 15 | All SAWG members to register as CMC reviewers in the KCDB before the end of August 2025. | All SAWG members |
| 16 | Dr Shard said that the content of Dr Stosch’s presentation can be put into a SAWG guidance document and asked for volunteers (preferably those who have experience writing or reviewing CMCs) to begin drafting this document before the end of May 2025. | All SAWG members |
| 17 | CCQM-SAWG to reconvene in September 2025 to provide updateson activities and actions. | All SAWG members |

# **Opening**

## **Opening of the meeting**

The Chair of the Surface Analysis Working Group (SAWG), Dr Shard welcomed everyone to the 23rd SAWG meeting and briefly stated the CCQM and SAWG scope and remit.

## **Appointment of the Rapporteur**

Dr Shard requested Dr Benjamen Reed (NPL, U.K.) to act as rapporteur for the April 2025 meeting. Dr Reed agreed to act as rapporteur.

## **Adoption of the agenda**

The draft agenda for this meeting, titled “Final agenda V5 CCQM SAWG 2025 hybrid meeting” was circulated on the 1st April 2025. The start time of “Interlaboratory comparison on microplastic characterization using spectroscopic methods. / Andrea Mario Rossi (INRIM)” was incorrectly placed at 16:45, this has been corrected to 16:35. The draft agenda was adopted following this correction.

## **Review of the SAWG 2024 meeting and progress report**

The draft minutes for the 22nd meeting of SAWG in 2024, titled “SAWG Draft Minutes 2024\_04 v1” were circulated on 25th June 2024. No comments or corrections were received by the 2nd April 2025. Dr Shard reminded SAWG members that ‘actions’ from SAWG meetings will be stated in the minutes. Dr Shard recommended that timescales for future actions should be included to ensure that progress is made. Four actions were raised from the 22nd meeting and there are no actions outstanding.

## **Progress Report**

Dr Shard then reported on the progress of four main activities of SAWG.

**SAWG Activity #1:** CCQM-K166/CCQM-P210; Measurement of nanoparticle number concentration in liquid suspension (joint with IAWG, led by LGC, UK)​

**SAWG Activity #2:** CCQM-P248; Measurement of measurement of mole fractions in PtxNi1-x alloy films (led by KRISS, South Korea)​ [with corrected pilot study number]

**SAWG Activity #3:** CCQM-P230; Measurement of surface elemental mole fractions in ionic liquids (led by BAM, Germany)​

**SAWG Activity #4:** CCQM-K136.2025 and P243; Measurement of porosity properties of nanopourous silica (led by TÜBİTAK UME, Türkiye)​

## **SAWG Representation**

Dr Shard then commented on the representation of SAWG in other TGs and WGs across CCQM. The current representatives are shown in the table below.

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| --- | --- |
| **Group** | **Main SAWG representative** |
| Strategic Planning Working Group | Alex Shard |
| Key Comparison Working Group | Alex Shard |
| *ad hoc* Working Group on the Mole | Alex Shard |
| Task Group on Food Measurement | Andrea Mario Rossi |
| IAWG-SAWG Task Group on Nanoparticles | Caterina Minelli |
| Task Group for gene delivery systems\* | ?Alex Shard |

Dr Shard asked if any SAWG members have a desire to represent SAWG in these groups, and if they do, they should email him. Dr Shard then detailed the draft terms of reference and planned activities for the TG for gene delivery systems. Dr Shard stated that said he is currently standing in as SAWG representation for this TG but has no expertise in this area.

* Dr Minelli from NPL volunteered to represent SAWG in the TG for gene delivery systems and suggested that a liaison is established with CCQM-IAWG-SAWG-TG-PARTICLE given the high synergy of their activities. Dr Shard thanked Dr Minelli and stated that he will discuss the possibility of a liaison with the Dr Julian Braybrook from LGC (chair of CCQM-CAWG setting up the TG for gene delivery systems) later.

## **Introduction of SAWG attendees**

SAWG contact persons have access to all restricted documents concerning past and upcoming meetings. Participants of this meeting have access to the documents related to this meeting only if they are registered in the BIPM database.​ Participants (both in-person and online) were invited to provide a short introduction of themselves and their activities / techniques that are within the scope of SAWG.

# **Brief report and actions from the IAWG-SAWG joint meeting on nanoparticles** / A. Shard (NPL)

Dr Shard summarised the discussion from the joint meeting IAWG and SAWG on nanoparticles activity was held on the 8th April 2025. Dr Shard shared the agenda of joint meeting and then quotes from stakeholders using nanoparticles in 2015. SAWG was able to access European metrology programs in 2015 to help start to develop metrology in this area, namely in the concentration (e.g. gold particle RMs used in pilot studies and ongoing KC in CCQM, parallel VAMAS study with polystyrene particles, etc), agglomeration, and surface chemistry (e.g. XPS and ion scattering for coatings of peptides on gold, VAMAS study showed good comparability, ISO standard published). With regards to surface chemistry, there are currently no activities in CCQM, but another European metrology program project is starting soon led by Dr Ute Resch-Genger.

Referencing Bell *et al.* [Analytical Methods 5 (2013), 4523], Dr Shard said that are many possible activities for SAWG but that they rely on obtaining good RMs of different references cores, shells, and mediums. The joint TG on particle metrology (CCQM-IAWG-SAWG-TG-PARTICLE) led by Dr Minelli is nearing its conclusion later this year and there will be a report detailing its findings as well as summarising the outcomes of the CCQM workshop on particle metrology. Dr Shard asked if SAWG members have any thoughts of contributions to this report or anything that stakeholders would find interesting, then they should contact Dr Minelli.

An overview of CCQM-P222 (particle number concentration measurement for cellular analysis) was presented at the IAWG-SAWG joint meeting. This pilot study was not advertised to SAWG because the particles of interest were too big, nevertheless Dr Shard expressed that SAWG should pay attention to how the study will proceed.

* Dr Tay asked how the participants using flow cytometry in P222 establish traceability. Dr Shard was unsure, but suggested the participants would have to know the volume flow rate and account for double counting. Dr Minelli clarified that the particles were not fluorescent, with scattering techniques used, and that the mass of the solution was measured to establish traceability.
* Dr Coleman asked whether the P222 draft report was available. Dr Minelli answered not yet but it will be ready in the autumn/fall.

# Brief Report of the KCWG meeting / A. Shard (NPL)

**Update on SAWG CMCs**

Dr Shard stated that there have been no CMC claims in this cycle. He referenced the recent completed KC on the thickness of hafnium oxide thin films (K-157). The organiser of the study (KRISS) has not made a CMC claim yet. In some cases, participants will be claiming traceability to the organiser of the study, and they cannot do that until the organiser makes their CMC claim. The only claims have been made so far (NPL and PTB) claim their traceability through other means. Dr Shard expressed concern that SAWG continues to not show activity in claiming CMCs, then questions will be raised by CCQM as to SAWG’s purpose.

Dr Shard then asked SAWG members (particularly newer members) what sorts of claims would be most useful and most relevant to them, and therefore what kinds of activity should SAWG be doing to help them with their measurement and/or calibration services. Another concern that Dr Shard raised was that SAWG has not conducted many pilot studies and KCs since 2020. Dr Shard showed the following table summarising SAWGs KC activities since 2005.

|  |  |  |
| --- | --- | --- |
| Number | Start Date | Topic |
| K32 | 2005 | Silicon oxide thickness on silicon |
| K67 | 2008 | Composition of Fe-Ni alloy films |
| K129 | 2014 | CIGS layer composition |
| K136 | 2015 | Surface area of nanoporous alumina |
| K153 | 2017 | Surface area of nanoporous silica |
| K157 | 2018 | Hafnium oxide thickness on silicon |
| K166 | 2020 | Gold nanoparticle number concentration |
| K172 | 2020 | Specific adsorption of argon on zeolite |
| K136.2025 | 2025 | Surface area of nanoporous silica |
| K??? | 2025/2026 | Composition of Pt-Ni alloy films |

Dr Shard expressed confidence that the KC on the composition of Pt-Ni alloy will commence soon. He commented that this follows on from K67 in theme, and that SAWG members should consider how to make these KCs support a general claim for capability rather than a material specific one. Dr Shard also brought attention to K32 and commented that CMC claims were made in 2008 and in most other WGs, these KCs are too old to make CMC claims against. Dr Shard suggested that this KC could be repeated because the metrology has already been established, one sample may be enough to demonstrate capability, and it gives newer SAWG member an opportunity to make CMC claims for the thickness of silicon oxide on silicon. A pilot study could be extended to higher energy sources for XPS, and thicker films could be included.

* Dr Krumrey comment that repeating the KC for newer members is okay but expressed that the scientific progress is limited and running a KC is considerable amount of work, so there must be a good argument to do it. Dr Shard commented that other WGs regularly repeat KCs to demonstrate continued capability. Dr Krumrey posited that the hafnia KC does this, and so if there is no interest from newer members, he sees no reason to repeat it. Dr Shard responded that the evidence suggests participants are not claiming a CMC for hafnium oxide. Dr Shard asked which SAWG members are planning to submit a CMC claim based on the successful K157: KRISS were unsure and NIST stated an intention to make a claim for XRR on the thicker range of hafnia.

**New TG on CMC quality**  
Led by Dr Mark Lewin (NMIA) from the OAWG. Dr Lewin will be putting together an e-learning module for writing and reviewing CMCs and is looking for case studies to help educate reviewers and writers. Contact Dr Shard if SAWG members want contact with Dr Lewin.

**Uncertainty convention in CCQM CMCs**At the 22nd SAWG meeting, Dr Maré Linsky (NMISA) presented slides regarding the regarding uncertainty conventions in the KCDB. There was a proposal to make convention “Two” standard in the KCDB, and Dr Shard asked SAWG members to review this proposal and feedback at this this meeting.As a reminder,convention “One” does not specify the relationship between the measurand value and the uncertainty range, whereas convention “Two” lists the uncertainty for the lowest and highest measurand value.

* Dr Shard asked for opinions. Dr Stosch pointed out that using convention “Two” does not exclude using convention “One”, and in fact “Two” contains “One”. Dr Shard agreed stating that it is only an issue if the uncertainty has a minimum or maximum between the highest and lowest measurand value.
* Dr Windover added that there is an assumption of linearity in “Two”, and that uncertainties in reflectivity could have non-linear nature so “Two” may not be explicit enough. Discussion continued, but generally SAWG agreed that “Two” is more appropriate.

**Potential future CMCs (non-agenda item)**Dr Shard invited SAWG members to say what CMCs they would like to claim and what activities in SAWG will support their measurements and metrology:

* Dr Minelli suggested activities toward characterization of coatings and functional coatings: CMCs, reference materials, and state-of-the-art technology that can characterize these coatings to validate and establish traceability for methods that are used by industry. She then gave several examples in the nanomedicine space (e.g. targeted drug delivery).
* Dr Windover suggested that oxidation states and stoichiometry of multilayer thin oxide film would be interesting to the semiconductor community. Buried layers and interfaces also interesting with regards to semiconductor capping layers. Multilayer reference materials would be useful. Dr Shard asked Dr Windover about dopant concentration in semiconductors, citing some CMCs in CCQM from NIST and BAM for dopants in silicon (from 2005). Dr Windover confirmed there is a project underway to replenish semiconductor RMs and develop new research grade test materials and there is possibly synergy there. Dr Shard offered to investigate the origin of the doped silicon CMCs and Dr Windover offered to contact the lead for the semiconductor RMs and test materials project and attempt to incorporate its activities into SAWG. Dr Shard asked Dr Windover to report back to SAWG by September 2025.
* Dr Jordaan commented that quantitative analysis of EDX is most interesting to them, citing pilot study P230 for example.

# **Update on P230: Elemental composition of an ionic liquid surface** / J. Radnik (BAM)

Dr Radnik gave an update on P230 on elemental composition of an ionic liquid surface. A copy of Dr Radnik’s slides will be available in the SAWG documents. The protocol has been tested and distributed to participants, and the samples are ready to be distributed. Dr Radnik confirmed that the following participants (and techniques) for pilot study: INMETRO (EDS), NIM (XPS, Element Analysis, IC, ICP-OES), KRISS (XPS), NMSIA (XPS, EDS?), PTB (XRF), NPL (XPS, EDS?), BAM (XPS, EDS). Dr Radnik confirmed that the current plan for the pilot study is to provide 2 to 3 samples by Summer 2025, and have results returned by November 2025. Preliminary results will be presented at the next SAWG meeting in April 2026.

* Dr Shard asked about the deadline for participants to register. Dr Radnik asked that participants are confirmed within one month (May 2025).
* Dr Windover suggested that the HAXPES group at NIST may be interested and will also contact Dr Justin Gorham for his opinion.
* Dr Kim asked whether the samples are stable over time. Dr Radnik confirmed that the one of the samples is stable over several years. Dr Radnik added that water can absorb into the samples but a protocol for removing it is including in the protocol.
* Dr Jordaan expressed interest to participate but is currently unsure whether they could provide EDS measurements.
* Dr Krumrey asked whether the January 2024 protocol is still valid. Dr Radnik confirmed it is.
* Dr Archanjo enquired about the sample thickness. Dr Radnik said that the ionic liquid samples are deposited as droplets, so they are several millimeters thick, and suitable for EDS.

# Update of the Raman Task Group / L.-L. Tay (NRC)

Dr Li-Lin Tay (NRC) presented a report on the Raman TG activities. A copy of Dr Tay’s slides will be available in the SAWG documents.

**(1) Raman measurement related activities within VAMAS TWA 41 and 42 as well as the various activities from the ad-hoc international Raman metrology group**

At the 22nd SAWG meeting, Dr Tay spoke about the CHARISMA project which is soon coming to an end. In this meeting, Dr Tay said that she will give a larger update on this activity at the next SAWG meeting when the results are available.

**(2) Progress on traceable quantitative measurements of polymer weight fraction with Raman spectroscopy**

Dr Tay gave a summary of the proposed pilot study on traceable quantitative measurements of weight fraction of a two-component mixture polymer film, with the measurand as weight fraction. They showed the results of the long-term stability test suggest at the previous meeting and demonstrated an issue involving the removal of benzene solvent from the samples.

* Dr Shard enquired about the Raman instrument calibration during the long-term study, specifically absolute Raman intensity. Dr Tay confirmed that the instrument was calibrated each time for wavenumber and relative intensity but believes the ratio should remove instrumental contribution. Dr Hight-Walker added that absolute Raman intensity is not comparable. Dr Shard asked if there is any reason for the calibration curve to be linear, to which Dr Tay responded they are unsure and they would discuss with Dr Shard later.
* Dr Yao asked whether the average spectra were calculated manually or by the acquisition software. Dr Tay said that they used the acquisition software but commented that the protocol is open to participants using a fewer number of random acquisition locations. Dr Rossi added that the advantage of mapping is that is avoids potential inhomogeneity issues. Dr Tay addressed the question of phase separation posed by Dr Sebastian Wood (NPL), stating that they did not observe any phase separation, but that mapping would identify the effect if it was happening.
* Dr Rossi asked whether an accelerated method was used to remove the observed solvent contribution. Dr Tay clarified that samples were heated to 50oC over 20 hours in vacuum to drive out any benzene sequenced into the film.
* Dr Windover asked whether entire spectra pattern modelling has been attempted. Dr Tay said not in this study but attempted it previously on a linear combination of polymers, and did not obtain a linear response. Dr Tay said they would try it on the long-term study data.
* Dr Shard asked about a timescale for the pilot study. Dr Tay said that due to the late discovery of the benzene solvent issue (around January 2025), they would need more time to resolve it fully, possibly a few months. Dr Shard offered to request a pilot study number in the meantime and then follow up with Dr Tay in August 2025.

# **Update on VAMAS TWA2 project on lattice strain in SixGe1-x** / R. Stosch (PTB)

Dr Rainer Stosch (PTB) presented results from a VAMAS joint ILC between TWA42 and TWA46 (new working area on semiconductors) on standardized lattice strain measurement in semiconductors using Raman spectroscopy. A copy of Dr Stosch’s slides will be available in the SAWG documents. Currently waiting for samples from NIST to redistribute to participants. Currently 15 participants registered (8 from NMIs, and 7 from industry). Sample distribution scheduled between April and May 2025. Deadline for result submission is August 31st 2025. Discussion of results at the ISO TC/201 meeting in September 2025 and publication in December 2025. Expected outcomes include: assessment of participants’ precision in Raman position and shift measurements; measurement uncertainties in position and shift; SI-traceable bracketing calibration for Raman spectral data; and qualification of Si­*x*Ge1-*x* standard RM for strain measurement.

* Dr Shard enquired about the thickness of the samples and whether a capping layer is present. Dr Stosch confirmed they are between 20 nm and 45 nm depending on the Si*x*Ge1-*x* composition. Dr Windover said they would check whether the samples are capped.
* Dr Rossi, Dr Tay, Dr Shard, and Dr Stosch revisited the traceability discussion from last year’s meeting, and the idea of a calibration certificate for each sample for traceability.
* Dr Hight-Walker pointed out that the PTB datapoints are outside other participants’ uncertainty. Dr Stosch commented that this likely due to the Senterra’s internal calibration procedure which they have no direct control over.
* Dr Hight-Walker asked whether the new bracketing calibration would be part of the procedure, and Dr Stosch confirmed it would not be.
* Dr Yao and Dr Stosch discussed how the strain will be calculated from the Raman shift.
* Dr Shard and Dr Stosch discussed possible influences on the measurand such as polarization, temperature and biaxial strain (e.g. local laser heating could affect measurement, so the lowest laser power is used to obtain good signal-to-noise whilst avoiding heating).
* Dr Tay enquired about penetration depths. Dr Stosch said using longer wavelength risks measuring the layer below epitaxial layer, and suggested no more than 532nm, 488 nm or ~300 nm would be best, but can’t state the exact penetration depth.
* Dr Shard, Dr Tay, and Dr Stosch discussed this study’s relevance to SAWG and whether there are better CC where this would be better placed. Dr Stosch suggested waiting until VAMAS study results are available and using next year’s meeting to decide whether to proceed to a pilot study.

# **Discussion topic: 2D materials and SAWG** / A. Hight-Walker (NIST)

Dr Shard started by confirming that there has been no objection at the CCQM plenary regarding the inclusion of activities related to 2D materials in SAWG, and then introduced Dr Angela Hight-Walker to lead a discussion on this topic. A copy of Dr Hight-Walker’s slides will be available in the SAWG documents.

* Materials were discussed. Dr Shard, Dr Reed, and Dr Hight-Walker discussed the importance of the Si and C faces of SiC and how XPS could be useful, particularly because NMI’s are considering using graphene quantum Hall resistance (QHR) as a resistance standard. Dr Reed suggested that the focus should be on the Si face as this produces the monolayer high-quality graphene that is most relevant to device manufacturers. Dr Kim added that growth of epitaxial graphene depends on SiC surface polarity (i.e. silicon or carbon face) but shows little variation for different SiC polytypes (such as 3C, 4H and 6H). Dr Tay asked whether the QHR standards doped or undoped. Dr Hight-Walker say NIST have ways to dope them, and the sample’s protective layer does dope them to some extent. Dr Hight-Walker also suggested epitaxially grown MoS2 as a material of great technological interest.
* Measurands were discussed. Dr Shard pointed out that coverage, defect density, and thickness or number of layers are all amount of material and within scope. Strain is tentative. Dr Tay said that dopant quantity would be well within SAWGs remit. Dr Radnik added that number of defects would also be of great interest. Dr Coleman agreed that 2D materials ought to be squarely in scope and suggested connecting with the CCEM TG and possibly the CCQM WG on Electrochemical Analysis with regards to batteries. Measurement methods were also discussed.
* Dr Shard suggested setting up a TG in SAWG on 2D materials and SAWG members agreed. Dr Shard said that he would appoint a TG lead within one month of the meeting. Dr Tay nominated Dr Hight-Walker, to which Dr Hight-Walker offered to take on leadership initially to help set up the TG. Dr Shard and Dr Hight-Walker will establish terms-of-reference for this new TG on 2D material within three to four months of this meeting and decide on possible pilot studies afterwards.

# **The EMP SMURFNano project** / U. Resch-Genger (BAM)

Dr Ute Resch-Genger (BAM, Germany) gave a presentation on validated and standardised measurements and quantification of surface functionalities on nanoparticles and the SMURFnano EMPIR project. A copy of Dr Resch-Genger’s slides will be available in the SAWG documents.

* Dr Tay and Dr Resch-Genger had a discussion regarding the primary cause for the offset between the optical assay results and the other techniques. Dr Resch-Genger confirmed that Fluram labelling and pH titration was done by BAM and pointed out that pH titration better matches qNMR results compared to Fluram. There was continued discussion about differences in the results between qNMR and other methods.
* Dr Tay clarified whether the optical assay was used in the VAMAS, and Dr Resch-Genger confirmed this and that participants were offered to use one type of measurement because for the small companies, optical assays are of interest, whereas a lot of the NMIs may not have a microplate reader, when participants were allowed to choose, qNMR and XPS were generally chosen by NMIs.
* Dr Tay asked whether participants would do the labelling step themselves. Dr Resch-Genger confirmed this stating this is due to the timeframe of stability of the Fluram assay.
* Dr Shard commented that SAWG may be less interested in assay techniques, and more interested in XPS and other related methods. Dr Shard ask whether there is a means to convert the N/Si ratio into density of amine groups. Dr Resch-Genger commented that this was how the data was reported by the XPS experts and how they wanted it presented in the paper. Dr Resch-Genger added that it is a goal of SMURFnano to make XPS traceable to qNMR.
* Dr Shard enquired whether any new studies will be launched soon on this topic, to which Dr Resch-Genger confirmed saying that a qNMR study will start in September 2025, with 3 samples and 6 months for measurements, and protocol to the distributed soon. Dr Shard commented that a discussion should be had regarding the XPS part of that study and that the protocol should be circulated the SAWG experts. Dr Shard asked for a show of interest amongst the SAWG member in this study: NPL, NRC (maybe after discussion with colleagues), INRIM (qNMR), and LGC.

# **Measurement of oxygen in graphene powders** / Y. Yao (NIM)

Dr Yaxuan Yao (NIM, China) gave a presentation on the measurement of oxygen in functionalised graphene powders using X-ray photoelectron spectroscopy. A copy of Dr Yao’s slides will be available in the SAWG documents. Generally, properties and applications of functionalised graphene powders depend on the oxygen content, so the accurate measurement of oxygen-to-carbon content is a useful tool for identification of GO and rGO. Dr Yao presented some results from VAMAS study A33 (led by Dr Radnik, BAM) and a recent publication from Dr Reed [Carbon, 211, 118054 (2023)] stating that current methods include using XPS but the method of sample preparation affects the measured ratio (i.e. O/C ratio decreases with applied pressure to form solid pellets).

* Dr Shard confirmed that these materials and the measurand fall within the SAWG remit. Dr Shard also suggested that the oxygen-to-carbon ratio (O/C) rather than carbon-to-oxygen (C/O) would be more appropriate measurand to avoid infinite values, and uncertainties would be less variable if you have the oxygen content expressed as a percentage.
* Dr Yao enquired about the need for accurate intensity measurements and how to ensure this. Dr Shard commented that an XPS intensity calibration is needed. In terms of traceability, either participants would have that themselves or they could get it from NPL via their online service.
* Dr Yao enquired about methods that could be used in this a potential pilot study. Dr Shard commented that are not many other methods that can accurately measure light elements for this purpose.
* Dr Krumrey asked about the size of the pellets and Dr Yao confirmed there are on the order of millimetres area and thickness. Dr Krumrey asked if there is an oxygen depth dependence which would give different results in XPS and XRF. Dr Shard commented that it is believed that most of the oxidation is on the edges of the graphene flakes so they must be lying flat to reduce oxygen enrichment at the surface and get a representative O/C ratio, hence pressed pellets are used and an XRF measurement should give similar results to XPS.
* Dr Shard asked if there is suitable RM available. Dr Yao said no, but that they have lots of graphene powders to choose from, and confirmed they are homogeneous and stable. Dr Shard commented that homogeneity and stability test may still be required.
* Dr A. Kim commented that the C 1s in XPS may contain contamination from adventitious carbon which could affect the O/C ratio and ask how we can be sure that full C 1s intensity is from the graphene powder. Dr Yao agreed that may be the case. Dr A. Kim suggested that peak fitting of the C 1s would be required.

# **Conclusion of P248 (formerly P229a) and progression to KC: P229: Quantitative analysis of alloy films** / S. M. Lee (KRISS)

Dr Lee gave an update regarding the P248 (formerly P229a) study and presented the results of pilot study. A copy of Dr Lee’s slides will be available in the SAWG documents. Most notable was the additional comparison between the certified values of Pt-content obtained with RBS vs ICP-MS. These generated new uncertainties for PtNi reference alloys, so participants will need to check their uncertainties again and report back to Dr Lee before the Draft B report. NIM proposed a new element for the uncertainty: uncertainty in the slope. XPS values are slightly above the mean value and XRF and SEM-EDS are below the mean. PERSF+MPC method gave larger uncertainties. MRRSF and ARRSF uncertainties were similar. Generally, the pilot study was successful and KRISS are planning to advance to a KC. The report on P248 (Draft B) will be circulated in August 2025.

* Dr Krumrey asked if the test alloy sample was measured by ICP-MS. Dr Lee confirmed that only the reference alloy samples were measured, not the test sample Dr Krumrey requested that the PTB measurements are separated in the comparison plot because their traceability is to themselves and their calibrated absolute scale.
* Dr Shard asked about the average Pt mole fraction value, stating that XPS results in the comparison graph are correlated and does KRISS want to weight them to the same extent as the other methods which may be more independent
* Dr Shard asked whether the NMIs have enough information that they don’t need the reference alloy, and so during the KC, could KRISS just provide test samples or would they issue a brand-new set of reference alloys. Dr Shard added that if during the KC we use the reference values, can participants get a certificate of calibration for the reference materials, because this will be part of the traceability chain Dr Lee was unsure and said that more discussion would be needed.
* Dr Windover added that by using the ICP-MS bias for the XPS corrections, they aligned better and perhaps this is worth revisiting. Dr Windover added that they think the bias between the two populations is due to the RBS reference values.
* Dr Shard asked that some discussion regarding the carbon content is included in the Draft B report.
* Dr Shard praised the results of the pilot study and agreed that we should move straight to the KC after providing a few more details for the Draft B report.
* Dr Shard said that the NPL will write up their XPS protocol fully (including intensity calibration and background subtraction method and all the parameter required to do this experiment so that it can be replicated by others and becomes a more general solution, i.e. traceability via the protocol, not via KRISS. This avoids the issue of waiting for the coordinating laboratory to make a CMC claim first. NPL will pass their protocol to KRISS.

# **SAWG Group Photo**

The in-person participants convened in the BIPM garden for a group photograph, which is shown below. An online participants’ photograph was also taken via screen capture which is shown below.

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**Back row, left to right:** Dr Andrea Rossi, Dr Michael Krumrey, Dr Rainer Stosch, Dr Egor Sobina, and Dr Hiroyuki Matsuzaki. **Front-row, left to right:** Dr Benjamen Reed, Dr Li-Lin Tay, Dr Alexander Shard, Dr Seung Mi Lee, Dr Benjamen Reed, and Dr Yaxuan Yao.



# Update on K-136.2025 and P243: **Measurement of porosity properties of nanoporous silica** / A. E. Sadak (TÜBİTAK)

Dr Ali Enis Sadak (TUBITAK, Republic of Turkiye) gave an update regarding K-136.2025 and P243 on the measurement of porosity properties of nanoporous SiO2. An overview of previous KCs led by UNIIM was provided (i.e. K136, K153, and K172). On December 12th 2024, two vials (one primary and one backup) were sent to each of the 5 participating NMIs. Dr Sadak presented the homogeneity and short time stability experiments (both within 1 % to 5 % range) as well as the long time stability results (within 1 % to 5 %). Participants include TUBITAK-UME, VNIIM, INMETRO, NIS, NPL, and NIM. Timeline was revised with the reporting of results due on June 10th 2025, Report A by the end of November 2025, and report B by the end of May 2026.

# **Interlaboratory comparison on microplastic characterization using spectroscopic methods** / A. M. Rossi (INRIM)

Dr Rossi gave a presentation about the ISO 16094-2 intercomparison study on vibrational spectroscopy for microplastics in water under the auspices of the EURAMET food project PlasticTrace. Dr Rossi presented the results, which were also presented to the intercomparison participants and to the ISO annual meeting in October 2024. ISO TC147/SC2/JWG1 Microplastics. Interlaboratory comparison between three reference samples generated by NIVA (Norwegian Institute for Water Research), JRC, and BAM respectively. Results are expected to be published next month (May 2025)

* Dr Shard asked whether there is a microplastics TG in CCQM. Dr Rossi confirmed this (CCQM-TG-NMMS) and the chair of this TG is Dr Åsa Jämting with vice-chair Dr Enrica Alasonati who is also leader of the interlaboratory comparison work package in the PlasticTrace project. Dr Shard and Dr Rossi discussed doing activities with other WGs.
* Dr Shard and Dr Rossi then discussed the methods of the interlaboratory study and how the particle counting is achieved. Dr Shard commented that it time-consuming to do a Raman map and asked whether simulated Raman spectroscopy (SRS) has been attempted. Dr Rossi agreed this is promising technique and a sample was being tested on an SRS system, but no results have been received yet.
* Dr Hight-Walker asked whether the microplastics TG is aware of the Raman TG in SAWG. Dr Rossi confirmed this, but Dr Tay commented that they have had no interaction yet. Dr Hight-Walker commented that there is a big opportunity available in that interaction.
* Dr Shard instead suggested that he will contact the chair of the TG on microplastics and express SAWG’s interest in a leading a study in the area of microplastics. Dr Shard asked Dr Rossi to suggest a study that SAWG could lead including what materials and measurands such a study would focus on.

# **The CMC review process and KCDB** / R. Stosch (PTB)

Dr Stosch gave an extensive tutorial on the CMC review process and the KCDB. Dr Stosch covered CMCs in CIPM MRA framework, the CMC review process, CMC review criteria, the MRA (Mutual Recognition Arrangement), using the KCDB, the KCDB review process and how to register as a CMC reviewer. Dr Stosch also demonstrated the KCDB application programming interface (API) which provides programmable access to the KCDB, and showed some SAWG specific CMC statistics.

* Dr Tay asked whether bilateral and supplementary comparisons can be used to support CMCs. Dr Stosch clarified that KCs, supplementary comparison, sometimes bilateral studies can be used to support a CMC, but not a pilot study. CMC writers should check that the KC “how far the light shines statement” can support the CMC being claimed. Claims beyond this scope require justification and additional evidence.
* There was a discussion on the importance of CMC to NMIs between Dr Hight-Walker, Dr, Stosch, Dr Tay, Dr Krumrey, and Dr Windover. There was continued discussion on how a quality management system QMS fits in and that if you want to claim to an internationally recognised measurement, you need a CMC or you can only reference own national standards.
* Dr Shard thanked Dr Stosch for his tutorial and requested that SAWG members register as CMC reviewers.
* Dr Shard said that the content of Dr Stosch’s presentation can be put into a SAWG guidance document and asked for volunteers (preferably those who have experience writing or reviewing CMCs) to begin drafting this document before September 2025.
* Dr Stosch stated that CIPM offer CMC submitters and CMC reviewers courses from time-to-time that are very useful and there is a training website that details when these courses are available including some online materials. Dr Shard said he would share a link to SAWG members [https://e-learning.bipm.org/].

# **Review of SAWG strategy and planning of future comparisons** / A. Shard (NPL)

The current SAWG strategy can be accessed from the BIPM website or the documents from this meeting. Dr Shard shared a document with updates to the SAWG strategy and primary aims, titled “SAWG-Strategy2021-2030\_2025update\_V2” circulated 28th March 2025. Key addition is: Develop the metrology infrastructure and highest point of reference for nanoscale chemical measurements in support of advanced semiconductor and quantum applications. For example, SAWG will develop a strategy and plan to underpin the chemical measurement of 2D materials such as graphene. There were no objections.

A pictorial view of future comparisons was shown including some of the studies discussed in this meeting. Dr Shard commented that SAWG underdelivered within 2020 and 2030, i.e. SAWG promised 8 KC but only delivered 4 or 5. So SAWG needs to show more activity.

* Dr Rossi suggested that SAWG meet twice a year to ensure progress is being made in SAWG activities. Dr Shard agreed and a review meeting is planned for September online.

# **AOB and Close**

Dr Shard asked if there was any other business, and none was brought forward by online and in-person participants. Dr Shard thanked all in-person and online participants and closed the 2025 SAWG meeting.